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FOR
FULL-SCALE OPERATIONS AT THE BIOREMEDIATION FACILITY

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Prepared for:

SOUTHERN DIVISION
NAVAL FACILITIES ENGINEERING COMMAND
2155 EAGLE DRIVE
P.O. BOX 190010
NORTH CHARLESTON, SOUTH CAROLINA 29419-9010

Prepared by:

MORRISON KNUDSEN CORPORATION
2420 MALL DRIVE
CORPORATE SQUARE 1 - SUITE 211
NORTH CHARLESTON, SOUTH CAROLINA 29406

SOUTHWEST LABORATORY OF OKLAHOMA, INC.

TM

Standard Operating Procedure

Determination of PETN in Soil and Water by HPLC

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APPROVALS	DATES
Procedure Prepared By	Date 04/01/98
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1.0 Scope and Application

1.1 Method SW846-8330, Rev 0, published September 1994 is intended for the analysis of explosives residues by High Pressure Liquid Chromatography (HPLC). This Standard Operating Procedure (SOP) is used to determine the concentration of the following compound in a water, soil or sediment matrix:

COMPOUND NAME	ABBREVIATION	CAS NUMBER
Pentaerythritol tetranitrate	PETN	75-11-5

1.2 The subject analyte is used as an initiating agent and it is extremely sensitive to initiation. When weighing, homogenizing, or making stock solution for calibration standards, caution should be taken. This method is restricted to use by or under the supervision of analysts experienced in the use of HPLC, skilled in the interpretation of chromatograms, and experienced in handling explosive materials. Each analyst must demonstrate the ability to generate acceptable results with this method.

1.3 The practical quantitation limits (PQL) of the target analyte is presented in Table 1.

2.0 Summary of Method

2.1 One extraction is performed for the nitroaromatics and nitramines (reference SOP SWL-OL-200) and PETN. All extracts are spiked with the surrogate 3,4 DNT. Extracts are first analyzed at a wavelength of 254nm for the nitroaromatics and nitramines, then the wavelength is adjusted to 220nm and the extracts are reanalyzed for PETN.

2.2 Aqueous samples are diluted 2X (v/v) with the appropriate solvent, filtered, separated on a spherisorb ODS-II reverse phase column (or equivalent) determined at 220 nm, and confirmed on a LC-CN column.

2.3 Soil and sediment samples are extracted using acetonitrile in an ultrasonic bath. The extracts are filtered and diluted 2X (v/v) with 0.5 % calcium chloride/water. Refer to Section 7.0 for extraction procedure.

3.0 Interferences

3.1 Any material which are extracted from soil with acetonitrile, coeluted with the explosives through the HPLC column, which absorb UV radiation at 220 nm may cause interferences.

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3.2 Carryover from analysis of a highly contaminated sample can result in an apparent contamination of the succeeding samples. This interference is minimized by re-analyzing heavily contaminated sample following dilution.

4.0 Apparatus and Materials

4.1 Primary HPLC System

- Pump — HP Model 1050 quaternary system (or equivalent.)
- Column – Spherisorb ODS-II column (or equivalent)
- Detector — HP Model 1050 Variable UV (or equivalent.)
- Autosampler — HP Model 1050 autosampler (or equivalent.)
- Data System — VG Minichrom data system or equivalent.

4.2 Confirmation HPLC System

- Pump – Waters model 510 high pressure pump capable of 6000 psi at 10 ml/min. or equivalent.
- Column – Supelco LC-CN, 5 µm, 25cm x 4.6mm ID (or equivalent).
- Detector Waters model 486 variable wavelength detector (or equivalent).
- Autosampler – Waters model 715 WISP or equivalent.
- Data System – VG Minichrom or equivalent.

4.3 Other Equipment

- NEY-300 Ultrasonic Bath equipped with drainage, that can hold water at room temperature to 35°C (or equivalent). The sonicator is kept covered during sonication to keep the samples in the dark.
- BLUE M Convection Heating Oven with the temperature range of 0-300°C or equivalent.
- Balance ± 0.0001 g.

4.4 Materials

- Filters – Gelman Accuspec CR 0.45 µm filters for filtering extracts, or equivalent
- Filters – Supelco Nylon 66 0.2 µm x 47 mm membranes for filtering chromatographic solvents or equivalent.
- Glass Pipettes – Class A, glass, 50 mL, 10 mL, 5 mL, 4 mL, 2 mL, 1 mL

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- Vials – 15 mL, 20 mL, 40 ml, glass, Teflon lined screw cap or crimp top.
- Syringes – 5 mL, 10 mL, glass or polypropylene.
- Volumetric flasks – Class A - 10 mL, 20 mL, 50 mL, 100 mL, 200 mL, 250 mL.
- Mortar and pestle.
- SMI pipetter – 50-250 µL, and 10-60 µL.

5.0 Reagents

5.1 HPLC grade chemicals shall be used in all tests. It is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lowering the accuracy of the determination.

- Acetonitrile, CH3CN – HPLC grade.
- Methanol, CH3OH – HPLC grade.
- Water, H2O – HPLC grade.

5.2 Stock Standard Solutions. Analyte Standards obtained from AccuStandard (equivalent vendor) at 1000 µg/mL.

- PETN – Standard Analytical Reference Material

5.3 These immediate stock standard solutions are kept at 4°C in the dark for up to one year or shorter according to manufacturer expiration date.

5.4 Intermediate stock standard solution is prepared from the immediate stock standard solution in methanol and the intermediate stock standard is refrigerated at 4±2°C and kept in the dark upon preparation for six months.

5.5 Working Calibration Standards for soil matrices.

5.5.1 Prepare 5 point working calibration standards fresh, if CCV response is ≥15% by diluting the intermediate stock standards by 50:50 (v/v) with a 5 g/L (0.5%) Calcium Chloride/water and acetonitrile. Analyst judgement should determine if new calibration standards are required.

5.5.2 Five-level calibration standards are prepared from the intermediate stock standard in the manner that the low level standard is at or near the MDL , and the upper extent of the high level standard is at the detector linear range. Each level is injected one time.

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5.6 Initial Calibration Check Standard: If a project requires and a source can be found, a source independent of that used for initial calibration will be used as a QC standard to check the initial calibration. The same criteria for the CCV will be used.

5.7 LCS (Laboratory Control Spike) and LCSD (Laboratory Control Spike Duplicate)

LCS and LCSD are prepared from stock standard solutions that differ from the intermediate stock standard solution which is used to prepare the standards. LCS contains the analytes of interest at midpoint level or higher.

5.8 MS (Matrix Spike) and MSD (Matrix Spike Duplicate)

Matrix Spikes and Matrix Spike Duplicates are utilized as verification spikes. The same solution which is used for LCS and LCSD is used for MS and MSD. The MS and MSD will be used to evaluate the effects of the matrix on the sample analysis.

5.9 Surrogate – Since only one extraction will be performed for nitroaromatics, nitramines, and PETN analysis, the laboratory uses surrogate results of 3,4 DNT from the analysis of the nitroaromatics and nitramines to determine extraction efficiency and matrix effects. PETN extraction efficiency and matrix effects are monitored with the LCS and matrix spike. If a surrogate recovery is outside acceptance windows in the nitroaromatic and nitramine analysis, the sample will be re-extracted for all compounds.

5.10 Eluent

5.10.1 To prepare 1 liter 50% Methanol/H2O for the mobile phase, add 500 ml of methanol to 500 mL of HPLC water.

5.10.2 To prepare 1 liter of 50% Acetonitrile/H2O, add 500 ml of Acetonitrile to 500 ml of HPLC water.

5.10.3 The eluent should be filtered through a 0.45 µm filter subsequent to use.

5.10.4 The eluent should have helium continuously bubbled through the solution in order to remove air.

5.10.5 Prepare Calcium chloride by adding 5 g of the solute to 1000 mL of HPLC water. Stir the solution on the stirring plate until all of the Calcium chloride has been dissolved. Place the solution in the refrigerator at 4° C if the solution is not used.

5.10.6 Prepare 32.5% of sodium chloride by dissolving 325g of NaCl in to 100 mL of Millipore water (or equivalent).

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6.0 Sample Collection, Preservation, and Handling

6.1 Grab samples must be collected and stored in glass containers. Follow conventional sampling procedures, or those developed for site specific cases.

6.2 Samples must be stored in the dark at 4 ± 2°C from the time of collection prior to analysis. Soil or sediment samples must be extracted within 14 days of the collection, and analyzed within 40 days of extraction. Water samples must be extracted within 7 days of collection, and analyzed within 40 days of extraction.

6.3 Soil and sediment samples after collection should be air dried at room temperature or colder to constant weight. While it is possible to analyze wet soil samples, it is much more difficult to obtain a homogeneous sub sample on a wet sample. If wet soil samples are to be analyzed, a moisture determination must be made on a separate sub sample.

7.0 Procedure

7.1 Sample Preparation – samples are extracted as per SOP, SWL-OL-200, *"Nitroaromatics and Nitramines by HPLC"*. This procedure is as follows:

7.1.1 High water Aqueous Samples - 5.0 mL of the sample will be spiked with appropriate amount of matrix spike solution, filtered and kept in the dark until analysis. Prior to analysis, 0.5 mL of the filtered sample will be diluted with 0.5 mL of methanol in the autosampler vial, capped and shaken thoroughly. The sample should be ready for analysis.

7.1.2 Soil and Sediment Samples - should be homogenized thoroughly prior to weighing. Using disposable weigh dishes, weigh out 20g of wet weight sample (the weight should yield 2.0 g after drying).

Dry the soil samples in air at room temperature or colder. A forced-air oven at room temperature may also be used and be careful not to expose the samples to direct sunlight. Place the samples in the disposable dishes and place the dishes in the flat container. Cover the samples with aluminum foil while drying in order not to be exposed to the excessive light or sunlight. Upon drying, grind the samples thoroughly in a methanol rinsed mortar.

7.1.2.1 Soil Sample extraction - Place a 2.0g sub-sample of each soil sample in a 40 mL glass VOA vial. Add 10.0 mL of acetonitrile volumetrically, cap the vial with a teflon lined cap, and place in a cool ultrasonic bath for 18 hours (or the time required by site specific QAPP's). Water at a constant flow rate should enter the sonicator and dispose out of the ultrasonic bath. During sonication, the samples should be kept at room temperature or below.

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7.1.2.2 After sonication, allow the samples to settle for 30 minutes. If the samples are not analyzed, promptly store the samples at 4° C until analysis.

7.1.2.3 If the samples are to be analyzed remove 5.0 mL of supernatant, using a 5.0 mL disposable glass pipette or Rainin pipetter with polypropylene disposable tips. Transfer the supernatant to 12 .0 mL glass vial, volumetrically transfer 5.0 mL of calcium chloride solution (see section 5.9.5). Shake or Vortex the vial for 15-30 seconds or until the solution is thoroughly mixed. Let the solution stand for 15 minutes. Transfer the supernatant to 10 mL disposable syringe containing Gelman Accuspec CR 0.45. Filter the solution by discarding the first 1-2 mL and retain the remainder in another 12 mL glass vial with teflon-lined screwcap. Keep the extracts in 4°C and in a dark place until analyzed, but not for longer than 40 days.

7.1.2.4 Prior to analysis, transfer the solution to autosampler vials. The sample has already been diluted by a factor 2X with a 0.5% calcium chloride solution.

7.2 Chromatographic Conditions

Mobile Phase: 50/50 (v/v) methanol/HPLC water
Flow rate: 1.0 mL/min
Injection volume: 200 µL
UV Detector: 220 nm

7.3 A number of standards are analyzed prior to the actual initial calibration standards in order to make certain that the HPLC is within operating conditions and the system has established the retention time windows.

7.4 Calibration of HPLC

7.4.1 Following SW-846, method 8330, the initial calibration standard at the minimal five- concentration is injected only one time. The %RSD of the calibration factors should be ≤20%. If the %RSD for the target analyte is greater than 20%, the correlation coefficient for the calibration line can be used if it is ≥0.995. The curve should be linear and intercept at zero. Use the mean calibration factors for all calculations as long as the continuing calibrations described below meet specifications.

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7.4.2 Calibration Factor (CF): 5-Level

$$CF_i = \frac{\text{Peak Area of Standard}}{\text{Mass Injected (ng)}}$$

$$\text{MEAN CF} = \sum_{i=5}^{n} \frac{CF_i}{n}$$

$$\%RSD = \frac{STD \times 100}{\text{MEAN CF}}$$

Where:

n = (5) Total number of values
STD = Standard deviation of the calibration factors
CF_i = Calibration factors

7.4.3 Continuing Calibration using the mid point standard should be injected at the beginning of an analysis day and about every ten injections and at the end of the analysis. This standard should agree within a percent deviation ≤ 15% with mean obtained from the initial calibration. If a continuing calibration fails, it may be reinjected and analyses restarted from that point if it meets specifications on reinjection. If it fails again, the initial calibration must be repeated.

7.4.4 Percent Deviation (%D)

$$\%D = \frac{R1 - R2}{R1} \times 100$$

Where:

R1 = Calibration factor from first analysis
R2 = Calibration factor from second analysis.

7.4.5 Spherisorb ODS-II reverse column (or equivalent) is utilized for the quantitation analysis.

7.4.6 LC-CN column (or equivalent) is utilized to confirm the above PQL analyte

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7.4.7 Retention time windows should be established for each new calibration. Average the retention time for each injection of PETN for all initial calibration analysis and continuing calibration analysis. Determine the standard deviation. The window for PETN is ± 3 times the standard deviation. If chromatographic retention drift should occur, make certain that the mobile phase is fresh and has not evaporated and that the pumps are functioning properly. If the column itself has deteriorated and caused loss of efficiency and retention, it may have to be replaced.

7.4.8 Analyst experience and judgment are utilized for interpretation of data if one of the analytes is slightly outside of the retention times windows.

7.5 Sample Analysis

7.5.1 Analyze the samples using the chromatographic conditions given in section 7.2. Confirm each measurement by injecting onto the CN column.

7.5.2 Calculation for positive hits.

$$\text{Concentration } (\mu g/L \text{ or } \mu g/Kg) = \frac{(A)(Vt)(DF)}{(CF)(Vo)(Vi)}$$

Where:

A	=Area of the peak for the compound to be measured
CF	=Mean calibration factor (ng)
Vo	=Volume of water (L) or amount of soil extracted (Kg)
Vi	=Volume of extract injected (mL)
Vt	=Volume of the concentrate extracted (mL)
DF	=Dilution factor

8.0 Quality Control

8.1 Prior to preparation of stock solutions, acetonitrile, methanol, and water blanks should be analyzed to determine possible interferences with analyte peaks. If the acetonitrile, methanol, or water blanks show contamination, a different batch should be used.

8.2 Method Blanks

8.2.1 Method blanks for the analysis of aqueous samples should be organic-free reagent water carried through all sample storage and handling procedures.

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8.2.2 Method blanks for the analysis of soil samples should be uncontaminated soil carried through all sample storage, extraction, and handling procedures.

8.3 Laboratory Control Spikes - The laboratory control spike (LCS) and the laboratory control spike duplicate (LCSD) are performed with each extraction batch at a rate of one LCS/LCSD per batch of twenty samples or fewer.

8.4 Matrix spike (MS) and matrix spike duplicate (MSD) - are performed at a rate of one set per batch of 20 samples or fewer.

8.5 Surrogate — The result from the analysis of the nitroaromatics and nitroamines is reported.

9.0 Method Performance

9.1 The accuracy and precision obtained in this method will be determined by the sample matrix, sample preparation technique, and the calibration procedures applied.

9.2 Sample PQLs are highly matrix dependent. the PQLs listed on Table 1 are reported based on the method and sample matrix. The MDLs are obtained using the SOP *"Definition and Determination of Detection Limits"*, SWL-GA-113.

10.0 Data Verification

10.1 If the compound is found above the PQL (reporting limit) in the quantitation column and it is within the established retention time window, the confirmational analysis of the compound will be performed. If the compound is found within the retention time window of the confirmational analysis, the compound will be reported. If the compound in the MS/MSD and LCS/LCSD exceeds the laboratory QC limits, the batch of samples will be reextracted and reanalyzed.

10.2 Due to insufficient data points, QC limits of 40-160 will be utilized for PETN.

11.0 References

11.1 Bauer, C.F., S.M. Koza, and T.F. Jenkins, "Collaborative Test Results for a Liquid Chromatographic Method for the Determination of Explosives Residues in Soil," manuscript submitted to the Journal of the AOAC, April 1989.

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11.2 Department of the Army, "Reversed-Phase HPLC Method for the Determination of Explosive Residues in Soil," Appendix B, provided by Dennis J. Wynne, Chief, Technology Division, U.S. Army Toxic and Hazardous Materials Agency, Aberdeen Proving Ground, Maryland 21010-5401.

11.3 Department of the Army, "An Improved RP-HPLC Method for the Determination of Nitroaromatics and Nitramines in Water" Appendix B, provided by Dennis J. Wynne, Chief, Technology Division, U.S. Army Toxic and Hazardous Materials Agency, Aberdeen Proving Ground, Maryland 21010-5401.

11.4 SW 846-8330, Rev.0, September, 1994.

11.5 U.S. army toxic and Hazardous Material Agency. 1987, USATHAMA QA program (December 1985, 2nd ED., March 1987)

12.0 Safety - Standard precautionary measures used for handling other organic compound should be sufficient for safe handling of PETN

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TABLE 1
PRACTICAL QUANTITATION LIMITS

Compound	Water (ug/L)	Soil (ug/Kg)
PETN	14.4	250

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SECTION 3
PREVENTIVE MAINTENANCE FOR
QUANTERRA ENVIRONMENTAL SERVICES

Functional Guidelines for Evaluating Inorganic Analyses", or modifications thereof, for non-CLP type analyses.

This form of data validation provides an impartial evaluation of the laboratory's results. It is usually performed by a third party, one which was not involved with the sample analysis. Qualifiers are assigned to data, when required, according to the requirements of the data validation protocol being used.

8.11 Preventive Maintenance and Service

Facilities, instruments, equipment, and parts are subject to wear, deterioration, or change in operational characteristics. Within Quanterra®, preventive maintenance, coupled with vendor service agreements, is an organized program of actions taken to maintain facilities and equipment.

8.11.1 Analytical Instrumentation and Equipment

The primary purpose of the maintenance program is to prevent instrument and equipment failure and to minimize down time. A properly implemented maintenance program increases the reliability of a measurement system.

At each laboratory each instrument or piece of equipment shall be uniquely identified. Each operating unit shall maintain the following:

- Instrument/equipment inventory list
- Instrument/equipment major spare parts list or inventory
- External service agreement documents (if applicable)
- Instrument-specific preventive maintenance logbook or file for each functional unit.

The record of maintenance shall include at a minimum:

- Actions taken, including parts replaced
- Analyst initials and the date maintenance was performed whether by the analyst or a contracted service representative.

Quanterra® documents and describes in detail instrument and equipment preventive maintenance procedures and requirements in operation-specific SOPs.

8.11.2 Facilities

Another important aspect of the laboratory operation is the existence and maintenance of adequate, safe, and clean facilities including appropriate engineering controls such as proper ventilation, lighting, dust control, hoods, air flow, protection from extreme

temperatures, waste disposal, and a source of stable power.

The maintenance and use of these facilities and proper operations are described in the Quanterra® Chemical Hygiene Plan (CHP). The Laboratory Manager, through a facilities maintenance staff, has responsibility for ensuring a properly maintained facility. The Laboratory Manager also has the responsibility for ensuring that samples are stored properly without contamination, work areas are equipped with adequate bench, hood and operational space, and the areas are free from chemical and radiological contamination that may affect analytical results.

8.11.3 Frequency of Maintenance

The frequency of maintenance must consider manufacturer's recommendations and previous experience. Schedules of preventive maintenance along with the recommended frequency are given in Tables 8.11-1 through 8.11-27 for analytical instrumentation and equipment. Frequency of maintenance for the facility systems is documented in the Quanterra® CHP.

8.12 Other Requirements

8.12.1 Water

High purity water (i.e., ASTM reagent grade or equivalent water) will be used in all metals, radiological, wet chemistry, and organic analyses. Proof of contaminant-free water is shown through the use of the reagent waters as method blanks which are analyzed on a daily basis for the analyte of interest. This water is obtained by the use of either a commercial ion-exchange deionizing, distillation, or reverse osmosis unit plus an appropriate polishing unit. The resulting water has a maximum conductivity of 1.0 umho-cm at 25°C or a minimum resistivity of 1.0 Mohm at 25°C. Conductivity and/or resistivity will be monitored and documented daily or on each day that water is dispensed for analytical use.

For volatile analyses the water may be further purified by purging with an inert gas before use to remove potential traces of organic solvents.

Water monitoring procedures used by Quanterra® operating units are detailed in operation-specific SOPs.

8.12.2 Compressed Air and Gases

Ultra high-purity compressed gases from preapproved vendors or in-house gas generators will be used when required for instrumentation. These air and gases must meet the requirements and specifications of the analytical methods performed. In-line filters

TABLE 8.11-1
Instrument Maintenance Schedule
Ion Chromatograph[1]

As Needed	Daily	Weekly	Monthly	Semi-annually
Clean micromembrane suppressor when decreases in sensitivity are observed.	Check plumbing/leaks.	Check pump heads for leaks.	Check all air and liquid lines for discoloration and crimping, if indicated.	Lubricate left hand piston.
Check fuses when power problems occur.	Check gases.	Check filter (inlet)	Check/change bed supports guard and analytical columns, if indicated.	Clean conductivity cell.
Reactivate or change column when peak shape and resolution deteriorate or when retention time shortening indicates that exchange sites have become deactivated.	Check pump pressure.			Check conductivity cell for calibration.
De-gas pump head when flow is erratic.	Check conductivity meter.			

TABLE 8.11-2
Instrument Maintenance Schedule
LACHAT Auto Analyzer[1]

As Needed	Daily	Monthly	Semi-annually	Annually
Prepare fresh reagents.	Clean detector cell and make sure there are no trapped bubbles in detector cell.	Replace tubing.	Lubricate pump roller.	Clean pump rollers with steel wool and lubricate.
	Check tubing.	Clean pump, diluter, and XYZ Sampler.		
	Clean sample probe shaft.			

TABLE 8.11-3
Instrument Maintenance Schedule
Total Organic Halide Analyzer[1]

Daily	As Needed
Check electrodes for damage, polish the electrodes.	Examine and clean or replace pyrolysis tube.
Replace dehydrating fluid and electrolyte fluid.	Clean titration cell.
Clean quartz boat.	Observe gas flow.
Observe check valves during use for backfeed.	Replace reference electrode fluid.
At end of each day of use, wash out absorption module, empty electrolyte and fill cell with DI water. Empty dehydrator tube	Change quartz wool.
Perform cell performance check.	Replace o-rings and seals.

TABLE 8.11-4
Instrument Maintenance Schedule
High Pressure Liquid Chromatograph[1]

Daily	As Needed
Check level of solution in reservoirs. If adding, verify that solvent is from the same source. If changing, rinse gas and delivery lines to prevent contamination of the new solvent.	Replace columns when peak shape and resolution indicate that chromatographic performance of column is below method requirements.
Check gas supply.	Oil autosampler slides when sample does not advance.
Flush with an appropriate solvent to remove all bubbles.	Rinse flow cell with 1N nitric acid if sensitivity low.
Pre-filter all samples.	Change pump seals when flow becomes inconsistent.
	Repack front end of column Backflush column.

TABLE 8.11-5
Instrument Maintenance Schedule
Flame Atomic Absorption Spectroscopy[1]

Daily	Monthly	As Needed
Verify proper safety precautions are working.	Clean all filters and fans.	Check drain receptacle.
Verify gas box operates properly and safely.	Change capillary tubing	Check background corrector for alignment.
Verify sensitivity using elements in UV/VIS spectrum.	Clean optical windows	Clean burner head.
		Clean nebulizer.
		Clean spray chamber.
		Check sample introduction O-rings.

TABLE 8.11-6
Instrument Maintenance Schedule
Inductively Coupled Argon Plasma/Mass Spectrometry (ICAP/MS)[1]

Daily	Weekly	Monthly	Quarterly	Annually	As Needed
Check sample waste container level.	Check peristaltic pump: proper roller pressure, sample introduction tubing, correct pump rotation, condition of drain tubing.	Clean all filters and fans.	Replace oil in roughing pumps.	Replace oil in turbo-molecular pump.	Check electronic settings for optimum sensitivity: resolution, mass calibration, ion optics, CEM, deflector voltage.
Check quartz torch condition.	Check condition of sampler and skimmer cones.	Check recirculator water level.			
Measure quartz torch for proper alignment.	Check and drain oil mist eliminator on roughing pumps.				
Clean spray chamber and nebulizer.					
Check oil level of roughing pumps.					

TABLE 8.11-7
Instrument Maintenance Schedule
ICP[1]

Daily	As Needed	Semi-annually	Annually
Check that argon tank pressure is 50-60 psi and that a spare tank is available.	Clean plasma torch assembly to remove accumulated deposits.	Change vacuum pump oil.	Notify manufacturer service engineer for scheduled preventive maintenance service.
Check vacuum pump gage. (<10 millitorr)	Clean nebulizer and drain chamber; keep free-flowing to maintain optimum performance.	Replace coolant water filter. (may require more or less frequently depending on the quality of water)	
Check that cooling water supply system is full and drain bottle is not full. Also that drain tubing is clear, tight fitting and has few bends.	Clean filters on back of power unit to remove dust.		
Check that nebulizer is not clogged.	Replace when needed: peristaltic pump tubing sample capillary tubing autosampler sipper probe		
Check that capillary tubing is clean and in good condition.	Check ytrium position.		
Check that peristaltic pump windings are secure.			
Check that high voltage switch is on.			
Check that exhaust screens are clean.			
Check that torch, glassware, aerosol injector tube, bonnet are clean.			

TABLE 8.11-8
Instrument Maintenance Schedule
Graphite Furnace Atomic Absorption[1]

Daily	Monthly	As Needed	Annually
Check gas lines and gas supply.	Check coolant level in cooling unit. Add coolant if error message appears.	Replace contact cylinder.	Notify manufacturer service engineer to clean optics.
Clean optical windows.		Adjust autosampler arm.	
Clean contact cylinders.			
Check tubes and platform: replace if corroded, faking, or if low absorbance results.			
PE4100ZL: clean fume extraction tip, replace fume extraction filter and H_2O trap.			
As needed, trim sampling capillary.			
Check drain lines and waste containers: empty as needed.			
Check acid rinse containers; fill as needed.			

TABLE 8.11-9
Instrument Maintenance Schedule
Cold Vapor Atomic Absorption (Leeman PS 200)[1]

Daily	As Needed	Annually
Change drying tube	Change pump tubing	Change Hg lamp.
Check pump tubing/drain tubing	Check/change Hg lamp	
Check gas pressure	Clean optical cell	
Check aperture reading	Lubricate pump	

TABLE 8.11-10
Instrument Maintenance Schedule
Cold Vapor Atomic Absorption (PE 5000)[1]

Daily	Monthly
Clean aspirator by flushing with DI water.	Clean cell in aqua regia.
Check tubing and replace if needed.	Clean aspirator in aqua regia.
Clean windows with methanol.	
Change silica gel in drying tube.	
Check argon gas supply.	
Adjust lamp.	

TABLE 8.11-11
Instrument Maintenance Schedule
Gas Chromatograph[1]

Daily	As Needed	Quarterly/Semi-annually/Annually
Check for sufficient supply of carrier and detector gases. Check for correct column flow and/or inlet pressures.	Replace front portion of column packing or break off front portion of capillary columns. Replace column if this fails to restore column performance or when column performance (e.g. peak tailing, poor resolution, high backgrounds, etc.) indicates it is required.	Quarterly ELCD: change roughing resin, clean cell assembly.
Check temperatures of injectors and detectors. Verify temperature programs.	Change glass wool plug in injection port and/or replace injection port liner when front portion of column packing is changed or front portion of capillary column is removed.	Semi-annually ECD: perform wipe test.
Check inlets, septa.	Replace septum (approximately every 100 injections).	Annually ELCD: change finishing resin, clean solvent filter.
Check baseline level.	Perform gas purity check (if high baseline indicates that impure carrier gas may be in use).	
Check reactor temperature of electrolytic conductivity detector.	Replace or repair flow controller if constant gas flow cannot be maintained.	
	Replace fuse.	
	Reactivate external carrier gas dryers.	
	Detectors: clean when baseline indicates contamination or when response is low. FID: clean/replace jet, replace ignitor. NPD: clean/replace collector assembly. PID: clean lamp window, replace seals. ELCD: check solvent flow weekly, change reaction tube, replace solvent, change reaction gas, clean/replace Teflon® transfer line. ECD: follow manufacturers suggested maintenance schedule	
	Reactivate flow controller filter dryers when presence of moisture is suspected.	

TABLE 8.11-11
Maintenance Schedule
Gas Chromatograph[1]
(Continued)

Daily	As Needed	Quarterly/Semi-annually/Annually
GC (continued)	HP 7673 Autosampler: replace syringe, fill wash bottle, dispose of waste bottle contents.	(continued)
	Purge & trap devices: periodic leak checks, replace/condition traps (when poor response or disappearance of reactive or poorly trapped compounds), clean sample lines, valves (if they become contaminated), clean glassware.	
	Purge & trap autosamplers: leak check system, clean sample lines, valves. PTA-30 autosampler also requires cleaning the syringes, frits, valves, and probe needles, adjustment of micro switches, replacement of Teflon® valve, and lubrication of components.	

TABLE 8.11-12
Instrument Maintenance Schedule
Mass Spectrometer[1]

Daily	As Needed[2]	Semi-Annually	Annually
Check for sufficient gas supply. Check for correct column flow and/or inlet pressure.	Check level of oil in mechanical pumps and diffusion pump if vacuum is insufficient. Add oil if needed between service contract maintenance.	Change oil in the mechanical rough pump. Relubricate the turbomolecular pump bearing wick.	Replace the exhaust filters on the mechanical rough pump every 1-2 years.
Check temperatures of injector, detector. Verify temperature programs.	Replace electron multiplier when the tuning voltage approaches the maximum and/or when sensitivity falls below required levels.		
Check inlets, septa.	Clean Source, including all ceramics and lenses - the source cleaning is indicated by a variety of symptoms including inability of the analyst to tune the instrument to specifications, poor response, and high background contamination.		
Check baseline level.	Repair/replace jet separator.		
Check values of lens voltages, electron multiplier, and relative abundance and mass assignments of the calibration compounds.	Replace filaments when both filaments burn out or performance indicates need for replacement.		

TABLE 8.11-13
Instrument Maintenance Schedule
TRAACS 800 Auto Analyzer [1]

As Needed	Daily	Monthly	Semi-annually	Annually
Replaces air filter when progressive loss of air pressure is observed.	Check air pressure gauge (22 ± 2 psi)	Change all pump tubes (or after 200 hours of pumping time)	(or after 1000 hours of pumping time)	Lightly lubricate the Linear Sample Rails (use semi-fluid lubricant)
Replace air valve tubing when occlusion in tubing is observed	Use recommended washout procedure (at end of analysis operations)	Clean sample probe shaft	Replace pump platens	Replace colorimeter lamp (or after 2500 hours of use)

TABLE 8.11-14
Instrument Maintenance Schedule
Sonicator [1]

Daily	As Needed
Daily when used: Inspect probe tips for inconsistencies (etching/pitting).	Replace probe tip.
	Disassemble and clean sonicator probe tips.
	Tune sonicator assembly.

TABLE 8.11-15
Instrument Maintenance Schedule
Analytical/Top Loading Balances[1]

Daily	Annually
Daily when used: Calibrate with check weights.	Internal weight train serviced. Gears and electronics serviced.

TABLE 8.11-16
Instrument Maintenance Schedule
Refrigerators/Walk-in Coolers[1]

Daily	As Needed
Temperatures checked and logged.	Refrigerant system and electronics serviced.

TABLE 8.11-17
Instrument Maintenance Schedule
Ovens[1]

Daily	As Needed
Temperatures checked and logged.	Electronics serviced.

TABLE 8.11-18
Instrument Maintenance Schedule
Specific Digital Ion Analyzer[1]

Daily	As Needed
Daily when used: Calibrate with check standards.	Electronics serviced.

TABLE 8.11-19
Instrument Maintenance Schedule
Turbidimeter[1]

Daily	As Needed
Daily when used: Calibrate with check standards.	Electronics serviced.

TABLE 8.11-20
Instrument Maintenance Schedule
Dissolved Oxygen Meter[1]

Daily	As Needed
Daily when used: Calibrate with check standards.	Electronics serviced.

TABLE 8.11-21
Instrument Maintenance Schedule
Conductance Meter[1]

Daily	As Needed
Daily when used: Calibrate with check standards.	Electronics serviced.

TABLE 8.11-22
Instrument Maintenance Schedule
Chemical Oxygen Demand (COD) Reactor

Daily	As Needed
Daily when used: Calibrate with check standards.	Electronics serviced.

TABLE 8.11-23
Instrument Maintenance Schedule
Spectrophotometer[1]

As Needed	Daily	Monthly	Annually
Dust the lamp and front of the front lens.	Check the zero %T adjustment.	Perform wavelength calibration at 530 nm.	Oil bearings.

TABLE 8.11-24
Instrument Maintenance Schedule
pH Meter[1]

As Needed	Daily
Clean electrode.	Verify electrodes are properly connected and filled.
Refill reference electrode.	Make sure electrode is stored in buffer.

TABLE 8.11-25
Instrument Maintenance Schedule
Fourier Transform Infrared Spectrometry (FTIR)[1]

Check desiccant every 3 months.
Check KBr window every 3 months.

TABLE 8.11-26
Instrument Maintenance Schedule
Radiological Analysis Equipment[1]

Instrument	Items Checked/Service	Minimum Frequency
Alpha Proportional	Check gas flow	Daily
	Clean sample tray	Weekly
	Check bubbler oil level	Monthly
Beta Proportional	Check gas flow	Daily
	Clean sample holders	Weekly
Liquid Scintillation	Clean sample changer	Weekly
	Check condensate trays	Weekly
	Check air filters	Monthly
Quad aß Proportional	Check gas flow	Daily
	Clean sample holders	Weekly
Gamma Spectroscopy	Check LN_2 level	Bi-weekly
	Replace plastic liner	Weekly
Alpha Spectroscopy	Clean sample holder	As needed
	Change vacuum pump oil	Every six months
LIPA	Clean sample changer	Weekly
	Check laser dye performance	Weekly
Benzene Synthesizer	Check gas tubes	Weekly
	Clean instrument	Monthly
Electrolytic Enrichment	Check electrical leads	Monthly
	Clean system	Monthly
Fluorometer	Clean sample holder	Weekly

TABLE 8.11-27
Instrument Maintenance Schedule
Total Organic Carbon Analyzer (OI 7000)

Daily	Weekly	Monthly	Semi-Annually
Check: Oxygen supply Persulfate supply Acid supply Carrier gas flow rate (~ 150 cc/min) IR millivolts for stability (after 30 min. warm-up)	Check liquid-flow-rate-pump-tubing conditions on autosampler Check injection port septum	Clean digestion vessel Clean condenser column Do the leak test	Change pump tubing

Footnotes to Preventive Maintenance Tables

(1) Refer to manufacturer's instructions for each instrument to perform maintenance operations.
(2) Also see Table 8.11-11 for applicable "As Needed" GC maintenance.

TABLE 8.11-28
Instrument Maintenance Schedule
APCI/ESI LC/MS/MS

Daily	As Needed	Semi-annually	Annually
Check helium sparge supply for adequate pressure	Oil autosampler when it is noisy or picking up the tray	Replace rough pump oil	Replace turbopump oil
Check solvent reservoirs for sufficient level of solvent	Change pump seals		Vacuum system components including fans and fan covers
Verify that pump is primed, operating pulse free	Change filters in autosampler		
Check needle wash reservoir for sufficient solvent	Replace column if excessive pressure or poor performance		
Verify capillary heater temperature	Rinse capillary with MeOH		
Verify vaporizer heater temperature	Clear capillary if clogged		
Verify manifold heater temperature	Rinse and clean corona needle		
Verify manifold pressure (~5 x 10^-6)	Replace sample inlet tube in APCI (10.1 cm)		
Verify forepump pressure (~30 to 200mtorr)	Replace fused silica tube in ESI interface		
Verify rough pump and turbopump oil levels	Clean lenses		
Verify nitrogen pressure for auxiliary and sheath gasses	Clean skimmer		
Verify that corona and multiplier are functioning			

SECTION 4
STANDARD OPERATING PROCEDURES FOR QUANTERRA ENVIRONMENTAL SERVICES

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QUANTERRA STANDARD OPERATING PROCEDURE

TITLE: DETERMINATION OF VOLATILE ORGANICS BY GC/MS BASED ON METHOD 8260B, 624 AND 524.2

(SUPERSEDES: REVIS ON 1)

Prepared by: Richard Burrows

Reviewed by: _____
Technology Standardization Committee

Approved by: _____
Director, Quality Assurance

Approved by: _____
Director, Environmental Health and Safety

Approved by: _____
Management

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1. SCOPE AND APPLICATION

1.1. This method is applicable to the determination of Volatile Organic Compounds in waters, wastewater, soils, sludges and other solid matrices. Standard analytes are listed in Tables 5 and 6.

1.2. This SOP is applicable to method 8260B. At client request, it may also be used for analysis following method 8240B. Appendices A and B present modifications to the procedures in the main SOP that are necessary for analysis of drinking water by method 524.2 and wastewater by method 624.

1.3. This method can be used to quantify most volatile organic compounds that have boiling points below 200°C and are insoluble or slightly soluble in water. Volatile water soluble compounds can be included in this analytical technique; however, for more soluble compounds, quantitation limits are approximately ten times higher because of poor purging efficiency.

1.4. The method is based upon a purge and trap, gas chromatograph/mass spectrometric (GC/MS) procedure. The approximate working range is 5 to 200 µg/L for 5 mL waters, 1 to 60 µg/L for 25 mL purge waters, 5 to 200 µg/kg for low-level soils, and 250 to 25,000 µg/kg for medium-level soils. Reporting limits are listed in Tables 1, 3 and 14.

1.5. Method performance is monitored through the use of surrogate compounds, matrix spike/matrix spike duplicates, and laboratory control spike samples.

2. SUMMARY OF METHOD

2.1. Volatile compounds are introduced into the gas chromatograph by the purge and trap method. The components are separated via the chromatograph and detected using a mass spectrometer, which is used to provide both qualitative and quantitative information.

2.2. Aqueous samples are purged directly. Generally, soils are preserved by extracting the volatile analytes into methanol. If especially low detection limits are required, soil samples may be preserved with sodium bisulfate and purged directly.

2.3. In the purge and trap process, an inert gas is bubbled through the solution at ambient temperature or at 40°C (40°C required for low level soils) and the volatile components are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbant column where the volatile components are trapped. After purging is completed, the sorbant column (trap) is heated and backflushed with inert gas to desorb the components onto a gas chromatographic column. The gas chromatographic column is then heated to elute the components which are detected with a mass spectrometer.

2.4. Qualitative identifications are confirmed by analyzing standards under the same conditions used for samples and comparing the resultant mass spectra and GC retention times. Each identified component is quantified by relating the MS response for an appropriate selected ion produced by that compound to the MS response for another ion produced by an internal standard.

3. DEFINITIONS

3.1. Batch

The batch is a set of up to 20 samples of the same matrix processed using the same procedures and reagents within the same time period. Using this method, each BFB analysis will normally start a new batch. Batches for medium level soils are defined at the sample preparation stage and may be analyzed on multiple instruments over multiple days, although reasonable effort should be made to keep the samples together.

3.1.1. The Quality Control batch must contain a matrix spike/spike duplicate (MS/MSD), a Laboratory Control Sample (LCS), and a method blank. In some cases, at client request, the MS/MSD may be replaced with a matrix spike and sample duplicate. Refer to the Quanterra QC Program document (QA-003) for further details of the batch definition.

3.2. Method Blank

A method blank consisting of all reagents added to the samples must be analyzed with each batch of samples. The method blank is used to identify any background interference or contamination of the analytical system which may lead to the reporting of elevated concentration levels or false positive data.

3.3. Laboratory Control Sample (LCS)

Laboratory Control Samples are well characterized, laboratory generated samples used to monitor the laboratory's day-to-day performance of routine analytical methods. The LCS, spiked with a group of target compounds representative of the method analytes, is used to monitor the accuracy of the analytical process, independent of matrix effects. Ongoing monitoring of the LCS results provides evidence that the laboratory is performing the method within accepted QC guidelines for accuracy and precision.

3.4. Surrogates

Surrogates are organic compounds which are similar to the target analyte(s) in chemical composition and behavior in the analytical process, but which are not normally found in environmental samples. Each sample, blank, LCS, and MS/MSD is spiked with surrogate

standards. Surrogate spike recoveries must be evaluated by determining whether the concentration (measured as percent recovery) falls within the required recovery limits.

3.5. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

A matrix spike is an environmental sample to which known concentrations of target analytes have been added. A matrix spike duplicate is a second aliquot of the same sample which is prepared and analyzed along with the sample and matrix spike. Matrix spikes and duplicates are used to evaluate accuracy and precision in the actual sample matrix.

3.6. Calibration Check Compound (CCC)

CCCs are a representative group of compounds which are used to evaluate initial calibrations and continuing calibrations. Relative percent difference for the initial calibration and % drift for the continuing calibration response factors are calculated and compared to the specified method criteria.

3.7. System Performance Check Compounds (SPCC)

SPCCs are compounds which are sensitive to system performance problems and are used to evaluate system performance and sensitivity. A response factor from the continuing calibration is calculated for the SPCC compounds and compared to the specified method criteria.

4. INTERFERENCES

4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. The use of ultra high purity gases, pre-purged purified reagent water, and approved lots of purge and trap grade methanol will greatly reduce introduction of contaminants. In extreme cases the purging vessels may be pre-purged to isolate the instrument from laboratory air contaminated by solvents used in other parts of the laboratory.

4.2. Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) into the sample through the septum seal during shipment and storage. A field blank prepared from reagent water and carried through the sampling and handling protocol can serve as a check on such contamination.

4.3. Matrix interferences may be caused by non-target contaminants that are coextracted from the sample. The extent of matrix interferences will vary considerably from source to source depending upon the nature and diversity of the site being sampled.

4.4. Cross-contamination can occur whenever high-level and low-level samples are analyzed sequentially or in the same purge position on an autosampler. Whenever an unusually concentrated sample is analyzed, it should be followed by one or more blanks to check for cross-contamination. The purge and trap system may require extensive bake-out and cleaning after a high-level sample.

4.5. Some samples may foam when purged due to surfactants present in the sample. When this kind of sample is encountered an antifoaming agent (e.g., J.T. Baker's Antifoam B silicone emulsion) can be used. A blank spiked with this agent must be analyzed with the sample because of the non-target interferences associated with the agent.

5. SAFETY

5.1. Procedures shall be carried out in a manner that protects the health and safety of all Quanterra associates.

5.2. The Chemical Hygiene Plan (CHP) gives details about the specific health and safety practices which are to be followed in the laboratory area. Personnel must receive training in the CHP, including the written Hazard Communication plan, prior to working in the laboratory. Consult the CHP, the Quanterra Health and Safety Policies and Procedures Manual, and available Material Safety Data Sheets (MSDS) prior to using the chemicals in the method.

5.3. Consult the Quanterra Health and Safety Policies and Procedures Manual for information on Personal Protective Equipment. Eye protection that satisfies ANSI Z87.1 (as per the Chemical Hygiene Plan) and a laboratory coat must be worn in the lab. Appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately. Disposable gloves shall not be reused.

5.4. The health and safety hazards of many of the chemicals used in this procedure have not been fully defined, therefore each chemical compound should be treated as a potential health hazard. Additional health and safety information can be obtained from the MSDS files maintained in the laboratory. The following specific hazards are known:

5.4.1. Chemicals that have been classified as carcinogens, or potential carcinogens, under OSHA include: Acrylonitrile, benzene, carbon tetrachloride, chloroform, 1,2-dibromo-3-chloropropane, 1,4-dichlorobenzene, and vinyl chloride.

5.4.2. Chemicals known to be flammable are: Methanol.

5.5. Exposure to chemicals must be maintained **as low as reasonably achievable**, therefore, unless they are known to be non-hazardous, all samples should be opened, transferred, and

prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.6. The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operations will permit.

5.7. All work must be stopped in the event of a known or potential compromise to the health and safety of a Quanterra associate. The situation must be reported **immediately** to a laboratory supervisor.

5.8. Laboratory personnel assigned to perform hazardous waste disposal procedures must have a working knowledge of the established procedures and practices outlined in the Quanterra Health and Safety Manual. These employees must have training on the hazardous waste disposal practices initially upon assignment of these tasks, followed by an annual refresher training.

6. EQUIPMENT AND SUPPLIES

6.1. Microsyringes: 10 µL and larger, 0.006 inch ID needle.

6.2. Syringe: 5 or 25 mL glass with luerlok tip, if applicable to the purging device.

6.3. Balance: Analytical, capable of accurately weighing 0.0001 g, and a top-loading balance capable of weighing 0.1 g

6.4. Glassware:

6.4.1. Vials: 20 mL with screw caps and Teflon liners.

6.4.2. Volumetric flasks: 10 mL and 100 mL, class A with ground-glass stoppers.

6.5. Spatula: Stainless steel.

6.6. Disposable pipets: Pasteur.

6.7. pH paper: Wide range.

6.8. Gases:

6.8.1. Helium: Ultra high purity, gr. 5, 99.999%.

6.8.2. Nitrogen: Ultra high purity, from cylinders of gas generators, may be used as an alternative to helium for purge gas.

6.8.3. Compressed air: Used for instrument pneumatics.

6.8.4. Liquid nitrogen: Used for cryogenic cooling if necessary.

6.9. Purge and Trap Device: The purge and trap device consists of the sample purger, the trap, and the desorber.

6.9.1. Sample Purger: The recommended purging chamber is designed to accept 5 mL samples with a water column at least 3 cm deep. The purge gas must pass through the water column as finely divided bubbles, each with a diameter of less than 3 mm at the origin. The purge gas must be introduced no more than 5 mm from the base of the water column. Alternative sample purge devices may be used provided equivalent performance is demonstrated. Low level soils are purged directly from a VOA vial.

6.9.2. Trap: A variety of traps may be used, depending on the target analytes required. For most purposes the Vocarb 3000 trap is suitable. Other traps, such as Vocarb 4000, or Tenax / Silica gel / Charcoal may be used if the Quality Control criteria are met.

6.9.3. Desorber: The desorber should be capable of rapidly heating the trap to 180°C. Many such devices are commercially available.

6.9.4. Sample Heater: A heater capable of maintaining the purge device at 40°C is necessary for low level soil analysis.

6.10. Gas Chromatograph/Mass Spectrometer System:

6.10.1. Gas Chromatograph: The gas chromatograph (GC) system must be capable of temperature programming.

6.10.2. Gas Chromatographic Columns: Capillary columns are used. Some typical columns are listed below:

6.10.2.1. Column 1: 105m x 0.53 ID Rtx-624 with 3 µm film thickness.

6.10.2.2. Column 2: 75 m x 0.53 ID DB-624 widebore with 3 µm film thickness.

6.10.2.3. Mass Spectrometer: The mass spectrometer must be capable of scanning 35-300 AMU every two seconds or less, using 70 volts electron energy in the electron impact mode and capable of producing a mass spectrum that meets the required criteria when 50 ng of 4-Bromofluorobenzene (BFB) are injected onto the gas chromatograph column inlet.

6.10.3. GC/MS interface: In general glass jet separators are used but any interface (including direct introduction to the mass spectrometer) that achieves all acceptance criteria may be used.

6.10.4. Data System: A computer system that allows the continuous acquisition and storage on machine readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer must have software that allows searching any GC/MS data file for ions of a specified mass and plotting such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). Software must also be available that allows integrating the abundances in any EICP between the specified time or scan-number limits. Also, for the non-target compounds, software must be available that allows for the comparison of sample spectra against reference library spectra. The most recent release of the NIST/EPA mass spectral library should be used as the reference library. The computer system must also be capable of backing up data for long-term off-line storage.

6.10.5. Cryogenic Cooling: Some columns require the use of liquid nitrogen to achieve the subambient temperature required for the proper separation of the gases.

7. REAGENTS AND STANDARDS

7.1. Reagents

7.1.1. Methanol: Purge and Trap Grade, High Purity

7.1.2. Reagent Water: High purity water that meets the requirements for a method blank when analyzed. (See section 9.4) Reagent water may be purchased as commercial distilled water and prepared by purging with an inert gas overnight. Other methods of preparing reagent water are acceptable.

7.2. Standards

7.2.1. Calibration Standard

7.2.1.1. Stock Solutions: Stock solutions may be purchased as certified solutions from commercial sources or prepared from pure standard materials as appropriate. These standards are prepared in methanol and stored in Teflon-sealed screw-cap bottles with minimal headspace at -10° to -20°C.

7.2.1.2. Working standards: A working solution containing the compounds of interest prepared from the stock solution(s) in methanol. These standards are stored in the freezer or as recommended by the manufacturer. Working standards are monitored by comparison to the initial calibration curve. If any of the calibration check compounds drift in response from the initial calibration by more than 20%

then corrective action is necessary. This may include steps such as instrument maintenance, preparing a new calibration verification standard or tuning the instrument. If the corrective actions do not correct the problem then a new initial calibration must be performed.

7.2.1.3. Aqueous Calibration Standards are prepared in reagent water using the secondary dilution standards. These aqueous standards must be prepared daily.

7.2.1.4. If stock or secondary dilution standards are purchased in sealed ampoules they may be used up to the manufacturers expiration date.

7.2.2. Internal Standards: Internal standards are added to all samples, standards, and blank analyses. Refer to Table 7 for internal standard components.

7.2.3. Surrogate Standards: Refer to Table 8 for surrogate standard components and spiking levels.

7.2.4. Laboratory Control Sample Spiking Solutions: Refer to Table 9 for LCS components and spiking levels.

7.2.5. Matrix Spiking Solutions: The matrix spike contains the same components as the LCS. Refer to Table 9.

7.2.6. Tuning Standard: A standard is made up that will deliver 50 ng on column upon injection. A recommended concentration of 25 ng/µL of 4-Bromofluorobenzene in methanol is prepared as described in Sections 7.2.1.1 and 7.2.1.2.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Holding times for all volatile analysis are 14 days from sample collection.

8.2. Water samples are normally preserved at pH ≤ 2 with 1:1 hydrochloric acid. If residual chlorine is present, 2 drops of 10% sodium thiosulfate are added.

8.3. Solid samples are field preserved with sodium bisulfate solution for low level analysis, or with methanol for medium level analysis. Soil samples can also be taken using the EnCore™ sampler and preserved in the lab within 48 hours of sampling. At specific client request, unpreserved soil samples may be accepted.

8.4. There are several methods of sampling soil. The recommended method, which provides the minimum of field difficulties, is to take an EnCore sample. (The 5 g or 25 g sampler can be used, depending on client preference). Following shipment back to the lab the soil is preserved in methanol. This is the medium level procedure. If very low detection limits

are needed (< 50 µg/kg for most analytes) then it will be necessary to use two additional 5 g EnCore samplers or to use field preservation.

8.5. Sample collection for medium level analysis using EnCore samplers.

8.5.1. Ship one 5 g (or 25 g) EnCore sampler per field sample position.

8.5.2. An additional bottle must be shipped for percent moisture determination.

8.5.3. When the samples are returned to the lab, extrude the (nominal) 5g (or 25 g) sample into a tared VOA vial containing 5 mL methanol (25 mL methanol for the 25 g sampler). Obtain the weight of the soil added to the vial and note on the label.

8.5.4. Add the correct amount of surrogate spiking mixture. (Add 50 uL of 1250 µg/mL solution for a nominal 25 g sample, 10µL for a nominal 5 g sample.)

8.5.5. Add the correct amount of matrix spiking solution to the matrix spike and matrix spike duplicate samples. (Add 50 uL of 1250 µg/mL solution for a nominal 25 g sample, 10µL for a nominal 5 g sample.) The addition of spike introduces a slight error (0.4%), which can be neglected, into the calculations.

8.5.6. Prepare an LCS for each batch by adding the correct amount of matrix spiking solution to clean methanol. (50 uL of spike to 25 mL methanol or 10 uL spike to 5 mL methanol).

8.5.7. Shake the samples for two minutes to distribute the methanol throughout the soil.

8.5.8. Allow to settle, then remove a portion of methanol and store in a clean Teflon capped vial at 4+2°C until analysis.

8.6. Sample collection for medium level analysis using field methanol preservation

8.6.1. Prepare a 2 oz sample container by adding 25 mL purge and trap grade methanol. (If a 5 g sample is to be used, add 5 mL methanol to a 2 oz container or VOA vial).

8.6.2. Seal the bottle and attach a label.

8.6.3. Weigh the bottle to the nearest 0.01g and note the weight on the label.

8.6.4. Ship with appropriate sampling instructions.

8.6.5. Each sample will require an additional bottle with no preservative for percent moisture determination.

8.6.6. At client request, the methanol addition and weighing may also be performed in the field.

8.6.7. When the samples are returned to the lab, obtain the weight of the soil added to the vial and note on the label.

8.6.8. Add the correct amount of surrogate spiking mixture. (Add 50 uL of 1250 µg/mL solution for a nominal 25 g sample, 10µL for a nominal 5 g sample.)

8.6.9. Add the correct amount of matrix spiking solution to the matrix spike and matrix spike duplicate samples. (Add 50 uL of 1250 µg/mL solution for a nominal 25 g sample, 10µL for a nominal 5 g sample.) The addition of spike introduces a slight error, (0.2%) which can be neglected, into the calculations.

8.6.10. Prepare an LCS for each batch by adding the correct amount of matrix spiking solution to clean methanol. (50 uL of spike to 25 mL methanol or 10 uL spike to 5 mL methanol).

8.6.11. Shake the samples for two minutes to distribute the methanol throughout the soil.

8.6.12. Allow to settle, then remove a portion of methanol and store in a clean Teflon capped vial at 4+2°C until analysis.

8.7. Low level procedure

8.7.1. If low detection limits are required (typically < 50 µg/kg) sodium bisulfate preservation must be used. However, it is also necessary to take a sample for the medium level (field methanol preserved or using the EnCore sampler) procedure, in case the concentration of analytes in the soil is above the calibration range of the low level procedure.

8.7.2. A purge and trap autosampler capable of sampling from a sealed vial is required for analysis of samples collected using this method. (Varian Archon or O.I. 4552).

8.7.3. The soil sample is taken using a 5g EnCore sampling device and returned to the lab. It is recommended that two EnCore samplers be used for each field sample position, to allow for any reruns than may be necessary. A separate sample for % moisture determination is also necessary.

8.7.4. Prepare VOA vials by adding a magnetic stir bar, approximately 1 g of sodium bisulfate and 5 mL of reagent water.

8.7.5. Seal and label the vial. It is strongly recommended that the vial is labeled with an indelible marker rather than a paper label, since paper labels may cause the autosampler to bind and malfunction. The label absolutely must not cover the neck of the vial or the autosampler will malfunction.

8.7.6. Weigh the vial to the nearest 0.1g and note the weight on the label.

8.7.7. Extrude the soil sample from the EnCore sampler into the prepared VOA vial. Reweigh the vial to obtain the weight of soil and note on the label.

8.7.8. **Note:** Soils containing carbonates may effervesce when added to the sodium bisulfate solution. If this is the case at a specific site, add 5 mL of water instead, and freeze at <10°C until analysis.

8.7.9. Alternatively the sodium bisulfate preservation may be performed in the field. This is not recommended because of the many problems that can occur in the field setting. Ship at least two vials per sample. The field samplers must determine the weight of soil sampled. Each sample will require an additional bottle with no preservative for percent moisture determination, and an additional bottle preserved with methanol for the medium level procedure. Depending on the type of soil it may also be necessary to ship vials with no or extra preservative.

8.8. Unpreserved soils

8.8.1. At specific client request unpreserved soils packed into glass jars or brass tubes may be accepted and subsampled in the lab. This is the old procedure based on method 5030A. It is no longer included and is likely to generate results that are biased low, possibly be more than an order of magnitude.

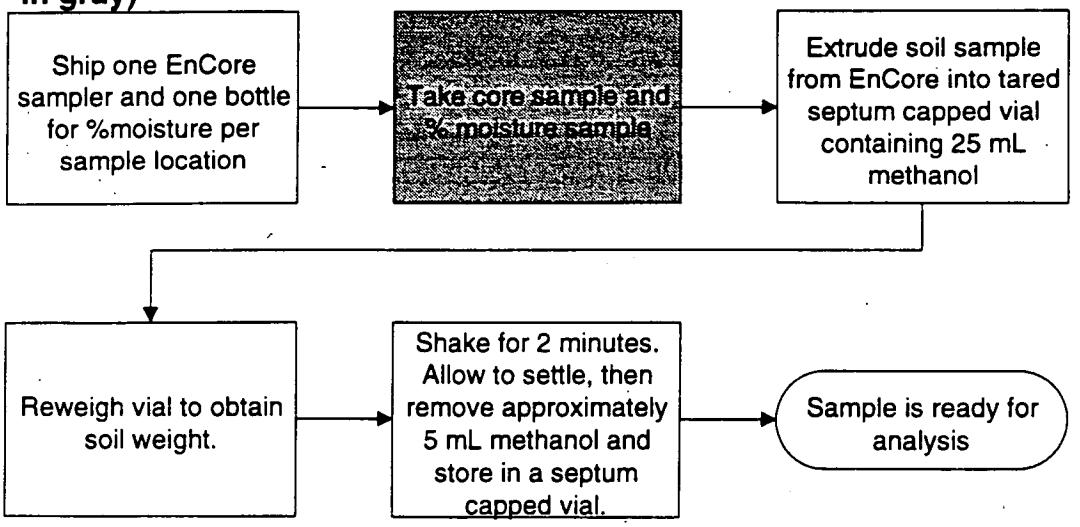
8.9. Aqueous samples are stored in glass containers with Teflon lined septa at 4°C +/- 2°C, with minimum headspace.

8.10. Medium level solid extracts are aliquoted into 2 - 5 mL glass vials with Teflon lined caps and stored at 4°C +/- 2°C. The extracts are stored with minimum headspace.

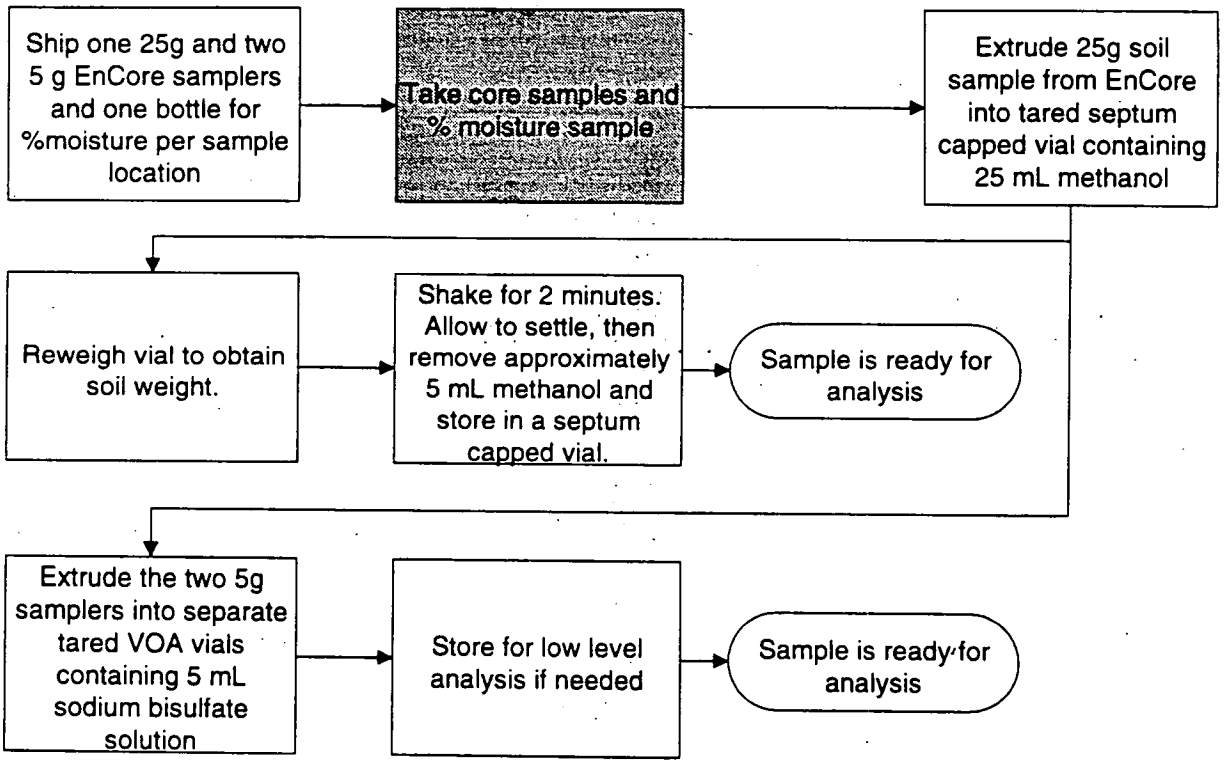
8.11. The maximum holding time is 14 days from sampling until the sample is analyzed. (Samples that are found to be unpreserved still have a 14 day holding time. However they should be analyzed as soon as possible. The lack of preservation should be addressed in the case narrative). Maximum holding time for the EnCore sampler (before the sample is added to methanol or sodium bisulfate) is 48 hours.

8.12. A holding blank is stored with the samples. This is analyzed and replaced if any of the trip blanks show any contamination. Otherwise it is replaced every 14 days.

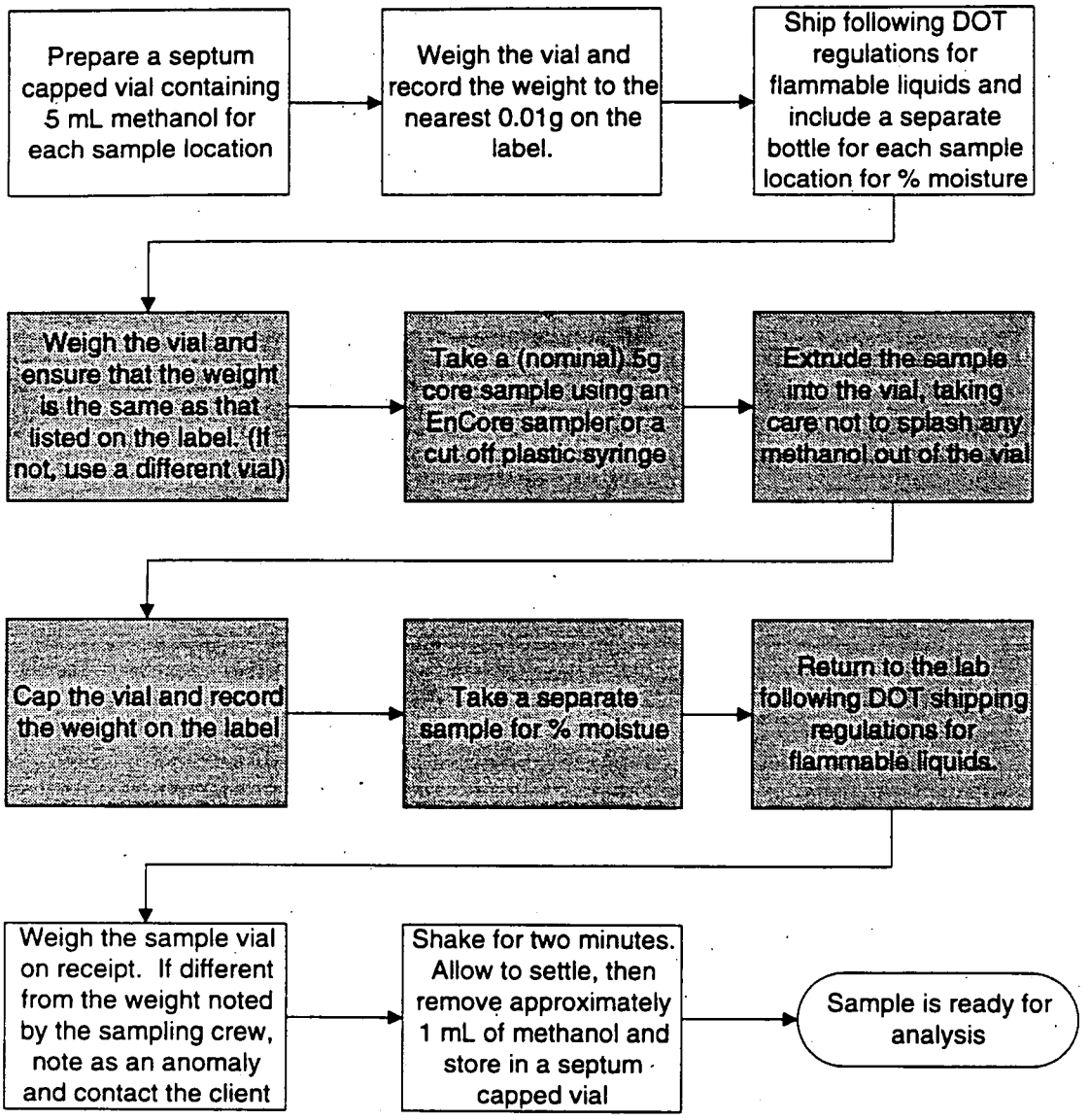
EnCore procedure when low level is not required (field steps in gray)



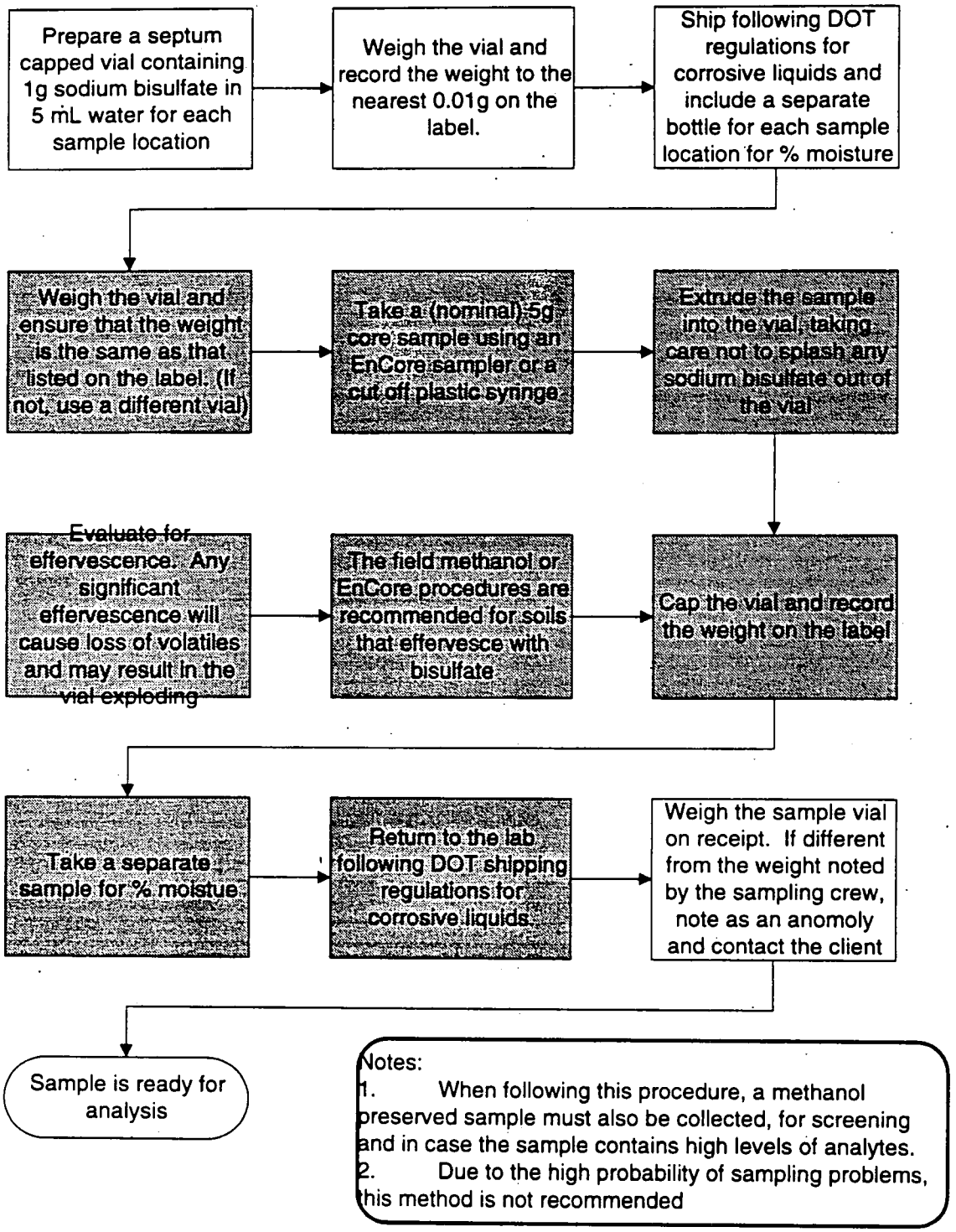
EnCore procedure when low level is required



Field methanol extraction procedure (field steps in gray)



Field bisulfate preservation procedure (field steps in gray)



Notes:
1. When following this procedure, a methanol preserved sample must also be collected, for screening and in case the sample contains high levels of analytes.
2. Due to the high probability of sampling problems, this method is not recommended

9. QUALITY CONTROL

9.1. Initial Demonstration of Capability

9.1.1. For the standard analyte list, the initial demonstration described in Section 13 and method detection limit (MDL) studies must be acceptable before analysis of samples may begin. MDLs should be analyzed for low and medium soils and aqueous samples.

9.1.2. For non-standard analytes, a MDL study must be performed and calibration curve generated before analyzing any samples, unless lesser requirements are previously agreed to with the client. In any event, the minimum initial demonstration required is analysis of a standard at the reporting limit and a single point calibration.

9.2. Control Limits

In-house historical control limits must be determined for surrogates, matrix spikes, and laboratory control samples (LCS). These limits must be determined at least annually. The recovery limits are mean recovery +/- 3 standard deviations for surrogates, matrix spikes and LCS. Precision limits for matrix spikes / matrix spike duplicates are 0 to mean relative percent difference + 3 standard deviations.

9.2.1. All surrogate, LCS, and MS recoveries (except for dilutions) must be entered into QuantIMS (when available) or other database so that accurate historical control limits can be generated. For tests without a separate extraction, surrogates and matrix spikes will be reported for all dilutions.

9.2.2. Refer to the QC Program document (QA-003) for further details of control limits.

9.3. Surrogates

Every sample, blank, and QC sample is spiked with surrogates. Surrogate recoveries in samples, blanks, and QC samples must be assessed to ensure that recoveries are within established limits. The compounds included in the surrogate spiking solutions are listed in Tables 8 and 16. If any surrogates are outside limits, the following corrective actions must take place (except for dilutions):

- Check all calculations for error.
- Ensure that instrument performance is acceptable.
- Recalculate the data and/or reanalyze if either of the above checks reveal a problem.

- Reprepare and reanalyze the sample or flag the data as "Estimated Concentration" if neither of the above resolves the problem.

The decision to reanalyze or flag the data should be made in consultation with the client. It is only necessary to reprepare/reanalyze a sample once to demonstrate that poor surrogate recovery is due to matrix effect, unless the analyst believes that the repeated out of control results are not due to matrix effect.

9.3.1. If the surrogates are out of control for the sample, matrix spike, and matrix spike duplicate, then matrix effect has been demonstrated for that sample and repreparation is not necessary. If the sample is out of control and the MS and/or MSD is in control, then reanalysis or flagging of the data is required.

9.3.2. Refer to the Quanterra QC Program document (QA-003) for further details of the corrective actions.

9.4. Method Blanks

For each batch of samples, analyze a method blank. The method blank is analyzed after the calibration standards, normally before any samples. For low-level volatiles, the method blank consists of reagent water. For medium-level volatiles, the method blank consists of 25.0 mL of methanol. Surrogates are added and the method blank is carried through the entire analytical procedure. The method blank must not contain any analyte of interest at or above the reporting limit (except common laboratory contaminants, see below) or at or above 5% of the measured concentration of that analyte in the associated samples, whichever is higher.

- If the analyte is a common laboratory contaminant (methylene chloride, acetone, 2-butanone) the data may be reported with qualifiers if the concentration of the analyte is less than five times the reporting limit. Such action must be taken in consultation with the client.

- Reanalysis of samples associated with an unacceptable method blank is required when reportable concentrations are determined in the samples.

- If there is no target analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. Such action should be done in consultation with the client.

9.4.1. The method blank must have acceptable surrogate recoveries. If surrogate recoveries are not acceptable, the data must be evaluated to determine if the method blank has served the purpose of demonstrating that the analysis is free of contamination. If surrogate recoveries are low and there are reportable analytes in the associated

samples re-extraction of the blank and affected samples will normally be required. Consultation with the client should take place.

9.4.2. If reanalysis of the batch is not possible due to limited sample volume or other constraints, the method blank is reported, all associated samples are flagged with a "B," and appropriate comments may be made in a narrative to provide further documentation.

9.4.3. Refer to the Quanterra QC Program document (QA-003) for further details of the corrective actions.

9.5. Laboratory Control Samples (LCS)

For each batch of samples, analyze a LCS. The LCS is analyzed after the calibration standard, and normally before any samples. The LCS contains a representative subset of the analytes of interest (See Table 9), and must contain the same analytes as the matrix spike. If any analyte or surrogate is outside established control limits, the system is out of control and corrective action must occur. Corrective action will normally be repreparation and reanalysis of the batch.

- If the batch is not re-extracted and reanalyzed, the reasons for accepting the batch must be clearly presented in the project records and the report. (Examples of acceptable reasons for not reanalyzing might be that the matrix spike and matrix spike duplicate are acceptable, and sample surrogate recoveries are good, demonstrating that the problem was confined to the LCS.)

- If re-extraction and reanalysis of the batch is not possible due to limited sample volume or other constraints, the LCS is reported, all associated samples are flagged, and appropriate comments are made in a narrative to provide further documentation.

9.5.1. Refer to the Quanterra QC Program document (QA-003) for further details of the corrective action.

9.5.2. If full analyte spike lists are used at client request, it will be necessary to allow a percentage of the components to be outside control limits as this would be expected statistically. These requirements should be negotiated with the client.

9.6. Matrix Spikes

For each QC batch, analyze a matrix spike and matrix spike duplicate. Spiking compounds and levels are given in Table 9. Compare the percent recovery and relative percent difference (RPD) to that in the laboratory specific historically generated limits.

- If any individual recovery or RPD falls outside the acceptable range, corrective action must occur. The initial corrective action will be to check the recovery of that analyte in the Laboratory Control Sample (LCS). Generally, if the recovery of the analyte in the LCS is within limits, then the laboratory operation is in control and analysis may proceed. The reasons for accepting the batch must be documented.

- If the recovery for any component is outside QC limits for both the matrix spike/ spike duplicate and the LCS, the laboratory is out of control and corrective action must be taken. Corrective action will normally include reanalysis of the batch.

- If a MS/MSD is not possible due to limited sample, then a LCS duplicate should be analyzed. RPD of the LCS and LCSD are compared to the matrix spike limits.

- The matrix spike/duplicate must be analyzed at the same dilution as the unspiked sample, even if the matrix spike compounds will be diluted out.

9.7. Nonconformance and Corrective Action

Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the facility QA Manager.

9.8. Quality Assurance Summaries

Certain clients may require specific project or program QC which may supersede these method requirements. Quality Assurance Summaries should be developed to address these requirements.

9.9. Quanterra QC Program

Further details of QC and corrective action guidelines are presented in the Quanterra QC Program document (QA-003). Refer to this document if in doubt regarding corrective actions.

10. CALIBRATION AND STANDARDIZATION

10.1. Summary

10.1.1. Prior to the analysis of samples and blanks, each GC/MS system must be tuned and calibrated. Hardware tuning is checked through the analysis of the 4-Bromofluorobenzene (BFB) to establish that a given GC/MS system meets the standard mass spectral abundance criteria. The GC/MS system must be calibrated initially at a minimum of five concentrations (analyzed under the same BFB tune), to determine the linearity of the response utilizing target calibration standards. Once the system has been calibrated, the calibration must be verified each twelve hour time period for each

GC/MS system. The use of separate calibrations is required for water and low soil matrices.

10.2. Recommended Instrument Conditions

10.2.1. General

Electron Energy:	70 volts (nominal)
Mass Range:	35–300 AMU
Scan Time:	to give at least 5 scans/peak, but not to exceed 2 second/scan
Injector Temperature:	200–250°C
Source Temperature:	According to manufacturer's specifications
Transfer Line	Temperature: 250–300°C
Purge Flow:	40 mL/minute
Carrier Gas	Flow: 15 mL/minute
Make-up Gas Flow:	25–30 mL/minute

10.2.2. Gas chromatograph suggested temperature program

10.2.2.1. BFB Analysis

Isothermal:	170°C

10.2.2.2. Sample Analysis

Initial Temperature:	40°C
Initial Hold Time:	4 minutes
Temperature Program:	8°C/minute
Final Temperature:	184°C
Second Temperature	Program: 40°C/minute
Final Temperature:	240°C
Final Hold Time:	2.6 minutes

10.3. Instrument Tuning

10.3.1. Each GC/MS system must be hardware-tuned to meet the abundance criteria listed in Table 10 for a maximum of a 50 ng injection or purging of BFB. Analysis must not begin until these criteria are met. These criteria must be met for each twelve-hour time period. The twelve-hour time period begins at the moment of injection of BFB.

10.4. Initial Calibration

10.4.1. A series of five initial calibration standards is prepared and analyzed for the target compounds and each surrogate compound. Typical calibration levels for a 5 mL purge are: 5, 20, 50, 100, and 200 µg/L. Certain analytes are prepared at higher concentrations due to poor purge performance. Typical calibration levels for a 25 mL purge are 1, 5, 10, 30, and 60 µg/L. Again, some analytes are prepared at higher levels. Tables 2, 4, and 17 list the calibration levels for each analyte. Other calibration levels and purge volumes may be used depending on the capabilities of the specific instrument. However, the same purge volume must be used for calibration and sample analysis, and the low level standard must be at or below the reporting limit.

10.4.2. It may be necessary to analyze more than one set of calibration standards to encompass all of the analytes required for same tests. For example, the Appendix IX list requires the Primary standard (Table 5) and the Appendix IX standard (Table 6). If acceptable analytical performance can be obtained the primary and appendix IX standards may be analyzed together.

10.4.3. Internal standard calibration is used. The internal standards are listed in Tables 7 and 15. Target compounds should reference the nearest internal standard. Each calibration standard is analyzed and the response factor (RF) for each compound is calculated using the area response of the characteristic ions against the concentration for each compound and internal standard. See equation 1, Section 12, for calculation of response factor.

10.4.4. The % RSD of the calibration check compounds (CCC) must be less than 30%. Refer to Table 12 for the CCCs.

10.4.4.1. If none of the CCCs are required analytes, project specific calibration specifications must be agreed with the client.

10.4.5. The average RF must be calculated for each compound. A system performance check is made prior to using the calibration curve. The five system performance check compounds (SPCC) are checked for a minimum average response factor. Refer to Table 11 for the SPCC compounds and required minimum response factors.

10.4.6. If the average of all the %RSDs in the calibration is $\leq$ 15%, then all analytes may use average response factor for calibration.

10.4.6.1. If the software in use is capable of routinely reporting curve coefficients for data validation purposes, and the necessary calibration reports can be generated, then the analyst should evaluate analytes with %RSD > 15% for calibration on a curve. If it appears that substantially better accuracy would be obtained using quantitation from a curve then the appropriate curve should be

used for quantitation. If Relative Standard Error (RSE) is used to evaluate the curve it must be better than 15%. Otherwise the correlation coefficient (coefficient of determination for non-linear curves) must be $\geq$ 0.990.

10.4.6.2. If the average of all the %RSDs in the calibration is > 15% then calibration on a curve must be used for all analytes with %RSD > 15%. The analyst should consider instrument maintenance to improve the linearity of response. If Relative Standard Error (RSE) is used to evaluate the curve it must be better than 15%. Otherwise the correlation coefficient, r (coefficient of determination, r^2 for non-linear curves) must be $\geq$ 0.990.

10.4.7. Weighting of data points

In a linear or quadratic calibration fit, the points at the lower end of the calibration curve have less weight in determining the curve generated than points at the high concentration end of the curve. However, in environmental analysis, accuracy at the low end of the curve is very important. For this reason it is preferable to increase the weighting of the lower concentration points. 1/Concentration^2 weighting (often called $1/X^2$ weighting) will improve accuracy at the low end of the curve and should be used if the data system has this capability.

10.4.8. If time remains in the 12-hour period initiated by the BFB injection before the initial calibration, samples may be analyzed. Otherwise, proceed to continuing calibration.

10.4.9. A separate five point calibration must be prepared for analysis of low level soils. Low level soils analysis requires the use of a closed vial autosampler such as the Varian Archon, O.I. 4552 or Tekmar Precept. Each standard is prepared by spiking the methanolic standard solution through the septum of a VOA vial containing 5 mL of water and 1 g sodium bisulfate. The standards are heated to 40°C for purging. All low-level soil samples, standards, and blanks must also be heated to 40°C for purging. Medium soil extracts should be analyzed using the water calibration curve.

10.4.10. Non-standard analytes are sometimes requested. For these analytes, it is acceptable to analyze a single standard at the reporting limit with each continuing calibration rather than a five point initial calibration. If the analyte is detected in any of the samples, a five point initial calibration must be generated and the sample(s) reanalyzed for quantitation. However, if the analyte is not detected, the non-detect may be reported and no further action is necessary.

10.5. Continuing Calibration: The initial calibration must be verified every twelve hours.

10.5.1. Continuing calibration begins with analysis of BFB as described in Section 10.3. If the system tune is acceptable, the continuing calibration standard(s) are analyzed. The level 3 calibration standard is used as the continuing calibration.

10.5.2. The RF data from the standards are compared with the average RF from the initial five-point calibration to determine the percent drift of the CCC compounds. The calculation is given in equation 4, Section 12.3.4.

10.5.3. The % drift of the CCCs must be ≤ 20% for the continuing calibration to be valid. The SPCCs are also monitored. The SPCCs must meet the criteria described in Table 11. In addition, the % drift of all analytes must be ≤ 50% with allowance for up to six target analytes to have % drift > 50%.

10.5.3.1. If none of the CCCs are required analytes, project specific calibration specifications must be agreed with the client.

10.5.3.2. Cyclohexanone, one of the components of the Appendix IX standard, is unstable in the calibration solution, forming 1,1-dimethoxycyclohexane. No calibration criteria are applied to cyclohexanone and quantitation is tentative. Cyclohexanone is included on the Universal Treatment Standard and FO-39 regulatory lists (but not on Appendix IX).

10.5.4. If the CCCs and or the SPCCs do not meet the criteria in Sections 10.5.3 and 10.5.4, the system must be evaluated and corrective action must be taken. The BFB tune and continuing calibration must be acceptable before analysis begins. Extensive corrective action such as a different type of column will require a new initial calibration.

10.5.5. Once the above criteria have been met, sample analysis may begin. **Initial calibration average RFs (or the calibration curve) will be used for sample quantitation, not the continuing calibration RFs.** Analysis may proceed until 12 hours from the injection of the BFB have passed. (A sample *desorbed* less than or equal to 12 hours after the BFB is acceptable.)

11. PROCEDURE

11.1. Procedural Variations

11.1.1. One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation shall be completely documented using a Nonconformance Memo and approved by a Supervisor or group leader and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

11.1.2. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

11.2. Preliminary Evaluation

11.2.1. Where possible, samples are screened by headspace or GC/MS off-tune analysis to determine the correct aliquot for analysis. Alternatively, an appropriate aliquot can be determined from sample histories.

11.2.2. Dilutions should be done just prior to the GC/MS analysis of the sample. Dilutions are made in volumetric flasks or in a Luerlok syringe. Calculate the volume of reagent water required for the dilution. Fill the syringe with reagent water, compress the water to vent any residual air and adjust the water volume to the desired amount. Adjust the plunger to the mark and inject the proper aliquot of sample into the syringe. If the dilution required would use less than 1 µL of sample then serial dilutions must be made in volumetric flasks.

11.2.2.1. The diluted concentration is to be estimated to be in the upper half of the calibration range.

11.3. Sample Analysis Procedure

11.3.1. All analysis conditions for samples must be the same as for the continuing calibration standards (including purge time and flow, desorb time and temperature, column temperatures, multiplier setting etc.).

11.3.2. All samples must be analyzed as part of a batch. The batch is a set of up to 20 samples of the same matrix processed using the same procedures and reagents within the same time period. The batch also must contain a MS/MSD, a LCS, and a method blank.

11.3.2.1. If there is insufficient time in the 12-hour tune period to analyze 20 samples, the batch may be continued into the next tune period. However, if any re-tuning of the instrument is necessary, or if a period of greater than 24 hours from the preceding BFB tune has passed, a new batch must be started. For medium level soils the batch is defined at the sample preparation stage.

11.3.2.2. Laboratory generated QC samples (Blank, LCS, MS/MSD) do not count towards the maximum 20 samples in a batch. Field QC samples are included in the batch count.

11.3.2.3. It is not necessary to reanalyze batch QC with reanalyses of samples. However, any reruns must be as part of a valid batch.

11.4. Water Samples

11.4.1. All samples and standard solutions must be at ambient temperature before analysis.

11.4.2. Fill a syringe with the sample. If a dilution is necessary it may be made in the syringe if the sample aliquot is ≥ 5 µL. Check and document the pH of the remaining sample.

11.4.3. Add 250 ng of each internal and surrogate standard (10 µL of a 25 µg/mL solution, refer to Tables 7, 8, 15 and 16). The internal standards and the surrogate standards may be mixed and added as one spiking solution (this results in a 50 µg/L solution for a 5 mL sample, and a 10 µg/L solution for a 25 mL sample). Inject the sample into the purging chamber.

11.4.3.1. For TCLP samples use 0.5 mL of TCLP leachate with 4.5 mL reagent water and spike with 10 µL of the 25 µg/mL TCLP spiking solution. (Note that TCLP reporting limits will be 10 times higher than the corresponding aqueous limits).

11.4.4. Purge the sample for eleven minutes (the trap must be below 35°C).

11.4.5. After purging is complete, desorb the sample, start the GC temperature program, and begin data acquisition. After desorption, bake the trap for 5-10 minutes to condition it for the next analysis. When the trap is cool, it is ready for the next sample.

11.4.6. Desorb and bake time and temperature are optimized for the type of trap in use. The same conditions must be used for samples and standards.

11.5. Methanol Extract Soils

11.5.1. Rinse a gas-tight syringe with organic free water. Fill the syringe with the same volume of organic free water as used in the calibrations. Add no more than 2% (v/v) (100 µL for a 5 mL purge) methanolic extract (from Section 8.5 or 8.6) to the syringe. Add internal standard (if used). Load the sample onto the purge and trap device and analyze as for aqueous samples. If less than 5µL of methanolic extract is to be added to the water, dilute the methanolic extract such that a volume greater than 5µL will be added to the water in the syringe.

11.6. Liquid wastes that are soluble in methanol and insoluble in water.

11.6.1. Pipet 2 mL of the sample into a tared vial. Use a top-loading balance. Record the weight to the nearest 0.1 gram.

11.6.2. Quickly add 7 mL of methanol, then add 1 mL of surrogate spiking solution to bring the final volume to 10 mL. Cap the vial and shake for 2 minutes to mix thoroughly. For a

MS/MSD or LCS, 6 mL of methanol, 1 mL of surrogate solution, and 1 mL of matrix spike solution is used.

11.6.3. Rinse a gas-tight syringe with organic free water. Fill the syringe with the same volume of organic free water as used in the calibrations. Add no more than 2% (v/v) (100 µL for a 5 mL purge) methanolic extract (from Section 8.5 or 8.6) to the syringe. Add internal standard (if used). Load the sample onto the purge and trap device and analyze as for aqueous samples. If less than 5µL of methanolic extract is to be added to the water, dilute the methanolic extract such that a volume greater than 5µL will be added to the water in the syringe.

11.7. Aqueous and Low level Soil Sample Analysis (Purge and Trap units that sample directly from the VOA vial)

11.7.1. Units which sample from the VOA vial should be equipped with a module which automatically adds surrogate and internal standard solution to the sample prior to purging the sample.

11.7.2. If the autosampler uses automatic IS/SS injection, no further preparation of the VOA vial is needed. Otherwise the internal and surrogate standards must be added to the vial. *Note:* Aqueous samples with high amounts of sediment present in the vial may not be suitable for analysis on this instrumentation, or they may need to analyzed as soils.

11.7.3. Soil samples must be quantitated against a curve prepared with standards containing about the same amount of sodium bisulfate as the samples (1 g in 5 mL).

11.7.4. Sample remaining in the vial after sampling with one of these mechanisms is no longer valid for further analysis. A fresh VOA vial must be used for further sample analysis.

11.7.5. For aqueous samples, check the pH of the sample remaining in the VOA vial after analysis is completed.

11.8. Low-Level Solids Analysis using discrete autosamplers

Note: This technique may seriously underestimate analyte concentration and must not be used except at specific client request for the purpose of comparability with previous data. It is no longer part of SW-846.

This method is based on purging a heated sediment/soil sample mixed with sodium bisulfate solution containing the surrogate and, if applicable, internal and matrix spiking standards. Analyze all reagent blanks and standards under the same conditions as the samples (e.g., heated). The calibration curve is also heated during analysis. Purge temperature is 40°C.

11.8.1. Do not discard any supernatant liquids. Mix the contents of the container with a narrow metal spatula.

11.8.2. Weigh out 5 g (or other appropriate aliquot) of sample into a disposable culture tube or other purge vessel. Record the weight to the nearest 0.1 g. If method sensitivity is demonstrated, a smaller aliquot may be used. Do not use aliquots less than 1.0 g. If the sample is contaminated with analytes such that a purge amount less than 1.0 g is appropriate, use the medium level method described in section 11.7.

11.8.3. Connect the purge vessel to the purge and trap device.

11.8.4. Rinse a 5 mL gas-tight syringe with organic free water, and fill. Compress to 5 mL. Add surrogate/internal standard (and matrix spike solutions if required.). Add directly to the sample from 11.5.2.

11.8.5. The above steps should be performed rapidly and without interruption to avoid loss of volatile organics.

11.8.6. Add the heater jacket or other heating device and start the purge and trap unit.

11.8.7. Soil samples that have low IS recovery when analyzed (<50%) should be reanalyzed once to confirm matrix effect.

11.9. Initial review and corrective actions

11.9.1. If the retention time for any internal standard in the continuing calibration changes by more than 0.5 minutes from the mid-level initial calibration standard, the chromatographic system must be inspected for malfunctions and corrected. Reanalysis of samples analyzed while the system was malfunctioning is required.

11.9.2. If the internal standard response in the continuing calibration is more than 200% or less than 50% of the response in the mid-level of the initial calibration standard, the chromatographic system must be inspected for malfunctions and corrected. Reanalysis of samples analyzed while the system was malfunctioning is required.

11.9.2.1. Any samples that do not meet the internal standard criteria for the continuing calibration must be evaluated for validity. If the change in sensitivity is a matrix effect confined to an individual sample reanalysis is not necessary. If the change in sensitivity is due to instrumental problems all affected samples must be reanalyzed after the problem is corrected.

11.9.3. The surrogate standard recoveries are evaluated to ensure that they are within limits. Corrective action for surrogates out of control will normally be to reanalyze the affected

samples. However, if the surrogate standard response is out high and there are no target analytes or tentatively identified compounds, reanalysis may not be necessary. Out of control surrogate standard response may be a matrix effect. It is only necessary to reanalyze a sample once to demonstrate matrix effect, but reanalysis at a dilution should be considered.

11.10. Dilutions

If the response for any compound exceeds the working range of the GC/MS system, a dilution of the extract is prepared and analyzed. An appropriate dilution should be in the upper half of the calibration range. Samples may be screened to determine the appropriate dilution for the initial run. If the initial diluted run has no hits or hits below 20% of the calibration range and the matrix allows for analysis at a lesser dilution, then the sample must be reanalyzed at a dilution targeted to bring the largest hit above 50% of the calibration range.

11.10.1.Guidance for Dilutions Due to Matrix

If the sample is initially run at a dilution and the baseline rise is less than half the height of the internal standards, or if individual non target peaks are less than twice the height of the internal standards, then the sample should be reanalyzed at a more concentrated dilution. This requirement is approximate and subject to analyst judgement.

11.10.2.Reporting Dilutions

The most concentrated dilution with no target compounds above the calibration range will be reported. Other dilutions will only be reported at client request.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Qualitative identification

An analyte is identified by retention time and by comparison of the sample mass spectrum with the mass spectrum of a standard of the suspected compound (standard reference spectrum). Mass spectra for standard reference may be obtained on the user's GC/MS by analysis of the calibration standards or from the NIST Library. Two criteria must be satisfied to verify identification: (1) elution of sample component at the same GC retention time as the standard component; and (2) correspondence of the sample component and the standard component characteristic ions. (Note: Care must be taken to ensure that spectral distortion due to co-elution is evaluated.)

- The sample component retention time must compare to within ± 0.2 min. of the retention time of the standard component. For reference, the standard must be run within the same twelve hours as the sample.

- All ions present in the standard mass spectra at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) should be present in the sample spectrum.

- The relative intensities of ions should agree to within ±30% between the standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample abundance must be between 20 and 80 percent.)

12.1.1. If a compound cannot be verified by all the above criteria, but in the technical judgment of the analyst, the identification is correct, then the analyst shall report that identification and proceed with quantitation.

12.2. Tentatively Identified Compounds (TICs)

12.2.1. If the client requests components not associated with the calibration standards, a search of the NIST library may be made for the purpose of tentative identification. Guidelines are:

12.2.1.1. Relative intensities of major ions in the reference spectrum (ions > 10% of the most abundant ion) should be present in the sample spectrum.

12.2.1.2. The relative intensities of the major ions should agree to within 20%. (Example: If an ion shows an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30% and 70%).

12.2.1.3. Molecular ions present in the reference spectrum should be present in the sample spectrum.

12.2.1.4. Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of coeluting compounds.

12.2.1.5. Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the spectrum because of background contamination or coeluting peaks. (Data system reduction programs can sometimes create these discrepancies.)

12.2.1.6. Computer-generated library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. Only after visual inspection of the sample with the nearest library searches should the analyst assign a tentative identification.

12.3. Calculations.

12.3.1. Response factor (RF):

Equation 1

$$RF = \frac{A_x C_{is}}{A_{is} C_x}$$

Where:

A_x = Area of the characteristic ion for the compound to be measured

A_{is} = Area of the characteristic ion for the specific internal standard

C_{is} = Concentration of the specific internal standard, ng

C_x = Concentration of the compound being measured, ng

12.3.2. Standard deviation (SD):

Equation 2

$$SD = \sqrt{\sum_{i=1}^{N} \frac{(Xi - X)^2}{N - 1}}$$

X_i = Value of X at i through N

N = Number of points

X = Average value of X_i

12.3.3. Percent relative standard deviation (%RSD):

Equation 3

$$\%RSD = \frac{\text{Standard Deviation}}{\overline{RF_i}} \times 100$$

$\overline{RF_i}$ = **Mean of RF values in the curve**

12.3.4. Percent drift between the initial calibration and the continuing calibration:

Equation 4

$$\% \text{ Drift} = \frac{C_{expected} - C_{found}}{C_{expected}} \times 100$$

Where

$C_{expected}$ = Known concentration in standard

C_{found} = Measured concentration using selected quantitation method

12.3.5. Target compound and surrogate concentrations:

Concentrations in the sample may be determined from linear or second order (quadratic) curve fitted to the initial calibration points, or from the average response factor of the initial calibration points. Average response factor may only be used when the % RSD of the response factors in the initial calibration is $\leq$ 15%.

12.3.5.1. Calculation of concentration using Average Response Factors

Equation 5

$$\text{Concentration } \mu g / L = \frac{x}{RF}$$

12.3.5.2. Calculation of concentration using Linear fit

Equation 6

$$\text{Concentration } \mu g / L = A + Bx$$

12.3.5.3. Calculation of concentration using Quadratic fit

Equation 7

$$\text{Concentration } \mu g / L = A + Bx + Cx^2$$

x is defined in equations 8, 9 and 10

A is a constant defined by the intercept

B is the slope of the curve

C is the curvature

12.3.5.4. Calculation of x for Water and water-miscible waste:

Equation 8

$$x = \frac{(A_x)(I_s)(D_f)}{(A_{is})(V_o)}$$

Where:

$\mathbf{A_x}$ = Area of characteristic ion for the compound being measured (secondary ion quantitation is allowed only when there are sample interferences with the primary ion)

$\mathbf{A_{is}}$ = Area of the characteristic ion for the internal standard

$\mathbf{I_s}$ = Amount of internal standard added in ng

$$\text{Dilution Factor} = D_f = \frac{\text{Total volume purged (mL)}}{\text{Volume of original sample used (mL)}}$$

$\mathbf{V_o}$ = Volume of water purged, mL

12.3.5.5. Calculation of x for Medium level soils:

Equation 9

$$x = \frac{(A_x)(I_s)(V_t)(1000)(D_f)}{(A_{is})(V_a)(W_s)(D)}$$

Where:

$\mathbf{A_x}$, $\mathbf{I_s}$, $\mathbf{D_f}$, $\mathbf{A_{is}}$, same as for water.

$\mathbf{V_t}$ = Volume of total extract, mL (Typically 25 mL)

$\mathbf{V_a}$ = Volume of extract added for purging, µL

$\mathbf{W_s}$ = Weight of sample extracted, g

$$\mathbf{D} = \frac{100 - \%\text{moisture}}{100}$$

12.3.5.6. Calculation of x for Low level soils:

Equation 10

$$x = \frac{(A_x)(I_s)}{(A_{is})(W_s)(D)}$$

Where:

A_x, I_s, A_{is}, same as for water.

D is as for medium level soils

W_s = Weight of sample added to the purge vessel, g

12.3.5.7. Calculation of TICs: The calculation of TICs (tentatively identified compounds) is identical to the above calculations with the following exceptions:

A_x = Area in the total ion chromatogram for the compound being measured

A_{is} = Area of the total ion chromatogram for the nearest internal standard without interference

RF = 1

In other words, the concentration is equal to x as defined in equations 8, 9 and 10.

12.3.6. MS/MSD Recovery

Equation 11

$$\textbf{Matrix Spike Recovery, } \% = \frac{SSR - SR}{SA} \times 100$$

SSR = Spike sample result
SR = Sample result
SA = Spike added

12.3.7. Relative % Difference calculation for the MS/MSD

Equation 12

$$\mathbf{RPD} = \frac{|\mathbf{MSR} - \mathbf{MSDR}|}{\frac{1}{2}(\mathbf{MSR} + \mathbf{MSDR})} \times 100$$

Where:

RPD = Relative percent difference

MSR = Matrix spike result

MSDR = Matrix spike duplicate result

13. METHOD PERFORMANCE

13.1. Method Detection Limit

Generally, each laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in QA Policy #: QA-005. When non-standard compounds are analyzed at client request, lesser requirements are possible with client agreement. At a minimum, a standard at the reporting limit must be analyzed to demonstrate the capability of the method.

13.2. Initial Demonstration

Each laboratory must make a one time initial demonstration of capability for each individual method. Demonstration of capability for both soil and water matrices is required. This requires analysis of QC check samples containing all of the standard analytes for the method. For some tests it may be necessary to use more than one QC check mix to cover all analytes of interest. The QC check sample is made up at 20 µg/L. (Some compounds will be at higher levels, refer to the calibration standard levels for guidance.)

13.2.1. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation.

13.2.2. Calculate the average recovery and standard deviation of the recovery for each analyte of interest. The %RSD should be $\leq$ 15% for each analyte, and the % recovery should be within 80-120%.

13.2.3. If any analyte does not meet the acceptance criteria, check the acceptance limits in the reference methods (Table 6 of method 8240B, paragraph 8.3.5 of method 8260A). If the recovery or precision is outside the limits in the reference methods, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

13.3. Training Qualification

The group/team leader has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

14. POLLUTION PREVENTION

14.1. This method does not contain any specific modifications that serve to minimize or prevent pollution.

15. WASTE MANAGEMENT

15.1. Waste generated in the procedure must be segregated and disposed according to the facility hazardous waste procedures. The Health and Safety Director should be contacted if additional information is required.

16. REFERENCES

16.1. SW846, *Test Methods for Evaluating Solid Waste,* Third Edition, Gas Chromatography/Mass Spectrometry for Volatile Organics, Method 8260B, Update III, December 1996

17. MISCELLANEOUS

17.1. Modifications from the reference method

17.1.1. Ion 119 is used as the quantitation ion for chlorobenzene-d5 for 25 mL purge tests.

17.1.2. A retention time window of 0.2 minutes is used for all components, since some data systems do not have the capability of using the relative retention time units specified in the reference method.

17.1.3. The quantitation and qualifier ions for some compounds have been changed from those recommended in SW-846 in order to improve the reliability of qualitative identification.

17.1.4. Method 8260A recommends that the purge vessel is run through an additional purge cycle after 25 mL sample analysis to remove carryover. Instead, purge vessels are oven baked between analyses or disposable vessels are used one time only.

17.1.5. SW-846 recommends that a curve be used for any analytes with %RSD of the response factors > 15%. However, some industry standard data systems and forms generation software cannot report this data with the necessary information for data validation. In addition most software available does not allow weighting of the curve. Unweighted curves may exhibit serious errors in quantitation at the low end, resulting in possible false positives or false negatives. Therefore, this SOP allows used of average response factors if the average %RSD for all compounds is ≤ 15%.

17.2. Modifications from previous revision

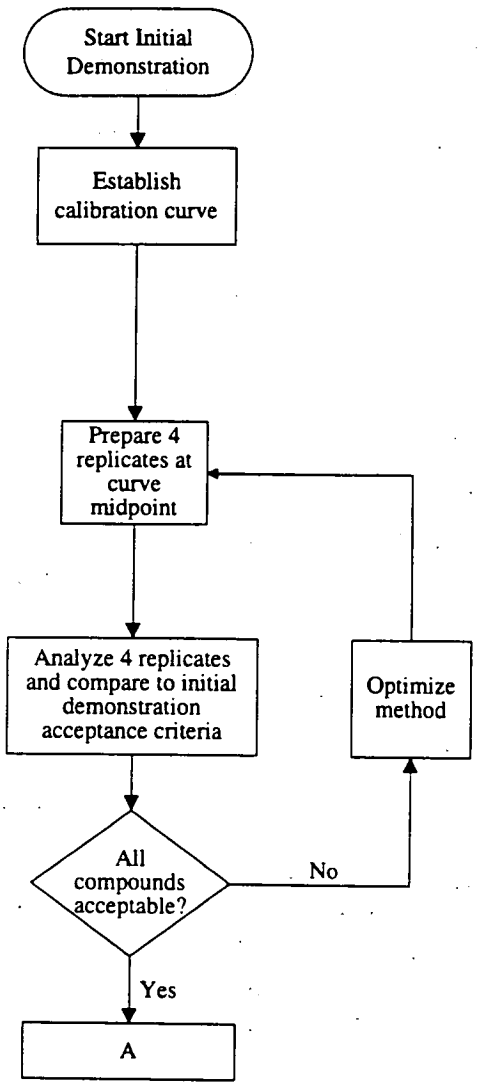
This SOP has been substantially revised to reflect the changes included in Update III to SW-846. Directions for method 524.2 and method 624 have also been added.

17.3. Facility specific SOPs

Each facility shall attach a list of facility-specific SOPs or approved attachments (if applicable) which are required to implement this SOP or which are used in conjunction with this SOP. If no facility specific SOPs or amendments are to be attached, a statement must be attached specifying that there are none.

17.4. Flow diagrams

17.4.1. Initial Demonstration and MDL



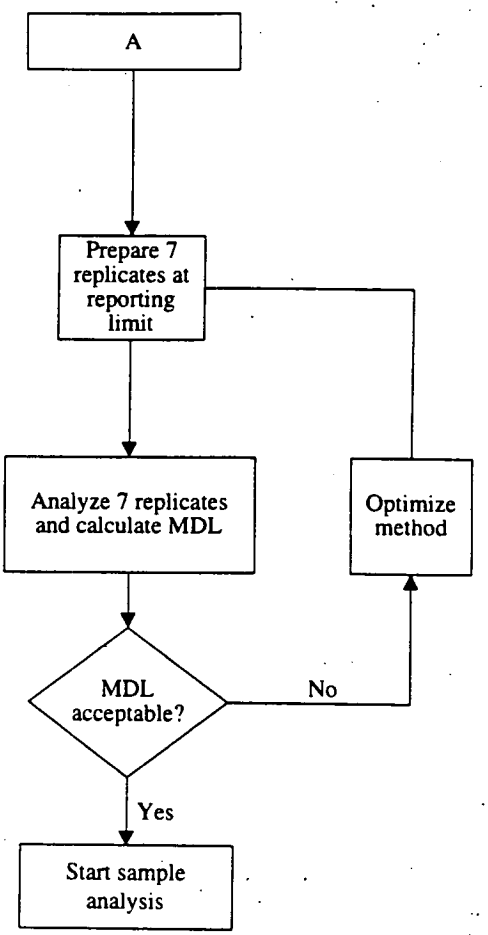


Table 1

Quanterra Primary Standard and Reporting Limits

Compound	CAS Number	Reporting Limits[1]			
		5 mL Water µg/L	25 mL water µg/L	Low soil µg/kg	Med. Soil µg/kg
Dichlorodifluoromethane	75-71-8	10	2	10	500
Chloromethane	74-87-3	10	2	10	500
Bromomethane	74-83-9	10	2	10	500
Vinyl chloride	75-01-4	10	2	10	500
Chloroethane	75-00-3	10	2	10	500
Trichlorofluoromethane	75-69-4	10	2	10	500
Acrolein	107-02-8	100	20	100	5000
Acetone	67-64-1	20	10	20	1000
Trichlorotrifluoroethane	76-13-1	5	1	5	250
Ethanol	64-17-5	500	200	500	25,000
Iodomethane	74-88-4	5	1	5	250
Carbon disulfide	75-15-0	5	1	5	250
Methylene chloride	75-09-2	5	1	5	250
tert-Butyl alcohol	75-65-0	200	50	200	10,000
1,1-Dichloroethene	75-35-4	5	1	5	250
1,1-Dichloroethane	75-34-3	5	1	5	250
trans-1,2-Dichloroethene	156-60-5	2.5	0.5	2.5	125
Acrylonitrile	107-13-1	100	20	100	5000
Methyl *tert*-butyl ether (MTBE)	1634-04-4	20	5	20	1000
Hexane	110-54-3	5	1	5	250
cis-1,2-Dichloroethene	156-59-2	2.5	0.5	2.5	125
1,2-Dichloroethene (Total)	540-59-0	5	1	5	250
Tetrahydrofuran	109-99-9	20	5	20	1000
Chloroform	67-66-3	5	1	5	250
1,2-Dichloroethane	107-06-2	5	1	5	250
Dibromomethane	74-95-3	5	1	5	250
2-Butanone	78-93-3	20	5	20	1000
1,4-Dioxane	123-91-1	500	200	500	25000
1,1,1-Trichloroethane	71-55-6	5	1	5	250
Carbon tetrachloride	56-23-5	5	1	5	250
Bromodichloromethane	75-27-4	5	1	5	250
1,2-Dichloropropane	78-87-5	5	1	5	250
cis-1,3-Dichloropropene	10061-01-5	5	1	5	250
Trichloroethene	79-01-6	5	1	5	250
Dibromochloromethane	124-48-1	5	1	5	250

Table 1

Quanterra Primary Standard and Reporting Limits

Compound	CAS Number	Reporting Limits[1]			
		5 mL Water µg/L	25 mL water µg/L	Low soil µg/kg	Med. Soil µg/kg
1,2-Dibromoethane	106-93-4	5	1	5	250
1,2,3-Trichloropropane	96-18-4	5	1	5	250
1,1,2-Trichloroethane	79-00-5	5	1	5	250
Benzene	71-43-2	5	1	5	250
Ethylmethacrylate	97-63-2	5	1	5	250
trans-1,3-Dichloropropene	10061-02-6	5	1	5	250
Bromoform	75-25-2	5	1	5	250
4-Methyl-2-pentanone	108-10-1	20	5	20	1000
2-Hexanone	591-78-6	20	5	20	1000
Tetrachloroethene	127-18-4	5	1	5	250
Toluene	108-88-3	5	1	5	250
1,1,2,2-Tetrachloroethane	79-34-5	5	1	5	250
2-Chloroethyl vinyl ether	110-75-8	N/A[2]	N/A	50	1000
Vinyl acetate	108-05-4	10	2	10	500
Chlorobenzene	108-90-7	5	1	5	250
Ethylbenzene	100-41-4	5	1	5	250
Styrene	100-42-5	5	1	5	250
t-1,4-Dichloro-2-butene	110-57-6	5	1	5	250
m and p Xylenes		2.5	0.5	2.5	125
o-xylene	95-47-6	2.5	0.5	2.5	125
Total xylenes	1330-20-7	5	1	5	250
1,3-Dichlorobenzene	541-73-1	5	1	5	250
1,4-Dichlorobenzene	106-46-7	5	1	5	250
1,2-Dichlorobenzene	95-50-1	5	1	5	250

[1] Reporting limits listed for soil/sediment are based on wet weight. The reporting limits calculated by the laboratory for soil/sediment, calculated on dry weight basis, will be higher.

[2] 2-Chloroethyl vinyl ether cannot be reliably recovered from acid preserved samples

Table 2

Quanterra Primary Standard Calibration Levels, 5 mL purge[1]

Compound	Calibration Level ug/L				
	Level 1	Level 2	Level 3	Level 4	Level 5
1,2-Dichloroethane-d4 (Surrogate)	5	20	50	100	200
Toluene-d8 (Surrogate)	5	20	50	100	200
4-Bromofluorobenzene (Surrogate)	5	20	50	100	200
Dichlorodifluoromethane	5	20	50	100	200
Chloromethane	5	20	50	100	200
Bromomethane	5	20	50	100	200
Vinyl chloride	5	20	50	100	200
Chloroethane	5	20	50	100	200
Trichlorofluoromethane	5	20	50	100	200
Acrolein	50	200	500	1000	2000
Acetone	5	20	50	100	200
Trichlorotrifluoroethane	5	20	50	100	200
Ethanol	500	2000	5000	10000	20000
Iodomethane	5	20	50	100	200
Carbon disulfide	5	20	50	100	200
Methylene chloride	5	20	50	100	200
tert-Butyl alcohol	100	400	1,000	2,000	4,000
1,1-Dichloroethene	5	20	50	100	200
1,1-Dichloroethane	5	20	50	100	200
trans-1,2-Dichloroethene	5	20	50	100	200
Acrylonitrile	50	200	500	1,000	2,000
Methyl *tert*-butyl ether (MTBE)	5	20	50	100	200
Hexane	5	20	50	100	200
cis-1,2-Dichloroethene	5	20	50	100	200
Tetrahydrofuran	5	20	50	100	200
Chloroform	5	20	50	100	200
1,2-Dichloroethane	5	20	50	100	200
Dibromomethane	5	20	50	100	200
2-Butanone	5	20	50	100	200
1,4-Dioxane	250	1000	2,500	5,000	10,000
1,1,1-Trichloroethane	5	20	50	100	200
Carbon tetrachloride	5	20	50	100	200
Bromodichloromethane	5	20	50	100	200
1,2-Dichloropropane	5	20	50	100	200
cis-1,3-Dichloropropene	5	20	50	100	200
Trichloroethene	5	20	50	100	200

Table 2

Quanterra Primary Standard Calibration Levels, 5 mL purge[1]

Compound	Calibration Level ug/L				
	Level 1	Level 2	Level 3	Level 4	Level 5
Dibromochloromethane	5	20	50	100	200
1,2-Dibromoethane	5	20	50	100	200
1,2,3-Trichloropropane	5	20	50	100	200
1,1,2-Trichloroethane	5	20	50	100	200
Benzene	5	20	50	100	200
Ethylmethacrylate	5	20	50	100	200
trans-1,3-Dichloropropene	5	20	50	100	200
Bromoform	5	20	50	100	200
4-Methyl-2-pentanone	5	20	50	100	200
2-Hexanone	5	20	50	100	200
Tetrachloroethene	5	20	50	100	200
Toluene	5	20	50	100	200
1,1,2,2-Tetrachloroethane	5	20	50	100	200
2-Chloroethyl vinyl ether	10	40	100	200	400
Vinyl acetate	5	20	50	100	200
Chlorobenzene	5	20	50	100	200
Ethylbenzene	5	20	50	100	200
Styrene	5	20	50	100	200
t-1,4-Dichloro-2-butene	5	20	50	100	200
m and p Xylenes	10	40	100	200	400
o-xylene	5	20	50	100	200
1,3-Dichlorobenzene	5	20	50	100	200
1,4-Dichlorobenzene	5	20	50	100	200
1,2-Dichlorobenzene	5	20	50	100	200

[1] **Levels for 25 mL purge are 5 times lower in all cases**

Table 3

Quanterra Appendix IX Standard and Reporting Limits, 5 mL purge[1]

Compound	CAS Number	Reporting Limits			
		5 mL Water µg/L	25 mL water µg/L	Low Soil µg/kg	Medium Soil µg/mL
Allyl Chloride	107-05-1	10	2	10	500
Acetonitrile	75-05-8	100	20	100	5000
Dichlorofluoromethane		10	2	10	500
Isopropyl ether	108-20-3	10	2	10	500
Chloroprene	126-99-8	5	1	5	250
n-Butanol	71-36-3	200	50	200	10,000
Propionitrile	107-12-0	20	4	20	1000
Methacrylonitrile	126-98-7	5	1	5	250
Isobutanol	78-83-1	200	50	200	10,000
Methyl methacrylate	80-62-6	5	1	5	250
1,1,1,2-Tetrachloroethane	630-20-6	5	1	5	250
1,2-Dibromo-3-chloropropane	96-12-8	10	2	10	500
Ethyl ether	60-29-7	10	2	10	500
Ethyl Acetate	141-78-6	20	4	20	1,000
2-Nitropropane	79-46-9	10	2	10	500
Cyclohexanone	108-94-1	N/A[2]	N/A[2]	N/A[2]	N/A[2]
Isopropylbenzene	98-82-8	5	1	5	250

[1] **Levels for 25 mL purge are 5 times lower in all cases**

[2] Cyclohexanone decomposes to 1,1-dimethoxycyclohexane in methanolic solution. Reporting limits cannot be accurately determined.

Table 4

Quanterra Appendix IX Standard Calibration Levels, µg/L

Compound	Level 1	Level 2	Level 3	Level 4	Level 5
Allyl Chloride	5	20	50	100	200
Acetonitrile	50	200	500	1,000	2,000
Dichlorofluoromethane	5	20	50	100	200
Isopropyl ether	5	20	50	100	200
Chloroprene	5	20	50	100	200
n-Butanol	100	400	1,000	2,000	4,000
Propionitrile	10	40	100	200	400
Methacrylonitrile	5	20	50	100	200
Isobutanol	100	400	1,000	2,000	4,000
Methyl methacrylate	5	20	50	100	200
1,1,1,2-Tetrachloroethane	5	20	50	100	200
1,2-Dibromo-3-chloropropane	10	40	100	200	400
Ethyl ether	5	20	50	100	200
Ethyl Acetate	10	40	100	200	400
2-Nitropropane	10	40	100	200	400
Cyclohexanone	50	200	500	1,000	2,000
Isopropylbenzene Mike wants this moved to primary standard	5	20	50	100	200

Table 5

Reportable Analytes for Quanterra Standard Tests, Primary Standard

Compound	CAS Number	Quanterra Standard List	TCLP	TCL	Appendix IX	UTS
Dichlorodifluoromethane	75-71-8				X	X
Chloromethane	74-87-3	X		X	X	X
Bromomethane	74-83-9	X		X	X	X
Vinyl chloride	75-01-4	X	X	X	X	X
Chloroethane	75-00-3	X		X	X	X
Trichlorofluoromethane	75-69-4				X	X
Acrolein	107-02-8				X	X
Acetone	67-64-1	X		X	X	X
Trichlorotrifluoroethane	76-13-1					X
Ethanol	64-17-5					
Iodomethane	74-88-4				X	X
Carbon disulfide	75-15-0	X		X	X	X
Methylene chloride	75-09-2	X		X	X	X
tert-Butly alcohol	75-65-0					
1,1-Dichloroethene	75-35-4	X	X	X	X	X
1,1-Dichloroethane	75-34-3	X		X	X	X
trans-1,2-Dichloroethene	156-60-5	X		X	X	X
Total dichloroethene		X		X	X	X
Acrylonitrile	107-13-1				X	X
Methyl *tert*-butyl ether (MTBE)	1634-04-4					
Hexane	110-54-3					
cis-1,2-Dichloroethene	156-59-2	X		X		
Tetrahydrofuran	109-99-9					
Chloroform	67-66-3	X	X	X	X	X
1,2-Dichloroethane	107-06-2	X	X	X	X	X
Dibromomethane	74-95-3				X	X
2-Butanone	78-93-3	X	X	X	X	X
1,4-Dioxane	123-91-1				X	X
1,1,1-Trichloroethane	71-55-6	X		X	X	X
Carbon tetrachloride	56-23-5	X	X	X	X	X
Bromodichloromethane	75-27-4	X		X	X	X
1,2-Dichloropropane	78-87-5	X		X	X	X
cis-1,3-Dichloropropene	10061-01-	X		X	X	X

Table 5

Reportable Analytes for Quanterra Standard Tests, Primary Standard

Compound	CAS Number	Quanterra Standard List	TCLP	TCL	Appendix IX	UTS
	5					
Trichloroethene	79-01-6	X	X	X	X	X
Dibromochloromethane	124-48-1	X		X	X	X
1,2-Dibromoethane	106-93-4				X	X
1,2,3-Trichloropropane	96-18-4				X	X
1,1,2-Trichloroethane	79-00-5	X		X	X	X
Benzene	71-43-2	X	X	X	X	X
Ethylmethacrylate	97-63-2				X	X
trans-1,3-Dichloropropene	10061-02-6	X		X	X	X
Bromoform	75-25-2	X		X	X	X
4-Methyl-2-pentanone	108-10-1	X		X	X	X
2-Hexanone	591-78-6	X		X	X	
Tetrachloroethene	127-18-4	X	X	X	X	X
Toluene	108-88-3	X		X	X	X
1,1,2,2-Tetrachloroethane	79-34-5	X		X	X	X
2-Chloroethyl vinyl ether	110-75-8					
Vinyl acetate	108-05-4				X	
Chlorobenzene	108-90-7	X	X	X	X	X
Ethylbenzene	100-41-4	X		X	X	X
Styrene	100-42-5	X		X	X	
t-1,4-Dichloro-2-butene	110-57-6				X	
m and p Xylenes		X		X	X	X
o-xylene	95-47-6	X		X	X	X
Total xylenes	1330-20-7	X		X	X	X
1,3-Dichlorobenzene	541-73-1					
1,4-Dichlorobenzene	106-46-7					
1,2-Dichlorobenzene	95-50-1					

Table 6

Reportable Analytes for Quanterra Standard Tests, Appendix IX standard

Compound	Number	Quanterra Standard List	TCLP	TCL	Appendix IX	UTS
Allyl Chloride	107-05-1				X	
Acetonitrile	75-05-8				X	X
Dichlorofluoromethane	75-43-4					
Isopropyl ether	108-20-3					
Chloroprene	126-99-8				X	
n-Butanol	71-36-3					
Propionitrile	107-12-0				X	
Methacrylonitrile	126-98-7				X	X
Isobutanol	78-83-1				X	X
Methyl methacrylate	80-62-6				X	X
1,1,1,2-Tetrachloroethane	630-20-6				X	X
1,2-Dibromo-3-chloropropane	96-12-8				X	X
Ethyl ether	60-29-7					X
Ethyl Acetate	141-78-6					X
2-Nitropropane	79-46-9					
Cyclohexanone	108-94-1					X
Isopropylbenzene	98-82-8					

Table 7

Internal Standards

	Standard Concentration µg/mL	Quantitation ion (5 mL purge)	Quantitation ion (25 mL purge)
Fluorobenzene	25	96	96
Chlorobenzene-d5	25	117	119
1,4-Dichlorobenzene-d4	25	152	152

Notes:

1) 10 µL of the internal standard is added to the sample. This results in a concentration of each internal in the sample of 50µg/L for a 5 mL purge or 10 µg/L for a 25 mL purge.

2) Except for medium level soils, the surrogate and internal standards may be combined in one solution.

Table 8

Surrogate Standards

Surrogate Compounds	Standard Concentration µg/mL
1,2-Dichloroethane-d_4	25
Dibromofluoromethane	25
Toluene-d_8	25
4-Bromofluorobenzene	25

Notes:

1) 10 µL of the surrogate standard is added to the sample. This results in a concentration of each surrogate in the sample of 50µg/L for a 5 mL purge or 10 µg/L for a 25 mL purge.

2) Except for medium level soils, the surrogate and internal standards may be combined in one solution.

3) Recovery limits for surrogates are generated from historical data and are maintained by the QA department.

Table 9

Matrix Spike / LCS Compounds

Compound	Standard Concentration µg /mL
1,1-Dichloroethene	25
Trichloroethene	25
Toluene	25
Benzene	25
Chlorobenzene	25

Notes:

1) 10 µL of the standard is added to the LCS or matrix spiked sample. This results in a concentration of each spike analyte in the sample of 50µg/L for a 5 mL purge or 10 µg/L for a 25 mL purge.

2) Recovery and precision limits for LCS and MS/MSD are generated from historical data and are maintained by the QA dedartment.

3) Full analyte spikes may also be used at the laboratories option or at client request.

Table 10

BFB Key Ion Abundance Criteria

Mass	Ion Abundance Criteria
50	15% to 40% of Mass 95
75	30% to 60% of Mass 95
95	Base Peak, 100% Relative Abundance
96	5% to 9% of Mass 95
173	Less Than 2% of Mass 174
174	Greater Than 50% of Mass 95
175	5% to 9% of Mass 174
176	Greater Than 95%, But Less Than 101% of Mass 174
177	5% to 9% of Mass 176

Table 11

SPCC Compounds and Minimum Response Factors

Compound	8240B Min. RF	8260B Min. RF
Chloromethane	0.300	0.100
1,1-Dichloroethane	0.300	0.100
Bromoform	>0.100	>0.100
1,1,2,2-Tetrachloroethane	0.300	0.300
Chlorobenzene	0.300	0.300

Table 12

CCC compounds

Compound	Max. %RSD from Initial Calibration	Max. %D for continuing calibration
Vinyl Chloride	<30.0	<20.0
1,1-Dichloroethene	<30.0	<20.0
Chloroform	<30.0	<20.0
1,2-Dichloropropane	<30.0	<20.0
Toluene	<30.0	<20.0
Ethylbenzene	<30.0	<20.0

Table 13

Characteristic ions

Compound	Primary*	Secondary	Tertiary
1,2-Dichloroethane-d_4 (Surrogate)	65	102	
Dichlorodifluoromethane	85	87	50, 101,103
Chloromethane	50	52	49
Vinyl chloride	62	64	61
Bromomethane	94	96	79
Chloroethane	64	66	49
Trichlorofluoromethane	101	103	66
1,1-Dichloroethene	96	61	98
Acrolein	56	55	58
Iodomethane	142	127	141

Table 13

Characteristic ions

Compound	Primary*	Secondary	Tertiary
Carbon disulfide	76	78	
Trichlorotrifluoroethane	151	101	153
Ethanol	45	46	
Acetone	43	58	
Methylene chloride	84	49	51. 86
tert-Butyl alcohol	59	74	
trans-1,2-Dichloroethene	96	61	98
Acrylonitrile	53	52	51
Methyl *tert* butyl ether	73		
Hexane	57	43	
1,1-Dichloroethane	63	65	83
cis-1,2-Dichloroethene	96	61	98
2-Butanone	43	72**	
Tetrahydrofuran	42	71	
Chloroform	83	85	47
1,2-Dichloroethane	62	64	98
Dibromomethane	93	174	95, 172, 176
1,4-Dioxane	88	58	
Vinyl acetate	43	86	
1,1,1-Trichloroethane	97	99	117
Carbon tetrachloride	117	119	121
Benzene	78	52	77
Trichloroethene	130	95	97, 132
1,2-Dichloropropane	63	65	41
Bromodichloromethane	83	85	129
2-Chloroethyl vinyl ether	63	65	106
cis-1,3-Dichloropropene	75	77	39
trans-1,3-Dichloropropene	75	77	39
1,1,2-Trichloroethane	97	83	85, 99
Chlorodibromomethane	129	127	131
Bromoform	173	171	175, 252
1,2,3-Trichloropropane	75	110	77, 112, 97
Toluene-d_8 (Surrogate)	98	70	100
4-Bromofluorobenzene (Surrogate)	95	174	176
Toluene	91	92	65
4-Methyl-2-pentanone	43	58	57, 100
Tetrachloroethene	164	166	131
Ethyl methacrylate	69	41	99, 86, 114
2-Hexanone	43	58	57, 100

Table 13

Characteristic ions

Compound	Primary*	Secondary	Tertiary
Chlorobenzene	112	114	77
Ethylbenzene	106	91	
Xylenes	106	91	
Styrene	104	103	78, 51, 77
Dichlorobenzene (all isomers)	146	148	111
trans 1,4-Dichloro-2-butene	53	75	89, 77, 124
1,1,2,2-Tetrachloroethane	83	85	131, 133
Allyl Chloride	76	41	78
Acetonitrile	40	41	
Dichlorofluoromethane	67	69	
Isopropyl ether	87	59	45
Chloroprene	53	88	90
n-Butanol	56	41	42
Propionitrile	54	52	55
Methacrylonitrile	41	67	52
Isobutanol	41	43	74
Methyl methacrylate	41	69	100
1,1,1,2-Tetrachloroethane	131	133	119
1,2-Dibromo-3-chloropropane	157	155	75
Ethyl ether	59	74	
Ethyl Acetate	43	88	61
2-Nitropropane	41	43	46
Cyclohexanone	55	42	98
Isopropylbenzene	105	120	

* The primary ion should be used for quantitation unless interferences are present, in which case a secondary ion may be used.

** m/z 43 may be used for quantitation of 2-Butanone, but m/z 72 must be present for positive identification.

18. SUMMARY

This appendix lists modifications to the main body of the SOP that are necessary for analysis of drinking water by method 524.2.

18.1. A target analyte list based on the list in method 524.2 is frequently requested for analysis by method 8260B. Quanterra's standard analyte list for this test, and the internal and surrogate standards used, are listed in Tables A-1 to A-4 below. In all other respects the method is as described in the main body of this SOP. Note that this without the modifications listed in Section 19, the method is ***not*** appropriate for drinking water analysis by method 524.2.

19. MODIFICATIONS REQUIRED FOR DRINKING WATER ANALYSIS BY METHOD 524.2

19.1. This method can be applied to surface water, ground water and drinking water.

19.2. Purge sample volume is normally 25 mL, but lesser volumes may be used if adequate sensitivity is obtained.

19.3. Sample concentrations are calculated using initial calibration curve.

19.4. Only one internal standard -- Fluorobenzene -- is used for this method, and therefore all target analytes are assigned to it.

19.5. A maximum of 25 ng of BFB is used for tuning for method 524.2

19.6. BFB tuning criteria for mass 75 are 30-80% of mass 95.

19.7. The recovery limits for the initial demonstration of capability are 80-120% with %RSD less than 20%.

19.8. Initial calibration curve requirements:

19.8.1. The number of calibration standards depends on the calibration range used. For a range of up to a factor of 20 (e.g. 1µg/L - 20µg/L) a minimum of three standards are necessary. For a factor of up to 50 four standards are necessary, and for a factor of up to 100 five standards are necessary.

19.8.2. All target compounds must have RSD ≤ 20%.

19.8.3. If this requirement can not be met, a regression curve must be constructed for the non-compliant compounds. There is no correlation coefficient requirement for the regression curve.

19.9. Continuing calibration verification (CCV) requirements:

19.9.1. All target compounds must have %D ≤ 30%.

19.9.2. The internal standards in each CCV must be over 70% of the abundance found in the CCV analysis immediately preceding it *and* over 50% of the calibration point in the initial calibration curve whose concentration matches that of the CCV.

19.9.3. The same analysis run may be used to satisfy the requirements for an LCS (also known as a laboratory fortified blank, LFB) and a continuing calibration verification. The LCS/CCV does not need to be a second source standard.

19.10. Method clarifications, modifications and additions

19.10.1. Section 7.1 requires that the trap packing materials be Tenax GC, Methyl silicone, silica gel and coconut charcoal. Quanterra routinely employs the Supelco VOCARB 3000, which consists of Carbopack B and Carboxen 1000 and 1001.

19.10.2. Section 7.8.2 of the source method requires that each calibration standard be prepared by diluting the appropriate volume of the working standard with organic-free water adjusted to pH < 2 in a volumetric flask. Quanterra prepares calibration standards by diluting the the appropriate volume of the working standard with organic-free water in the gas-tight syringe that will be used to inject the sample into the purge and trap device.

19.10.3. Sections 9.8 and 9.9 of the source method require that duplicate spiked blanks and a second-source initial calibration verification standard be analyzed at least quarterly. Since some Quanterra do not normally analyze drinking waters samples, these QC samples will be analyzed only during the conduct of projects that require this method.

Table A-1

Quanterra 8260 Drinking Water List Standard and Reporting Limits

Compound	CAS Number	Reporting Limits[1]			
		5 mL water µg/L	25 mL water µg/L	Low soil µg/kg	Med. Soil µg/kg
Dichlorodifluoromethane	75-71-8	10	2	10	500
Chloromethane	74-87-3	10	2	10	500

Table A-1

Quanterra 8260 Drinking Water List Standard and Reporting Limits

Compound	CAS Number	Reporting Limits[1]			
		5 mL water µg/L	25 mL water µg/L	Low soil µg/kg	Med. Soil µg/kg
Bromomethane	74-83-9	10	2	10	500
Vinyl chloride	75-01-4	10	2	10	500
Chloroethane	75-00-3	10	2	10	500
Trichlorofluoromethane	75-69-4	10	2	10	500
Acetone[1]	67-64-1	20	10	20	1000
Methylene chloride	75-09-2	5	2	5	250
1,1-Dichloroethene	75-35-4	5	1	5	250
1,1-Dichloroethane	75-34-3	5	1	5	250
trans-1,2-Dichloroethene	156-60-5	2.5	0.5	2.5	125
Methyl *tert*-butyl ether (MTBE)[1]	1634-04-4	20	5	20	250
2,2-Dichloropropane	590-20-7	5	1	5	250
cis-1,2-Dichloroethene	156-59-2	2.5	0.5	2.5	125
1,2-Dichloroethene (Total)	540-59-0	5	1	5	250
Chloroform	67-66-3	5	1	5	250
Bromochloromethane	74-97-5	5	1	5	250
1,2-Dichloroethane	107-06-2	5	1	5	250
Dibromomethane	74-95-3	5	1	5	250
2-Butanone[1]	78-93-3	20	5	20	1000
1,1,1-Trichloroethane	71-55-6	5	1	5	250
Carbon tetrachloride	56-23-5	5	1	5	250
1,1-Dichloropropene	563-58-6	5	1	5	250
Bromodichloromethane	75-27-4	5	1	5	250
1,2-Dichloropropane	78-87-5	5	1	5	250
1,3-Dichloropropane	142-28-9	5	1	5	250
cis-1,3-Dichloropropene	10061-01-5	5	1	5	250
Trichloroethene	79-01-6	5	1	5	250
Dibromochloromethane	124-48-1	5	1	5	250
1,2-Dibromoethane	106-93-4	5	1	5	250
1,2,3-Trichloropropane	96-18-4	5	1	5	250
1,1,2-Trichloroethane	79-00-5	5	1	5	250
Benzene	71-43-2	5	1	5	250
trans-1,3-Dichloropropene	10061-02-6	5	1	5	250
Bromoform	75-25-2	5	1	5	250
4-Methyl-2-pentanone[1]	108-10-1	20	5	20	1000
2-Hexanone[1]	591-78-6	20	5	20	1000

Table A-1

Quanterra 8260 Drinking Water List Standard and Reporting Limits

Compound	CAS Number	Reporting Limits[1]			
		5 mL water µg/L	25 mL water µg/L	Low soil µg/kg	Med. Soil µg/kg
Tetrachloroethene	127-18-4	5	1	5	250
Toluene	108-88-3	5	1	5	250
1,1,2,2-Tetrachloroethane	79-34-5	5	1	5	250
Chlorobenzene	108-90-7	5	1	5	250
1,1,1,2-Terrachloroethane	630-20-6	5	1	5	250
Ethylbenzene	100-41-4	5	1	5	250
Styrene	100-42-5	5	1	5	250
m and p Xylenes		2.5	0.5	2.5	125
o-xylene	95-47-6	2.5	0.5	2.5	125
Total xylenes	1330-20-7	5	1	5	250
Isopropylbenzene	98-82-8	5	1	5	250
Bromobenzene	108-86-1	5	1	5	250
n-Propylbenzene	103-65-1	5	1	5	250
2-Chlorotoluene	95-49-8	5	1	5	250
4-Chlorotoluene	106-43-4	5	1	5	250
1,3,5-Trimethylbenzene	108-67-8	5	1	5	250
tert-Butylbenzene	98-06-6	5	1	5	250
1,2,4-Trimethylbenzene	95-63-6	5	1	5	250
sec-butylbenzene	135-98-8	5	1	5	250
1,3-Dichlorobenzene	541-73-1	5	1	5	250
1,4-Dichlorobenzene	106-46-7	5	1	5	250
1,2-Dichlorobenzene	95-50-1	5	1	5	250
4-Isopropyltoluene	99-87-6	5	1	5	250
n-Butylbenzene	104-51-8	5	1	5	250
1,2-Dibromo-3-chloropropane	96-12-8	5	1	5	250
1,2,4-Trichlorobenzene	120-82-1	5	1	5	250
Napthalene	91-20-3	5	1	5	250
Hexachlorobutadiene	87-68-3	5	1	5	250
1,2,3-Trichlorobenzene	87-61-6	5	1	5	250

[1] Not included on the method 524.2 analyte list, but includes in the calibration standard as an add on frequently requested by method 8260B.

Table A-2

Internal Standards, Method 8260A Drinking water list

	Standard Concentration µg/mL	Quantitation ion
Fluorobenzene	25	96
Chlorobenzene-d5	25	119
1,4-Dichlorobenzene-d4	25	152

Notes:

1) Fluorobenzene only is used for method 524.2

2) 10 µL of the internal standard is added to the sample. This results in a concentration of each internal in the sample of 50µg/L for a 5 mL purge or 10 µg/L for a 25 mL purge.

3) Except for medium level soils, the surrogate and internal standards may be combined in one solution.

Table A-3

Surrogate Standards, Drinking water list

Surrogate Compounds	Standard Concentration µg/mL
1,2-Dichloroethane-d_4[1]	25
Dibromofluoromethane[1]	25
Toluene-d_8[1]	25
1,2-Dichlorobenzene-d4[2]	25
4-Bromofluorobenzene[1,2]	25

[1] 8260B surrogate

[2] 524.2 surrogate

Notes:

1) 10 µL of the surrogate standard is added to the sample. This results in a concentration of each surrogate in the sample of 50µg/L for a 5 mL purge or 10 µg/L for a 25 mL purge.

2) Except for medium level soils, the surrogate and internal standards may be combined in one solution.

Table A-4

Quanterra Drinking water list Standard: Calibration Levels

Compound	Level 1		Level 2		Level 3		Level 4		Level 5	
	5 mL	25 mL	5 mL	25 mL	5 mL	25 mL	5 mL	25 mL	5 mL	25 mL
Dichlorodifluoromethane	10	2	40	10	100	20	200	60	400	120
Chloromethane	10	2	40	10	100	20	200	60	400	120
Bromomethane	10	2	40	10	100	20	200	60	400	120
Vinyl chloride	10	2	40	10	100	20	200	60	400	120
Chloroethane	10	2	40	10	100	20	200	60	400	120
Trichlorofluoromethane	10	2	40	10	100	20	200	60	400	120
Acetone[1]	10		40	10	100	20	200	60	400	120
Methylene chloride	5	1	20	5	50	10	100	30	200	60
1,1-Dichloroethene	5	1	20	5	50	10	100	30	200	60
1,1-Dichloroethane	5	1	20	5	50	10	100	30	200	60
trans-1,2-Dichloroethene	5	1	20	5	50	10	100	30	200	60
Methyl *tert*-butyl ether (MTBE)	10		40	10	100	20	200	60	400	120
2,2-Dichloropropane	5	1	20	5	50	10	100	30	200	60
cis-1,2-Dichloroethene	5	1	20	5	50	10	100	30	200	60
Chloroform	5	1	20	5	50	10	100	30	200	60
Bromochloromethane	5	1	20	5	50	10	100	30	200	60
1,2-Dichloroethane	5	1	20	5	50	10	100	30	200	60
Dibromomethane	5	1	20	5	50	10	100	30	200	60
2-Butanone[1]	10		40	10	100	20	200	60	400	120
1,1,1-Trichloroethane	5	1	20	5	50	10	100	30	200	60
Carbon tetrachloride	5	1	20	5	50	10	100	30	200	60
Bromodichloromethane	5	1	20	5	50	10	100	30	200	60
1,2-Dichloropropane	5	1	20	5	50	10	100	30	200	60
cis-1,3-Dichloropropene	5	1	20	5	50	10	100	30	200	60
Trichloroethene	5	1	20	5	50	10	100	30	200	60

Table A-4

Quanterra Drinking water list Standard: Calibration Levels

Compound	Level 1		Level 2		Level 3		Level 4		Level 5	
Dibromochloromethane	5	1	20	5	50	10	100	30	200	60
1,2-Dibromoethane	5	1	20	5	50	10	100	30	200	60
1,2,3-Trichloropropane	5	1	20	5	50	10	100	30	200	60
1,1,2-Trichloroethane	5	1	20	5	50	10	100	30	200	60
Benzene	5	1	20	5	50	10	100	30	200	60
trans-1,3-Dichloropropene	5	1	20	5	50	10	100	30	200	60
Bromoform	5	1	20	5	50	10	100	30	200	60
4-Methyl-2-pentanone[1]	10		40	10	100	20	200	60	400	120
2-Hexanone[1]	10		40	10	100	20	200	60	400	120
Tetrachloroethene	5	1	20	5	50	10	100	30	200	60
Toluene	5	1	20	5	50	10	100	30	200	60
1,1,2,2-Tetrachloroethane	5	1	20	5	50	10	100	30	200	60
Chlorobenzene	5	1	20	5	50	10	100	30	200	60
Ethylbenzene	5	1	20	5	50	10	100	30	200	60
Styrene	5	1	20	5	50	10	100	30	200	60
m and p Xylenes	5	1	20	5	50	10	100	30	200	60
o-xylene	5	1	20	5	50	10	100	30	200	60
Isopropylbenzene	5	1	20	5	50	10	100	30	200	60
Bromobenzene	5	1	20	5	50	10	100	30	200	60
n-Propylbenzene	5	1	20	5	50	10	100	30	200	60
2-Chlorotoluene	5	1	20	5	50	10	100	30	200	60
4-Chlorotoluene	5	1	20	5	50	10	100	30	200	60
1,3,5-Trimethylbenzene	5	1	20	5	50	10	100	30	200	60
tert-Butylbenzene	5	1	20	5	50	10	100	30	200	60
1,2,4-Trimethylbenzene	5	1	20	5	50	10	100	30	200	60
sec-butylbenzene	5	1	20	5	50	10	100	30	200	60
1,3-Dichlorobenzene	5	1	20	5	50	10	100	30	200	60
1,4-Dichlorobenzene	5	1	20	5	50	10	100	30	200	60

Table A-4

Quanterra Drinking water list Standard: Calibration Levels

Compound	Level 1		Level 2		Level 3		Level 4		Level 5	
1,2-Dichlorobenzene	5	1	20	5	50	10	100	30	200	60
4-Isopropyltoluene	5	1	20	5	50	10	100	30	200	60
n-Butylbenzene	5	1	20	5	50	10	100	30	200	60
1,2-Dibromo-3-chloropropane	5	1	20	5	50	10	100	30	200	60
1,2,4-Trichlorobenzene	5	1	20	5	50	10	100	30	200	60
Napthalene	5	1	20	5	50	10	100	30	200	60
Hexachlorobutadiene	5	1	20	5	50	10	100	30	200	60
1,2,3-Trichlorobenzene	5	1	20	5	50	10	100	30	200	60

[1] Not included in the Quanterra Standard test, but included in the standard as a frequently requested add-on.

20. REQUIREMENTS FOR EPA 624

20.1. Method 624 is required for demonstration of compliance with NPDES wastewater discharge permits. This method can be applied only to aqueous matrices. The standard analyte list and reporting limits are listed in Table B-1.

20.2. The tune period for this method is defined as 24 hours.

20.3. The initial calibration curve for this method requires at least three points.

20.4. Sample concentrations are calculated using the average RRF from the initial calibration curve.

20.5. Each target analyte is assigned to the closest eluting internal standard.

20.6. Initial demonstration of Proficiency

20.6.1. The spiking level for the four replicate initial demonstration of proficiency is 20 µg/L. The acceptance criteria are listed in Table B-2

20.7. Initial calibration curve requirements:

20.7.1. Target compounds must have RSD ≤ 35%.

20.7.2. If this requirement can not be met, a regression curve must be constructed for the non-compliant compounds. There is no correlation coefficient requirement for the regression curve.

20.8. Continuing calibration verification requirements:

20.8.1. The continuing calibration standard is from a different source than the initial calibration standard. The acceptance criteria are listed in Table B-2.

20.9. Matrix Spike and LCS requirements

20.9.1. The matrix spike and LCS are spiked at 20 µg/L. A matrix spike duplicate is not necessary for this method. The recovery limits for matrix spike and LCS recovery are listed in Table C-2.

20.10. Method clarifications, modifications and additions

20.10.1. Section 5.2.2 of the source method describes the trap packing materials as Tenax GC, Methyl silicone, silica gel and coconut charcoal. Quanterra routinely employs the Supelco VOCARB 3000, which consists of Carbopack B and Carboxen 1000 and 1001.

20.10.2. Section 5.3.2 of the source method describes a packed analytical column. Quanterra routinely employs capillary columns when performing this method.

20.10.3. The source method provides a suggested list of compounds for internal and surrogate standards. Quanterra uses the following two compounds which are not on the table: Chlorobenzene-d_5 (internal standard) and 1,2-Difluorobenzene-d_4 (surrogate).

Table B-1.

Method 624 Analytes and Reporting Limits

Analytes	µg/L
Benzene	5
Bromodichloromethane	5
Bromoform	5
Bromomethane	5
Carbon tetrachloride	5
Chlorobenzene	5
Chloroethane	5
2-Chloroethyl vinyl ether	5
Chloroform	5
Chloromethane	5
Dibromochloromethane	5
1,2-Dichlorobenzene	5
1,3-Dichlorobenzene	5
1,4-Dichlorobenzene	5
1,1-Dichloroethane	5
1,2-Dichloroethane	5
1,1-Dichloroethene	5
trans-1,2-Dichloroethene	5
1,2-Dichloropropane	5
cis-1,3-Dichloropropene	5
trans-1,3-Dichloropropene	5
Ethylbenzene	5
Methylene chloride	5
1,1,2,2-Tetrachloroethane	5
Tetrachloroethene	5
Toluene	5
1,1,1-Trichloroethane	5
1,1,2-Trichloroethane	5
Trichloroethene	5
Trichlorofluoromethane	5
Vinyl chloride	5

Table B-2.

Method 624 QC Acceptance Criteria

Analytes	Daily QC check acceptance criteria (20µg/L spike)	Mean recovery, 4 replicate initial demonstration acceptance criteria (20µg/L spike)	Standard deviation, 4 replicate initial demonstration acceptance criteria (20µg/L spike)	Matrix spike and LCS acceptance criteria (% recovery)
Benzene	12.8-27.2	15.2-26.0	6.9	37-151
Bromodichloromethane	13.1-26.9	10.1-28.0	6.4	35-155
Bromoform	14.2-25.8	11.4-31.1	5.4	45-169
Bromomethane	2.8-37.2	D-41.2	17.9	D-242
Carbon tetrachloride	14.6-25.4	17.2-23.5	5.2	70-140
Chlorobenzene	13.2-26.8	16.4-27.4	6.3	37-160
Chloroethane	7.6-32.4	8.4-40.4	11.4	14-230
2-Chloroethyl vinyl ether	D-44.8	D-50.4	25.9	D-305
Chloroform	13.5-26.5	13.7-24.2	6.1	51-138
Chloromethane	D-40.8	D-45.9	19.8	D-273
Dibromochloromethane	13.5-26.5	13.8-26.6	6.1	53-149
1,2-Dichlorobenzene	12.6-27.4	11.8-34.7	7.1	18-190
1,3-Dichlorobenzene	14.6-25.4	17.0-28.8	5.5	59-156
1,4-Dichlorobenzene	12.6-27.4	11.8-34.7	7.1	18-190
1,1-Dichloroethane	14.5-25.5	14.2-28.5	5.1	59-155
1,2-Dichloroethane	13.6-26.4	14.3-27.4	6.0	49-155
1,1-Dichloroethene	10.1-29.9	3.7-42.3	9.1	D-234
trans-1,2-Dichloroethene	13.9-26.1	13.6-28.5	5.7	54-156
1,2-Dichloropropane	6.8-33.2	3.8-36.2	13.8	D-210
cis-1,3-Dichloropropene	4.8-35.2	1.0-39.0	15.8	D-227
trans-1,3-Dichloropropene	10.0-30.0	7.6-32.4	10.4	17-183
Ethylbenzene	11.8-28.2	17.4-26.7	7.5	37-162
Methylene chloride	12.1-27.9	D-41.0	7.4	D-221
1,1,2,2-Tetrachloroethane	12.1-27.9	13.5-27.2	7.4	46-157
Tetrachloroethene	14.7-25.3	17.0-26.6	5.0	64-148
Toluene	14.9-25.1	16.6-26.7	4.8	47-150
1,1,1-Trichloroethane	15.0-25.0	13.7-30.1	4.6	52-162
1,1,2-Trichloroethane	14.2-25.8	14.3-27.1	5.5	52-150
Trichloroethene	13.3-26.7	18.6-27.6	6.6	71-157
Trichlorofluoromethane	9.6-30.4	8.9-31.5	10.0	17-181
Vinyl chloride	0.8-39.2	D-43.5	20.0	D-251

Quanterra
Environmental Services

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DETERMINATION OF NITROAROMATICS, NITRAMINES, AND SPECIALTY EXPLOSIVES IN WATER AND SOIL BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY/ULTRAVIOLET DETECTOR (HPLC/UV) AND LIQUID CHROMATOGRAPHY/THERMOSPRAY/MASS SPECTROMETRY (LC/TSP/MS)

SUPERSEDES: Revision 4.0; August 3, 1995

Prepared by: Dennis E. Gall/Mike Filigenzi

Reviewed by: _____
Technical Specialist

Approved by: _____
Quality Assurance Manager

Approved by: _____
Environmental, Health and Safety Coordinator

Approved by: _____
Laboratory Director

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1. SCOPE AND APPLICATION

1.1 This method is suitable for the extraction and analysis of explosive residues (8330 components) listed below in water by high level and low level methods and in soil and sediment by High Performance Liquid Chromatography with Ultraviolet Detector (HPLC/UV) and with Triple-Stage Quadrapole Mass Spectrometer (LC/TSP/MS).

Compound	CAS No
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX, Octogen)	2691-41-0
1,3,5-trinitrobenzene	99-35-4
Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX, Hexogen)	121-82-4
1,3-dinitrobenzene	99-65-0
Nitrobenzene	98-95-3
2,4,6-trinitrotoluene (TNT)	118-96-7
Methyl-2,4,6-trinitrophenylnitramine (TETRYL)	479-45-8
2,4-dinitrotoluene	121-14-2
2,6-dinitrotoluene	606-20-2
2-Amino-4,6-Dinitrotoluene	35572-78-2
4-Amino-2,6-Dinitrotoluene	1946-51-0
2-nitrotoluene (o-nitrotoluene)	88-72-2
4-nitrotoluene (p-nitrotoluene)	99-99-0
3-nitrotoluene (m-nitrotoluene)	99-08-1

1.1.1 This method is NOT applicable for high concentrations of explosives in soil or waste samples. Method 8510 should be used to determine whether high concentrations of explosives are present in soil or waste samples.

1.1.2 For HPLC/UV, the standard range for most of the 8330 compounds is from 5.0 ng/mL to 1000 ng/mL, except for 1,3-dinitrobenzene and 2,4-dinitrotoluene which are from 2.5 ng/mL to 1000 ng/mL, in 25/75 0.1% acetic acid in acetonitrile/water,

1.2 This method is applicable for the extraction and analysis of 8330 compounds previously listed above and the following specialty explosives listed below by LC/TSP/MS.

Compound	CAS No
Nitroglycerine	55-63-0
Picric Acid (2,4,6-Trinitrophenol)	88-89-1
PETN	78-11-5

1.2.1 For LC/TSP/MS, the standard range is from 10 ng/mL to 1000 ng/mL for 8330 components, nitroglycerin, and PETN, and from 20 ng/mL to 2000 ng/mL for picric acid in 20/80 acetonitrile/0.2M ammonium acetate containing 0.1% acetic acid.

1.2.2 Aqueous samples for 8330 and specialty explosives can be extracted according to the low level or high level method, except for picric acid and nitroguanidine which are performed by the high level direct aqueous injection (DAI) method.

1.2.3 Soil samples for 8330 and specialty explosives can be extracted with 0.1% acetic acid in acetonitrile according to 8330 soil method except for picric acid which results in poor recovery.

1.2.4 Excellent recoveries for picric acid can be obtained from soil by extracting with 0.1% hydrochloric acid in 10% acetonitrile/water, however, this is not suitable for 2-amino-4,6-dinitrotoluene and 4-amino-2,6-dinitrotoluene which degrade rapidly.

1.2.5 With the addition of acetic acid in the extracting solvent and in the standard solutions, TETRYL has been stable for months without affecting the other nitroaromatics and nitramines.

1.3 This method is also suitable for the extraction and analysis of nitroguanidine in water and in soil by HPLC/UV.

Compound	CAS No
Nitroguanidine	556-88-7

1.3.1 The standard range for nitroguanidine by HPLC/UV is from 0.05 ug/mL to 2.0 ug/mL in 80/20 acetonitrile/water.

1.3.2 Nitroguanidine is not extractable from water, but water samples are diluted with acetonitrile and analyzed by DAI on the HPLC/UV.

1.3.3 Nitroguanidine is extractable from soil as per 8330 soil with 0.1% acetic acid in acetonitrile.

2. SUMMARY OF METHOD

2.1 Aqueous samples are either extracted and analyzed by the high level or the low level method, and analyzed by HPLC/UV or by LC/TSP/MS.

2.2 Aqueous samples for the high level method can be performed by DAI, where the samples must be analyzed within 7 days of sampling, or by the salting-out method or by solid phase extraction (SPE) where the extraction is performed within 7 days of sampling and analyzed within 40 days of extraction.

2.2.1 There is no extraction of aqueous samples for nitroguanidine except by DAI; samples are aliquotted, diluted with acetonitrile to extend the analytical hold time, filtered and analyzed by HPLC/UV.

2.2.2 Picric acid is performed by the high level DAI method; samples are aliquotted, diluted in ammonium acetate, filtered, internal standards are added, and the sample is analyzed by LC/TSP/MS.

2.2.3 Most other components can be performed by the high level or the low level salting-out method or SPE method.

2.2.4 The high level salting-out method and the high level SPE method are similar to the low level methods where the volume of the samples and reagents are downscaled approximately 10 to 1 with higher fortification levels.

2.3 Aqueous samples for the low level salting-out method are performed by saturating the water sample with sodium chloride and extracting with acetonitrile several times. The volume of the acetonitrile extracts are reduced by re-extracting with salt water, and adjusted to 5.0 mL. The extracts are diluted 1:3 before analysis. For HPLC/UV the extracts are diluted in water, and for LC/TSP/MS the extracts are diluted in 0.2 M ammonium acetate and internal standards are added.

2.4 Aqueous samples for the low level SPE method are performed by passing water samples through Porapak RDX cartridges, where the components are absorbed to and eluted from with 0.1% acetic acid in acetonitrile. The eluants are diluted with water for HPLC/UV or with 0.2 M ammonium acetate and internal standards added for LC/TSP/MS.

2.5 This method is NOT applicable for high concentrations of explosives in soil or waste samples.

2.6 Soil and sediment samples that appear to be non-homogeneous and are not suspicious are air dried, ground to a finer texture, and sieved through 30 mesh. A subsample is extracted for 8330, nitroguanidine, nitroglycerine, and PETN by using 0.1% acetic acid in acetonitrile in a cooled ultrasonic water bath for 18 hours, aliquotted, treated with calcium chloride solution (except for LC/MS), filtered, and analyzed by HPLC/UV or by LC/TSP/MS.

2.6.1 Acetic acid at 0.1% in acetonitrile improves the stability of TETRYL, but gives poor recoveries for picric acid in soil.

2.6.2 Picric acid is extracted from soil with 0.1% hydrochloric acid in 10% acetonitrile/water in a cooled ultrasonic water bath for 18 hours, filtered, aliquotted, internal standards added, and analyzed by LC/TSP/MS.

2.7 The 8330 extracts for HPLC/UV are analyzed on a C-8 reverse phase column with UV detection at 250 nm and confirmed on a Cyano (CN) column using reverse phase conditions and UV detection at 250 nm, or on a C-8 reverse phase column by LC/TSP/MS.

2.8 The extracts may be analyzed and confirmed by LC/TSP/MS using a C-8 reverse phase column.

3. DEFINITIONS

3.1 Interference - an obstruction or contamination at a similar retention time as the compound of interest.

3.2 Method Blank (MB) - A single control sample using similar controlled matrix in a set or batch for monitoring and to demonstrate that the reagents and solvents used are free from method interferences.

3.3 Laboratory Control Samples (LCS) - A single control sample using similar controlled matrix and fortified with target analytes.

3.4 Duplicate Control Samples (DCS) - A LCS performed in duplicate and fortified with target analytes.

3.5 Internal Standard - A compound either may be similar or not similar to the target analytes and may be isotopically labelled, is added to the final extracts to aid in determining that a valid injection has been made and may be used in the quantitation.

3.6 Matrix Spike (MS) - A single sample in a set or batch either selected at random or selectively choosen and fortified with target analytes.

3.7 Matrix Spike Duplicate (MSD) - A matrix spike performed in duplicate and fortified with target analytes.

3.8 Surrogate - A compound similar to the target analytes, may be isotopically labelled, to demonstrate the efficiency of the extraction on the sample.

3.9 Low Level method - Aqueous samples to be extracted according to the low level method for lower reporting limits of approximately 0.1 to 1.0 ng/mL (ppb).

3.10 High Level method - Aqueous samples to be extracted according to the high level method for higher reporting limits of approximately 4.0 to 14 ng/mL (ppb). NOTE: Explosive components are not very soluble in water, thus, there is no danger of possible re-crystalization and becoming dangerously explosive.

4. INTERFERENCES

4.1 Glassware must be cleaned and dried, free from interference.

4.2 The solvents and reagents used in the extraction and analysis of nitroaromatics, nitramines, and specialty explosives must be free of interference.

5. SAFETY

Procedures shall be carried out in a manner that protects the health and safety of all Quanterra associates.

5.1 Eye protection that satisfies ANSIZ87.1 (as per the Chemical Hygiene Plan), laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately. {VITON gloves may be worn when halogenated solvents are used for extractions or sample preparation. Nitrile gloves may be used when other solvents are handled. [Note: VITON is readily degraded by acetone; all solvents will readily pass through disposable latex rubber gloves.]}

5.2 The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the MSDS files maintained in the laboratory. The following specific hazards are known:

Chemicals that have been classified as carcinogens, or potential carcinogens, under OSHA include methylene chloride (used primarily in the rinsing of glassware).

Chemicals known to be flammable are acetone, acetonitrile, hexane, methanol, n-propanol, and tetrahydrofuran.

Tetrahydrofuran is known to be explosive as it forms explosive peroxides on prolonged storage.

Explosive standards in this method are secondary explosives. As neats, they are by nature unstable materials and they must be stored according to manufacturer's directions, preferrably wet; However, if the standards are not purchased as neats but as solutions at low concentrations in organic solvents, safety hazards is minimized as compare to neats.

Chemicals known to be corrosive are acetic acid and hydrochloric acid.

Hearing protection may be recommended when ultasonic digestion is carried out.

5.3 Exposure to chemicals must be maintained as low as reasonably achievable, therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.4 The preparation of standards and reagents (and glassware cleaning procedures that involve solvents such as methylene chloride) will be conducted in a fume hood with the sash closed as far as the operations will permit.

5.5 All work must be stopped in the event of a known or potential compromise to the health and safety of a Quanterra associate. The situation must be reported immediately to a laboratory supervisor.

5.6 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, from this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of Material Safety Data Sheets [MSDS] for chemicals used or encountered in this procedure should be and are made available to all personnel involved in the chemical analysis.

5.7 Each chemical compound must be treated as a potential health hazard, especially when the components are considered extremely explosive and should be kept wet in small quantities. All the compounds in this method are either used in the manufacturing of explosives or are the degradation products of compounds used in the manufacturing of explosives. Treat each compounds as if it were extremely explosive when handling and preparing stock solutions for calibration.

5.8 Soil samples having as high as 2% TNT can be safely ground. Samples containing higher levels should not be ground. Lumps of material that have a chemical appearance should be suspect and not ground. Explosives are generally a very finely ground grayish-white material.

5.8.1 Soil samples may be screened or tested before grinding if they are suspected to contain high levels of explosives using the TNT/RDX Soil Extraction Pac. Follow the manufacturer's instructions.

6. EQUIPMENT AND SUPPLIES

6.1 Balances - Top loading, capable of accurately weighing to the nearest 0.01 gram. Analytical, capable of accurately weighing to the nearest 0.0001 gram.

6.2 Centrifuge.

6.3 Filter assembly, 25-mm, 0.45 um pore size PTFE filters, such as Gelman Acrodisc #4219.

6.4 Liquid chromatograph

6.4.1 HPLC/UV - High Performance Liquid Chromatography with an Ultraviolet Detector. An analytical system complete with sample injection loop, analytical columns, a UV detector, and data system. A guard column is not used. See section 12.7, 12.8, and 12.9 for the appropriate HPLC conditions.

6.4.2 LC/TSP/MS - Liquid Chromatography/Thermospray/Mass Spectrometer. An analytical system complete with sample injection loop, analytical columns, a triple-stage quadrapole mass spectrometer, and data system. See section 12.10 for LC/TSP/MS conditions.

6.5 Mesh sieve and pan, brass or stainless steel, mesh size # 30.

6.6 Mortar and pestle, ceramic.

6.7 Recirculator cooler.

6.8 Spatula, stainless steel, or equivalent.

6.9 Stirring bars, magnetic.

6.10 Stirring plate, magnetic.

6.11 Syringe, 3-cc and 10-cc disposable.

6.12 Temperature controlled ultrasonic water bath.

6.13 Test tubes, 8-mL and 16-mL, glass with teflon-lined screwcap.

6.14 Vials, 40-mL, glass with teflon-lined screwcap.

6.15 Volumetric flask, 100-mL and 1000-mL.

7. REAGENTS AND SOLVENTS

All preparations of reagents and solutions are entered in the Reagent Prep Notebook.

7.1 Acetic acid, Glacial, Reagent grade.

7.1.1 Acetic acid in acetonitrile, 0.1% v/v.

7.2 Acetone, pesticide quality.

7.3 Acetonitrile, HPLC grade or equivalent.

7.4 Ammonium acetate, HPLC grade or equivalent.

7.4.1 Ammonium acetate, 0.2 M and 4 M (aqueous).

7.5 Calcium chloride, $CaCl_2$, aqueous solution; 1.3 grams/liter in distilled water.

7.6 Hexane, pesticide quality.

7.7 Hydrochloric acid, concentrated, reagent grade.

7.7.1 Hydrochloric acid in 10% acetonitrile/water, 0.1% v/v.

7.8 Methanol, HPLC grade or equivalent.

7.9 Methylene chloride, pesticide quality.

7.10 n-Propanol, HPLC grade or equivalent.

7.11 RDX/TNT Soil Extraction Pac, D TECH product, Accustandard catalog # EM-TK-1001S-1 or equivalent. Each kit contains materials to perform four tests.

7.12 Sodium chloride, reagent grade.

7.12.1 Salt water; 325 grams of sodium chloride in 1 liter of HPLC grade water (distilled water may be used for 8330 by LC/TSP/MS).

7.13 Tetrahydrofuran (THF), HPLC grade or equivalent.

7.14 Water, distilled.

7.15 Water, HPLC grade or equivalent.

8. STANDARDS

8.1 Stock standards and solutions may be prepared from standard materials or purchased as certified solutions. All standard preparations must be entered in the standards prep log (see Appendix 1).

A prepared mix solution of the 8330 components at 1.0 mg/mL in acetonitrile/methanol (1:1) is used for the preparation of fortification and analytical solutions. They are obtained from EM Science, Accustandard, Ultra Scientific, or other reputable source. The mix solutions from Accustandard and Ultra Scientific contained 14 components at 1.0 mg/mL, and expires in approximately one year if properly stored. Once the ampule is broken and the unused portion transferred to an amber vial, sealed with teflon-lined screwcap, and stored in the freezer, this solution is good for up to 30 days.

8.1.1 Prepare a 50 ug/mL mix solution in 0.1% acetic acid in acetonitrile from the primary mix standard above. Replace the solution after 30 days.

8.1.2 Prepare a 5.0 ug/mL mix solution from the 50 ug/mL mix solution from above in 0.1% acetic acid in acetonitrile. Replace the solutions after approximately 2 weeks.

8.1.3 Both solutions above are stored in the freezer in the dark and at -10 °C or less. These solutions are also used for fortification of samples.

8.2 Standards must be reviewd for expiration dates at least monthly. All expired standards must be rotated out of the laboratory to the HazWaste storage area for disposal per the CHP.

8.3 Surrogate for HPLC/UV.

8.3.1 Several chemicals listed below can be used as surrogate.

1-Chloro-3-nitrobenzene (CAS No. 121-73-3)

2,4-Dinitrofluorobenzene (CAS No. 70-34-8)

NOTE: Both compounds resolve from the 8330 analytes on the C8 analytical column, but only 2,4-dinitrofluorobenzene resolves from the 8330 analytes on the cyano confirmation column. Depending on the manufacturer and lot of the cyano column, 1-chloro-3-nitrobenzene co-elutes with RDX or with 3-nitrotoluene.

8.3.2 Prepare a stock solution from neat (obtained from Aldrich) at approximately 1.0 mg/mL in acetonitrile. Store the stock solution in the refrigerator and replace the stock after 1 year.

8.3.3 Prepare the surrogate solution from stock at 50 ug/mL in acetonitrile. Store the solution in the freezer and replace every six months.

8.4 Surrogate for LC/TSP/MS; d_5-nitrobenzene (CAS No. 4165-60-0)

8.4.1 Prepare a stock solution d_5-nitrobenzene from neat (obtained from CIL) at approximately 1.0 mg/mL in acetonitrile. Store the stock solution in the refrigerator and replace the solution after 1 year.

8.4.2 Prepare the surrogate solution from stock at 50 ug/mL in acetonitrile. Store the solution in the freezer and replace every six months.

8.5 Analytical Curve for 8330 HPLC/UV.

8.5.1 The analytical curve is prepared from the 5.0 ug/mL 8330 mix solution and the 50 ug/mL surrogate solution at the following concentrations in 25:75 0.1% Acetic acid in acetonitrile:HPLC grade water.

LEVEL:	T1	1	2	3	5	6	7	
8330 mix	2.5	5.0	10	25	100	500	1000	ng/mL
Surrogate	5.0	25	50	100	150	--	--	

Level T1 standard is the lowest level used for 1,3-dinitrobenzene, 2,4-dinitrotoluene, and the surrogate whereas the others used level 1 standard in the initial calibration for the HPLC/UV.

Store the solutions in the refrigerator and replace every 45 days. TETRYL is stable when properly stored with acetic acid.

8.5.2 A continuing calibration standard is prepared from the 5.0 ug/mL 8330 mix solution and the 5.0 ug/mL surrogate solution at 25 ng/mL with the surrogate at 100 ug/mL in 25:75 of 0.1% acetic acid in acetonitrile:HPLC grade water. Store the solution in the refrigerator and replace every 45 days.

8.6 Nitroguanidine is prepared from neat (obtained from Aldrich or Chem Service) at approximately 1.0 mg/mL in acetonitrile. Store the stock solution in the refrigerator and replace after 1 year.

8.6.1 Prepare nitroguanidine at 50 ug/mL and 5.0 ug/mL from the 1.0 mg/mL stock in acetonitrile. Store the solutions in the refrigerator and replace after six months. These solutions can also be used for fortification of samples.

8.6.2 Prepare the analytical curve for HPLC/UV from the 50 ug/mL standard at the following concentrations in 80:20 acetonitrile:water. Store the solutions in the refrigerator and replace after three months.

LEVEL:	1	2	3	4	5
NGu	0.05	0.10	0.20	1.0	2.0 ug/mL

8.7 SPECIALTY EXPLOSIVES (SPEX) - Nitroglycerine, picric acid, and PETN are received as solutions at 0.10 mg/mL or at 1.0 mg/mL in ethanol, methanol, or acetonitrile (obtained from EM Science, Accustandard, Ultra Scientific, or other reputable source).

8.7.1 A mixture of nitroglycerine, picric acid, and PETN (NGl/PA/PETN) is prepared at 20/20/10 ug/mL in acetonitrile. Store the solution in the freezer and replace the solution after 6 months. This solution can also be used for fortification of samples.

8.8 Internal Standards for LC/TSP/MS;

d_3-2,6-Dinitrotoluene (CAS No. 606-20-2) and d_4-1,3-dinitrobenzene (CAS No. 99-65-0)

8.8.1 Prepare individual stock solutions of d_3-2,6-dinitrotoluene and d_4-1,3-dinitrobenzene from neats (obtained from CIL) at approximately 1.0 mg/mL in acetonitrile. Store the stock solutions in the refrigerator and replace after 1 year.

8.8.2 Prepare an internal standard mix of d_3-2,6-dinitrotoluene and d_4-1,3-dinitrobenzene solution at 10 ug/mL in acetonitrile. Store the solution in the refrigerator and replace after 6 months.

8.9 Analytical Curve for LC/TSP/MS.

8.9.1 The 8330 and specialty explosives (SPEX) analytical curve is prepared at the following concentrations listed below in 2:8 acetontrile:0.2M ammonium acetate containing 0.1% acetic acid.

LEVEL:	1	2	3	4	5
8330 mix	10	50	100	500	1000 ng/mL
NGl	20	100	200	1000	2000
PA	20	100	200	1000	2000
PETN	10	50	100	500	1000
d_5-NB	50	100	150	200	250
d_3-DNT	50	50	50	50	50
d_4-DNB	50	50	50	50	50

where the 8330 mix is the 14 components, NGl is nitroglycerin, PA is picric acid, d_5-NB is the surrogate d_5-nitrobenzene, and d_3-DNT and d_4-DNB are the internal standards d_3-2,6-dinitrotoluene and d_4-1,3-dinitrobenzene.

8.9.2 Store the solutions in the refrigerator and replace after six months. TETRYL is stable when properly stored with 0.1% acetic acid.

8.10 Reference Standards

8.10.1 Prepare reference standards to verify the quality of the fortification solutions and analytical standards above.

8.10.2 Reference standard is any standard solution made from a source other than the stock standard. Reference standard may be from the EPA, the manufacturer, or from another reliable source.

8.10.3 If the secondary source is not available, a separate intermediate stock solution will be made from the same neat by another chemist or from a neat with a different lot number.

9. SAMPLE COLLECTION, PRESERVATION, CONTAINERS, AND HOLDING TIMES

9.1 Aqueous samples are collected in amber glass bottles with teflon-lined caps and iced or refrigerated at 2 - 6 °C from the time of collection until analysis.

9.2 Aqueous samples for the high level method must be aliquotted, filtered, and diluted within 7 days of sampling or extracted within 7 days and analyzed within 40 days from extraction.

9.2.1 If aqueous samples are not diluted in organic solvent, then the samples must be analyzed by DAI within 7 days of sampling.

9.3 Soil samples are collected in glass jars with teflon-lined caps and iced or refrigerated at 2 to 6 °C and covered from sunlight from time of collection until extraction.

9.4 Soil samples for 8330 and specialty explosives are to be air dried, finely ground and sifted, and extracted within 14 days of sampling and analyzed within 40 days of extraction.

9.4.1 Soil and sediment samples should be air dried until there is no visible appearance of moisture at room temperature or cooler after collection. Successful grinding and sifting verifies dryness. While it is possible to analyze wet soil samples, it is much more difficult to obtain a homogeneous subsample on a wet sample.

9.4.2 If wet soil samples are to be analyzed, a moisture determination must be made on a separate subsample (refer to SOP #LM-CAL-3055, "Determination of Percent Moisture (or Percent Solids)").

10. QUALITY CONTROL

10.1 A set or batch is a group of samples processed at the same time and by the same technique.

10.2 Any method blanks (MB) and control samples should be subjected to the same analytical procedures as those used on the actual samples at the same time.

10.2.1 A minimum of one method blank per batch or every twenty samples is required, whichever is more frequent.

The MB for aqueous samples consists of distilled water and for soil samples consists of Ottawa sand taken through the sample prep with the samples.

10.3 Laboratory Control Samples (LCS) are required and must be prepared for every 20 samples of each matrix, where MS/MSD are performed in the batch.

10.3.1 Duplicate Control Samples (DCS) are required with every batch where MS/MSD are not included.

LCS and DCS for water samples consists of distilled water and for soil samples consists of Ottawa sand fortified and taken through the sample prep with the samples.

10.4 Matrix Spike (MS) and Matrix Spike Duplicate (MSD) are required per batch.

10.4.1 The MS/MSD consists of two equal aliquots of a specified sample and are fortified at the same concentrations as the LCS or at client's request.

10.5 The surrogate is added to all samples in the batch, including the MB, LCS, DCS, MS, MSD, and Duplicates.

11. CALIBRATION AND STANDARDIZATION

11.1 A fixed injection volume is used for quantitation purposes and is to be the same for both the sample and standards.

11.2 Initial Calibration - A minimum of five analytical standards of different analyte concentrations are used to generate the curve.

11.2.1 On the HPLC/UV for 8330 and for nitroguanidine analysis, the initial calibration is injected once to obtain the peak height for each analyte at each concentration, and the response factor is calculated using the following formula (from VG Multichrom data system):

$$R_f = \text{(peak height)}/\text{(analyte concentration)}$$

11.2.2 The average response factor (R_fa) and the percent relative standard deviation (% RSD) is determined for each analyte. The average response factor is the summation of the R_f/n where n is the number of data points (n = 5 for five levels).

11.2.3 The initial calibration is valid for each analyte when the % RSD is equal to or less than 20%; linearity can be assumed and the average response factor can be used in place of a calibration curve (on the HPLC/UV).

11.2.3.1 If the % RSD is greater than 20 %, then the cause must be determined to bring the system back in control before the samples are analyzed, and the system must be recalibrated.

11.2.4 On the LC/TSP/MS for 8330 and specialty explosives, the initial calibration curve is generally injected at the beginning of the batch to obtain the area counts for each analyte at each concentration, and the response factor is calculated using the following formula (on Finnigan DEC Station 5000 data system):

$$R_f = \frac{C_s}{R_s} \times \frac{R_{is}}{C_{is}}$$

where C_s is the concentration of the standard in ng/mL,
R_s is the area count of the standard
C_{is} is the concentration of the internal standard,
R_{is} is the area count of the internal standard.

11.2.5 The average response factor (R_fa) and the percent relative standard deviation (% RSD) is determined for each analyte, and the initial calibration is valid for each analyte when the % RSD is equal to or less than 30%; linearity can be assumed and the average response factor can be used in place of a calibration curve (on the LC/TSP/MS).

11.2.5.1 If the % RSD is greater than 30 %, then the cause must be determined to bring the system back in control before samples are analyzed, and the system must be recalibrated.

11.2.6 All units used in the calculations must be consistently uniform, such as concentration in ng/mL and response in area or height counts.

11.3 Calibration Criteria - On the HPLC/UV, at the beginning of the batch, the continuing calibration standard is injected singly to determine the concentration for each analyte using the response factors from the initial calibration. The value calculated is compared to the expected value and should be equal to or less than 15% to be valid, and the samples may be analyzed.

11.3.1 The continuing calibration standard is the midpoint standard, and throughout the run it is the same.

11.3.2 If the continuing calibration standard is out of control, another injection of the continuing calibration standard may be injected singly to determine if it is back in control. If it's still out of control, then either the midpoint standard has degraded or the column has degraded and a new initial calibration is needed.

11.3.3 The initial calibraton curve is valid if the continuing calibration standard injected at the beginning of the analytical batch holds within the 15% criteria.

11.3.3.1 Minimally, if the 15% criteria is met, a five point curve is performed each year. However, the typical life of the column for 8330 by HPLC/UV is approximately two months of continuous use.

11.3.4 Calibration Criteria on the LC/TSP/MS - At the beginning of the batch, the initial calibration standards are injected to obtain the average respose factor for each analyte and the % RSD is calculated. If the % RSD is equal to or less than 25% then the initial calibration is valid and the analytical batch is analyzed.

11.3.5 A continuing calibration standard is injected at the beginning and singly for every 10 samples or less, and at the end of the batch for HPLC/UV and LC/TSP/MS.

11.3.6 The single injections of the continuing calibration standard on the HPLC/UV must be within 15% of the expected value based on the R_fa from the initial calibration to be valid.

11.3.7 On the LC/TSP/MS the continuing calibration standard must be within 25% of the expected value from the initial calibration.

12. PROCEDURE

One time procedural variations are allowed only if deemed necessary in the professional judgment of a supervisor to accommodate variation in sample matrix, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

Any unauthorized deviations from this procedure must be documented as a nonconformance, with a cause and corrective action described.

NOTES: If Direct Aqueous Injection can not be performed within the 7 day holding time for the high level 8330 and specialty explosives then the hold times can be extended by performing the salting-out method or the SPE method, however, the high level salting-out method and the SPE method are not applicable for nitroguanidine and picric acid.

The SPE method for 8330 has been approved by the USACOE and may be used in place of the salting-out method.

12.1 **HIGH LEVEL method by DAI**; for nitroguanidine (NGu), 8330, and Specialty Explosives (SPEX).

12.1.1 Label the sample ID with the appropriate suffix '8330-H-A', 'SPEX-H-A', 8330/SPEX-H-A, and 'NGu-A'.

12.1.2 Pipet 10 mL water sample into a 16-mL screwcap test tube.

For control, use distilled water or HPLC grade water.

12.1.3 Add the surrogate to the samples, including the method blank, DCS, LCS, and MS/MSD.

12.1.3.1 Surrogate is not applicable for nitroguanidine. The surrogates used for HPLC/UV and LC/TSP/MS do not inferfere with the analysis.

12.1.3.2 For 8330 by HPLC/UV, add 20 uL of the 50 ug/mL surrogate to the 10 mL water samples to yield 100 ppb (DO NOT ADD d_5-Nitrobenzene as it interferes with nitrobenzene on HPLC/UV).

12.1.3.3 For 8330, 8330/SPEX, or SPEX by LC/TSP/MS, add 40 uL of the 50 ug/mL d_5-Nitrobenzene standard to the 10 ml water samples to yield 200 ppb.

12.1.4 Add the spiking solution if applicable.

12.1.4.1 For nitroguanidine, add 160 uL of the 50 ug/mL nitroguanidine solution to the 10 mL water samples to yield 800 ppb.

12.1.4.2 For 8330 by HPLC/UV, add 100 uL of the 5.0 ug/mL 8330 mix solution to the 10 mL water samples to yield 50 ppb.

12.1.4.3 For SPEX by LC/TSP/MS, add 50 uL of the 20/20/10 ug/mL Nitroglycerine/Picric Acid/PETN solution to the 10 mL water samples to yield 100/100/50 ppb.

12.1.4.4 For 8330/SPEX by LC/TSP/MS, add both standards below to 10 mL water samples. Add 100 uL of the 5.0 ug/mL 8330-mix to yield 50 ppb, and 50 uL of the 20/20/10 ug/mL NGl/PA/PETN mix to yield 100/100/50 ppb.

12.1.5 Mix the contents well, and filter the water sample, the method blank, LCS/DCS, and MS/MSD.

12.1.5.1 Transfer the water sample into a 10-cc disposable syringe fitted with a 25-mm PTFE 0.45 um filter on the syringe tip and press the plunger. Collect the sample into another 16-mL screwcap test tube.

12.1.6 For 8330 by HPLC/UV, the extracts are ready for analysis. The final sample concentration is 10mL/10mL. If the extracts are to be split for another test, then aliquot the required amount of the 10/10 into a separate 8-mL test tubes for other test.

12.1.6.1 Store the 8330-H-A extracts in the freezer until ready for analysis by HPLC/UV; This analysis must be completed within 7 days of sampling.

12.1.7 For Nitroguanidine (NGu-A), aliquot 1.0 mL of the 10/10 water sample into an 8-mL test tube and dilute with acetonitrile to final volume of 4.0 mL (a 1:3 dilution). Mix the contents well.

12.1.7.1 Store the NGu-A extracts in the freezer until ready for analysis by HPLC/UV. The final concentration is 1.0 mL/4.0 mL, and the analysis is to be completed within 40 days from dilution with acetonitrile.

12.1.8 For 8330 by LC/TSP/MS and Specialty Explosives (SPEX-A), aliquot 2.0 mL of the 10/10 water sample into an 8-mL test tube. Add 100 uL of 4 M ammonium acetate, 10 uL of internal standard mix of d_3-2,6-DNT and d_4-1,3-DNB at 50 ng/mL, and mix the contents well. The final sample concentration is 2.0mL/2.1mL, except for the LCS/DCS and MS/MSD is 1.9mL/2.1mL.

12.1.8.1 Store the extracts in the freezer until ready for analysis by LC/TSP/MS.

12.2 **HIGH LEVEL** salting-out method for 8330 and specialty explosives.

This method is not applicable for nitroguanidine or picric acid - use DAI.

12.2.1 Label the sample tape with the appropriate suffix '8330-H-A' or '8330/SPEX-H-A'.

12.2.2 Measure 77.0 mL of water sample by weight into a specialized 100-mL volumetric flask.

12.2.2.1 For control, use HPLC grade water for 8330 by HPLC/UV or distilled water for 8330 by LC/TSP/MS.

12.2.3 Add surrogate to each sample, MB, LCS/DCS, and MS/MSD.

12.2.3.1 For 8330 by HPLC/UV, add 100 uL of the 50 ug/mL surrogate solution to the 77 mL water samples to yield 65 ppb.

12.2.3.2 For 8330/SPEX by LC/TSP/MS, add 200 uL of the 50 ug/mL d_5-Nitrobenzene solution to the 77 mL water samples to yield 130 ppb.

12.2.4 If applicable, fortify the LCS/DCS, and MS/MSD with the appropriate spiking standards.

12.2.4.1 For 8330-HPLC/UV, add 77 uL of the 50 ug/mL 8330 mix standard to the 77.0 mL water samples to yield 50 ppb.

12.2.4.2 For 8330/SPEX by LC/TSP/MS, add 77 uL of the 50 ug/mL 8330 mix and 0.40 mL of the 20/20/10 ug/mL NGl/PA/PETN mix to yield 50 ppb for 8330 mix and 100/100/50 ppb for SPEX.

12.2.5 Add 25.1 grams of sodium chloride (± 0.2 grams) to each water sample in the specialized volumetric flasks, including the control and spikes. Add the stir bar and mix the contents at maximum speed on a magnetic stirrer until the salt dissolves.

12.2.6 Add 16 mL of acetonitrile while the solution is being stirred and stir for approximately 5 minutes at high speed. An efficient stirring is observed when the acetonitrile layer is vortexing in the water sample.

12.2.7 After stirring for 5 minutes, allow the phases to separate to determine if any acetonitrile is recoverable. Occassionally there is little or no acetonitrile layer visible but the sample is not lost. Add an extra 1 to 3 mL of acetonitrile, and stir the solution again.

12.2.8 After stirring for 5 minutes, check again to determine if acetonitrile is recoverable if extra volume had been added.

12.2.9 If there is sufficient volume of acetonitrile (2 to 4 mL), continue to stir for an additional 15 minutes (total time approximately 20 minutes).

12.2.10 When the stirring is completed, turn off stirrer and allow the phases to separate for a minimum of 10 minutes.

12.2.11 Carefully transfer the upper acetonitrile phase to an 8-mL test tube with a pasteur pipet.

12.2.12 Extract the aqueous sample once more with another 1 to 2 mL of acetonitrile (but do not exceed a total of 5 mL when combined), stir at high speed for 15 to 20 minutes followed by 10 minutes for phase separation. Combine the second acetonitrile portion with the initial extract.

12.2.13 Carefully adjust the final volume to 5.0 mL with acetonitrile - NO nitrogen blowdown permitted.

12.2.14 Add 5 uL of concentrated acetic acid to each to stablize TETRYL, and mix the contents well.

12.2.15 Filter the extract through a 25-mm PTFE 0.45 um filter assembly using a 10-cc disposable syringe into another 8-mL test tube.

12.2.16 The acetonitrile extracts are stored in the freezer. A dilution of 1/10 is required for analysis to obtain a final concentration of 77mL/50mL - see below.

12.2.16.1 For HPLC/UV, dilute 0.4 mL of the extract with 3.6 mL distilled water in an 4-mL vial when ready for analysis.

12.2.16.2 For LC/TSP/MS, dilute 0.4 mL of the extract with 3.6 mL distilled water in a 4-mL vial. Add 20 uL of the internal standard mix d_3-DNT/d_4-DNB, seal with a teflon-lined screw cap and mix well. Store the extracts in the freezer until ready for analysis by LC/TSP/MS.

12.3 **HIGH LEVEL SPE method - EXTRACTION ON THE ZYMARK AUTOTRACE WORKSTATION**

12.3.1 Label the sample ID with the appropriate suffix '8330-H-A'.

12.3.2 QC's setup (MB, DCS, LCS, and MS/MSD) for Zymark AutoTrace SPE

12.3.2.1 For MB, DCS, and LCS, use HPLC grade water for 8330 by HPLC/UV or distilled water for 8330 by LC/TSP/MS.

12.3.2.2 If requested, control water supplied by the client may be used for MS/MSD. See section 12.3.4.1 below if sample contains sediments or suspended particulates.

12.3.2.3 Measure 100 mL of water sample by weight into an 8-oz glass bottle for QC's.

12.3.3 If applicable, fortify the DCS, LCS, and MS/MSD.

12.3.3.1 For 8330 by HPLC/UV, add 100 uL of the 50 ug/mL 8330 mix to the 100 mL water samples to yield 50 ppb.

12.3.3.2 For 8330/SPEX by LC/TSP/MS, add 100 uL of the 50 ug/mL 8330 mix to yield 50 ppb and 500 uL of the 20/20/10 ug/mL NGl/PA/PETN mix to yield 100/100/50 ppb in the 100 mL water samples.

12.3.4 Measure 100 ml of aqueous samples by weight into glass bottles in order to be able to add the surrogate to the samples.

12.3.4.1 If the water sample contains sediments or suspended particles, separate the particulates by centrifuging, filtering, or by decanting into a cleaned 1-L amber glass bottle.

12.3.5 Add surrogate to each sample, MB, DCS, LCS, and MS/MSD.

12.3.5.1 For 8330 by HPLC/UV, add 100 uL of the 50 ug/mL surrogate solution to the 100 mL water samples to yield 50 ppb.

12.3.5.2 For 8330 and 8330/SPEX by LC/TSP/MS, add 250 uL of the 50 ug/mL d_5-Nitrobenzene standard to the 100 mL water samples to yield 125 ppb.

12.3.6 Weigh and record the initial mass of each sample bottle, including the QC's (with the screw caps on).

12.3.7 Samples ready for extraction by the Zymark Autotrace SPE.

12.3.8 The program for the Autotrace SPE workstation must be set up using the PC computer with the following parameters on a 3.5" disk.

Use the disk - 8330-A, and edit the sample volume to 80 mL.

12.3.9 Below is an overview of the program. Whenever the method is modified, edit and update the method on a 3.5 inch disk.

Step 1: Process 6 samples using the following procedure:

Step 2: Condition column with 10 mL of acetonitrile into SOLVENT WASTE

Step 3: Condition column with 5 mL of acetonitrile into SOLVENT WASTE

Step 4: Condition column with 5 mL of water into AQUEOUS WASTE

Step 5: Condition column with 5 mL of HOAc/water 0.1% into AQUEOUS WASTE

Step 6: Load 80 mL of sample onto column

Step 7: Rinse column with 5 mL of HOAc/Water 0.1% into AQUEOUS WASTE

Step 8: Dry Column with gas for 0.5 minutes

Step 9: Wash syringe with 5 mL of HOAc/MeCN 0.1%

Step 10: Collect 5 mL fraction into sample tube using HOAc/MeCN 0.1%

Step 11: END

FLOW RATES	
Conditioning flow	15.0 mL/min
Load flow	10.0 mL/min
Rinse flow	20.0 mL/min
Elute flow	5.0 mL/min
Conditioning Air Push	15.0 mL/min
Rinse Air Push	20.0 mL/min
Elute Air Push	5.0 mL/min
SPE PARAMETERS	
Push Delay	5 sec
Air Factor	1.0
Autowash Volume	1.00 mL
WORKSTATION PARAMETERS	
Maximum Elution Volume	12.0 mL
Exhaust Fan on	Y Y=Yes N=No
Beeper on	Y Y=Yes N=No
NAME SOLVENTS	
Solvent 1	water
Solvent 2	methanol
Solvent 3	acetonitrile
Solvent 4	HOAc/Water 0.1%
Solvent 5	HOAc/MeCN 0.1%

HOAc/Water = acetic acid in water, 0.1% v/v
HOAc/MeCN = acetic acid in acetonitrile, 0.1% v/v
gas = compressed air or nitrogen

NEVER USE ACETONE ON THE ZYMARK AUTOTRACE SPE.

12.3.10 After the system is completed, the screen displays "program completed". Move the extracts aside and seal them with a teflon-lined screw cap. Remove the SPE columns and discard them, and record the final mass of the sample bottles and caps.

12.3.11 Run the 'Cleaning Sample Lines program' to clean sample lines, using emptied columns.

12.3.12 After the lines are cleaned, either continue with the next set of samples or turn off the Zymark - place the sample lines in a plastic bag.

12.3.13 Adjust the final volume to 5.0 mL with acetonitrile, and add 5 uL of concentrated acetic acid to stablize TETRYL. The extract concentration is 80 mL/5 mL.

12.3.14 Mix the contents well, and filter the acetonitrile extract through an Acrodisc LC25 filter assembly using a 10-cc disposable syringe into a new test tube. DO NOT READJUST THE VOLUME.

12.3.15 The acetonitrile extracts are stored in the freezer. A dilution of 1/10 is required for analysis to obtain a final concentration of 80mL/50mL.

12.3.15.1 For HPLC/UV, dilute 0.4 mL of the extract with 3.6 mL of distilled water in an 4-mL vial when ready for analysis.

12.3.15.2 For LC/TSP/MS, dilute 0.4 mL of the extract with 3.6 mL of 0.2 M ammonium acetate in a 4-mL vial. Add 20 uL of the internal standard mix d_3-DNT/d_4-DNB, seal with a teflon-lined screw cap and mix the contents well. The extracts are stored in the freezer until ready for analysis by LC/TSP/MS.

12.4 **LOW LEVEL salting-out method;** for 8330 by HPLC/UV and 8330/SPEX by LC/TSP/MS.

This method is not applicable for nitroguanidine and picric acid - use DAI.

The efficiency of extraction depends on the size of the stirring bar, the speed of the magnetic stirrer, and the design of the volumetric flask as the extraction vessel.

It is imperative that the amount of water sample, the mass of the sodium chloride, and the amount of acetonitrile be ACCURATELY measured and used as specified in this method.

12.4.1 Label the sample tape with the appropriate suffix '8330-LOW-A' for 8330 by HPLC/UV or 8330/SPEX-LOW-A for 8330 and specialty explosives by LC/TSP/MS.

12.4.2 Measure 770 mL of water sample into a 1000-mL volumetric flask by weight.

12.4.2.1 For control, use HPLC grade water for 8330 by HPLC/UV or distilled water for 8330 by LC/TSP/MS.

12.4.3 Add surrogate to each sample, MB, DCS, LCS, and MS/MSD.

12.4.3.1 For 8330 by HPLC/UV, add 40 uL of the 50 ug/mL surrogate solution to the 770 mL water samples to yield 2.6 ppb.

12.4.3.2 For 8330/SPEX by LC/TSP/MS, add 80 uL of the 50 ug/mL standard of d_5-Nitrobenzene to the 770 mL water samples to yield 5.2 ppb.

12.4.4 If applicable fortify the DCS, LCS, and the MS/MSD.

12.4.4.1 For 8330 by HPLC/UV, add 77 uL of the 5.0 ug/mL 8330-Mix standard to the 770 mL water samples to yield 0.50 ppb.

12.4.4.2 For 8330/SPEX by LC/TSP/MS, add 77 uL of the 5.0 ug/mL 8330 mix and 77 uL of the 20/20/10 ug/mL NGl/PA/PETN mix to the 770 ml water samples to yield 0.50 ppb on the 8330 mix and 2.0/2.0/1.0 ppb on the SPEX mix.

12.4.5 Add 251.4 grams of sodium chloride (± 0.2 grams) to each water sample in the volumetric flasks, including the control and spikes. Carefully add the stir bar and mix the contents at maximum speed on a magnetic stirrer until the salt dissolves.

12.4.6 Add 164 mL of acetonitrile (measured with a 250-mL graduated cylinder) while the solution is being stirred and stir for an additional 15 to 20 minutes at high speed. An efficient stirring is observed when the acetonitrile layer is vortexing in the water sample.

12.4.7 When the stirring is completed, turn off stirrer and allow the phases to separate for a minimum of 10 minutes.

12.4.8 Carefully transfer the upper acetonitrile phase (about 8 mL) to a 100-mL volumetric flask containing the 7/8 inch stirring bar with a pasteur pipet.

12.4.8.1 The amount of acetonitrile recovered should be approximately 6 to 10 mL. A larger volume could be indicative of inefficient vortexing of the extraction solvent into the water sample.

12.4.9 Extract the aqueous sample once more with another 8 to 10 mL of acetonitrile, stir at high speed for 15 to 20 minutes followed by 10 minutes for phase separation. Combine the second acetonitrile portion with the initial extract in the 100-mL volumetric flask. The inclusion of a few drops of salt water at this point is not important.

12.4.10 Add 84-85 mL of salt water (325 g sodium chloride per 1000 mL of distilled water) to the extracts in the 100-mL volumetric flask and stir the contents on the magnetic stirrer for 15 to 20 minutes at high speed. Again, an efficient stirring is observed when the acetonitrile layer is vortexing in the salt water.

12.4.10.1 After stirring for 5 minutes, allow the phases to separate to determine if any acetonitrile is recoverable. Occassionally there is little or no acetonitrile layer visible but the sample is not lost. Add an extra 1 to 4 mL of acetonitrile, and stir the solution again.

12.4.11 When the stirring is completed, turn off stirrer and allow the phases to separate for a minimum of 10 minutes.

12.4.12 Carefully transfer the upper acetonitrile phase (about 3 to 4 mL) to an 8-mL test tube with a pasteur pipet (and minimize the amount of water being transferred with the acetonitrile).

12.4.13 Extract the salt water once more with another 1.0 mL of acetonitrile, stir at high speed for 15 to 20 minutes followed by 10 minutes for phase separation. Combine the second acetonitrile portion with the initial extract in the test tube.

12.4.13.1 If the volume of acetonitrile from the first extraction in the 8-mL test tube is less than 4 mL, then use 1 mL of acetonitrile on the second extraction (not to exceed 5.0 mL final volume).

12.4.13.2 If the volume of acetontrile is over 4 mL, use less acetonitrile; do not exceed 5 mL.

12.4.13.3 If the volume of acetonitrile is over 5 mL, proceed with the second extraction with 1 mL acetonitrile, then transfer the combined acetonitrile in another 100-mL volumetric flask quantitatively with acetonitrile rinses, and proceed with steps 12.4.10 to 12.4.13 to reduce the volume.

12.4.14 Carefully adjust the volume to 5.0 mL with acetonitrile (NO nitrogen blowdown permitted), and add 5 uL of concentrated acetic acid to stablize TETRYL. Mix the contents well.

12.4.15 Filter the extract through a 25-mm PTFE 0.45 um filter assembly using a 10-cc disposable syringe into another 8-mL test tube.

12.4.16 The acetonitrile extracts are stored in the freezer. A dilution of 1/4 is required for analysis to obtain a final concentration of 770mL/20mL.

12.4.16.1 For HPLC/UV, dilute 1.0 mL of the extract with 3.0 mL of distilled water in an 8-mL test tube when ready for analysis.

12.4.16.2 For LC/TSP/MS, dilute 1.0 mL of the extract with 3.0 mL of 0.2 M ammonium acetate in a 4-mL vial. Add 20 uL of the internal standard mix d_3-DNT/d_4-DNB, seal with a teflon-lined screw cap and mix the contents well. The extracts are stored in the freezer until ready for analysis by LC/TSP/MS.

12.5 **LOW LEVEL SPE method - EXTRACTION ON THE ZYMARK AUTOTRACE WORKSTATION**

SPE column, 6-mL, 500 mg Porapak RDX Cartridges, Waters Catalog number WAT047220, or equivalent.

12.5.1 Label the sample tape with the appropriate suffix '8330-LOW-A' or '8330/SPEX-A'.

12.5.2 QC's setup (MB, DCS, LCS, and MS/MSD) for Zymark AutoTrace SPE

12.5.2.1 For MB, DCS, and LCS, use HPLC grade water for 8330 by HPLC/UV or distilled water for 8330 by LC/TSP/MS.

12.5.2.2 If requested, control water supplied by the client may be used for MS/MSD. See section 12.5.4.1 if sample contains sediments or suspended particulates.

12.5.2.3 Measure 900 mL of water sample by weight into a 1-L amber glass bottle for QC's.

12.5.3 If applicable, fortify the DCS, LCS, and MS/MSD.

12.5.3.1 For 8330 by HPLC/UV, add 90 uL of the 5.0 ug/mL 8330 Mix standard to the 900 mL water samples to yield 0.5 ppb.

12.5.3.2 For 8330/SPEX by LC/TSP/MS, add 90 uL of the 5.0 ug/mL 8330 Mix standard and 90 uL of the 20/20/10 ug/mL NGl/PA/PETN Mix to the 900 mL water samples to yield 0.5 ppb on the 8330 and 2/2/1 ppb on NGl/PA/ and PETN.

12.5.4 Measure 900 ml of aqueous samples by weight into glass bottles in order to be able to add surrogate to the samples.

12.5.4.1 If the water sample contains sediments or suspended particles, the samples must be separated from its particulates by centrifuging or by decanting into a cleaned 1-L amber glass bottle.

12.5.5 Add surrogate to each sample, MB, DCS, LCS, and MS/MSD.

12.5.5.1 For 8330 by HPLC/UV, add 45 uL of the 50 ug/mL surrogate solution to the 900 mL water samples to yield 2.5 ppb.

12.5.5.2 For 8330 by LC/TSP/MS, add 100 uL of the 50 ug/mL d_5-nitrobenzene standard to the 900 mL water samples to yield 5.6 ppb.

12.5.6 Weigh and record the initial mass of the sample bottles, including the QC's (with the screw caps on).

12.5.7 Samples ready for extraction by the Zymark Autotrace SPE.

12.5.8 The program for the Autotrace SPE workstation must be set up using the PC computer with the following parameters on a 3.5" disk.

12.5.9 Below is an overview of the program. Whenever the method is modified, edit and update the method on a 3.5 inch disk.

Step 1: Process 6 samples using the following procedure:

Step 2: Condition column with 10 mL of acetonitrile into SOLVENT WASTE

Step 3: Condition column with 5 mL of acetonitrile into SOLVENT WASTE

Step 4: Condition column with 5 mL of water into AQUEOUS WASTE

Step 5: Condition column with 5 mL of HOAc/water 0.1% into AQUEOUS WASTE

Step 6: Load 800 mL of sample onto column

Step 7: Rinse column with 5 mL of HOAc/Water 0.1% into AQUEOUS WASTE

Step 8: Dry Column with gas for 0.5 minutes

Step 9: Wash syringe with 5 mL of HOAc/MeCN 0.1%

Step 10: Collect 5 mL fraction into sample tube using HOAc/MeCN 0.1%

Step 11: END

FLOW RATES

Conditioning flow	15.0 mL/min
Load flow	10.0 mL/min
Rinse flow	20.0 mL/min
Elute flow	5.0 mL/min
Conditioning Air Push	15.0 mL/min
Rinse Air Push	20.0 mL/min
Elute Air Push	5.0 mL/min

SPE PARAMETERS		
Push Delay	5	sec
Air Factor	1.0	
Autowash Volume	1.00 mL	
WORKSTATION PARAMETERS		
Maximum Elution Volume	12.0 mL	
Exhaust Fan on	Y	Y=Yes N=No
Beeper on	Y	Y=Yes N=No
NAME SOLVENTS		
Solvent 1	water	
Solvent 2	methanol	
Solvent 3	acetonitrile	
Solvent 4	HOAc/Water 0.1%	
Solvent 5	HOAc/MeCN 0.1%	

HOAc/Water = acetic acid in water, 0.1% v/v
HOAc/MeCN = acetic acid in acetonitrile, 0.1% v/v
gas = compressed air or nitrogen.

NEVER USE ACETONE ON THE ZYMARK AUTOTRACE SPE.

12.5.10 Zymark Autotrace process time for six samples is approximately 124 minutes.

12.5.11 Before starting for the day, solvent lines should be purged by loading the solvent purging program. Check the solvents and replace the solvents if necessary.

12.5.12 The sample lines should have been in a plastic bag, indicating the lines have been cleaned with methanol followed by distilled water from the Cleaning Sample Lines program.

12.5.13 Set the SPE columns in place and depress the plunger. Once in place the green light should be on.

12.5.14 Set the receiving vessels (test tubes) in place. Be sure the first sample is on your left and the sixth sample on your right.

12.5.15 Check the solvents - replace if neccessary, and set the samples in its place.

12.5.16 Check the waste containers (Aqueous and Organic) and replace if necessary. Do NOT allow waste containers to overflow.

12.5.17 Load the program '8330-A' in the disk drive of the Zymark Autotrace, and press Load.

12.5.18 When the disk has been loaded, the Zymark Autotrace is ready to start. Follow instructions displayed on the screen "Press CONT to start" if ready to start.

12.5.19 After the system is completed, the screen displays "program completed". Move the extracts aside and seal them with teflon-lined screw caps. Remove the SPE columns and discard them, and record the final mass of the sample bottles and caps.

12.5.20 Run the 'Cleaning Sample Lines program' to clean sample lines, using emptied columns.

12.5.21 After the lines are cleaned, either continue with next set of samples or turn off the Zymark - place the sample lines in a plastic bag.

12.5.22 Adjust the final volume to 5.0 mL with acetonitrile, and add 5 uL of concentrated acetic acid to stablize TETRYL. Mix the contents well. The extract concentration is 800mL/5mL.

12.5.23 Filter the acetonitrile extract through an Acrodisc LC25 filter assembly using a 10-cc disposable syringe into a new test tube. DO NOT READJUST THE VOLUME.

12.5.24 The acetonitrile extracts are stored in the freezer. A dilution of 1/4 is required for analysis to obtain a final concentration of 800mL/20mL.

12.5.24.1 For HPLC/UV, dilute 1.0 mL of the extract with 3.0 mL of distilled water in a 4-mL vial when ready for analysis.

12.5.24.2 For LC/TSP/MS, dilute 1.0 mL of the extract with 3.0 mL of 0.2 M ammonium acetate in a 4-mL vial. Add 20 uL of the internal standard mix d_3-DNT/d_4-DNB, seal with a teflon-lined screw cap and mix the contents well. The extracts are stored in the freezer until ready for analysis by LC/TSP/MS.

12.6 Soil/Sediment Samples.

NOTE: Do NOT grind samples containing high concentrations of explosives. Lumps of material that have a chemical appearance should be suspect and not ground. Explosives are generally a very finely ground grayish-white material.

12.6.1 Subsample approximately 20 to 50 grams of soil into a 16-oz wide mouth jar for drying.

12.6.2 If the samples do not appear homogeneous and not suspicious, dry the samples in a cool, ventilated area and away from direct light. DO NOT HEAT the samples.

12.6.2.1 If samples do appear suspicious, stop work and consult with the project manager and client regarding to possible high levels of explosives.

12.6.3 After the samples are dried, grind thoroughly in mortar with pestle (both items previously rinsed with acetonitrile and dried).

12.6.4 Sift the sample through a sieve size 30 mesh. If samples are not to be extracted on the same day, store the sifted soil in the freezer and protect from light.

12.6.5 Label the sample tape with "8330-S" for 8330 by HPLC/UV, "NGU-S" for Nitroguanidine, "SPEX-S" for specialty explosives (Nitroglycerine, PETN, and Picric Acid), and 8330/SPEX-S for 8330 mix and specialty explosives by LC/TSP/MS.

12.6.5.1 Label the sample tape with 'PA-S' for picric acid as this is a separate extraction.

12.6.6 When the extractions are to be performed, turn on the recirculator cooler to cool the sonication bath to operating temperature (5 to 25 ^{o}C). The sonicator bath generates heat when the sonication bath is on.

12.6.7 Weigh 2.0 grams of homogeneous soil into a 40-mL vial.

12.6.7.1 For control, use Ottawa sand. If a larger sample size is desired, then the ratio of sample to solvent must be consistent to 1 gram/5 mL.

12.6.7.2 For picric acid, weigh out a separate set.

12.6.8 Add surrogate to each sample, MB, DCS, LCS, and MS/MSD.

12.6.8.1 No surrogate is used for the nitroguanidine analysis.

12.6.8.2 For 8330 by HPLC/UV, add 80 uL of the 50 ug/mL surrogate solution to each 2.0 gram soil sample to yield 2.0 ppm.

12.6.8.3 For 8330/SPEX and SPEX by LC/TSP/MS, add 200 uL of the 50 ug/mL d_5-Nitrobenzene standard to each 2.0 gram soil sample to yield 5.0 ppm.

12.6.8.4 For picric acid, add 50 uL of the 50 ug/mL d_5-Nitrobenzene standard to each 2.0 gram soil sample to yield 1.25 ppm.

12.6.9 If applicable, fortify the DCS/LCS, and MS/MSD.

12.6.9.1 For nitroguanidine, fortify at 1.0 ppm with Nitroguanidine by adding 40 uL of the 50 ug/mL standard to the 2 gram sample.

12.6.9.2 For 8330-HPLC/UV, fortify at 1.0 ppm with 8330 mix by adding 40 uL of the 50 ug/mL standard to the 2 gram sample.

12.6.9.3 For 8330 by LC/TSP/MS, fortify at 2.0 ppm with 8330 mix by adding 80 uL of the 50 ug/mL standard to the 2 gram sample.

12.6.9.4 For SPEX-LC/TSP/MS, fortify at 5/5/2.5 ppm of NGl/PA/PETN with SPEX Mix by adding 0.50 mL of the 20/20/10 mix to the 2 gram samples.

12.6.9.5 For PA in soils using the method for picric acid, fortify at 1.25 ppm with SPEX Mix by adding 125 uL to the 2 gram samples.

12.6.10 Adjust the final volume to 10.0 mL with 0.1% (v/v) acetic acid in acetonitrile (except for 'PA-S' see below). Cap, and shake briefly to mix the contents well.

12.6.10.1 To the separate set of PA-S (picric acid in soil) adjust the final volume to 10.0 mL with 0.1% hydrochloric acid in 10% acetonitrile in water, cap, and shake briefly to mix the contents well.

12.6.11 Place the samples in the ultrasonic bath, and sonicate for 18 hours, keeping the bath at 35 °C or cooler.

12.6.11.1 Record the initial time, date, and temperature of the water bath on the benchsheet. Cover the sonication bath with aluminum foil or a cover to protect samples from light.

12.6.11.2 After sonication, record the final time, date, and temperature of the water bath.

12.6.12 Allow the samples to settle for 30 minutes or centrifuge the samples at approximately 1000 rpm for about 5 minutes.

12.6.13 Decant the extracts (minimally 4 mLs) into 8-mL test tubes. The extracts may be worked up immediately or stored in the freezer until ready for further prep.

12.6.14 Filter the required amount needed for analysis by transferring into a 10-cc disposable syringes fitted with 0.45 um PTFE 25-mm filters, and filter them with positive pressure using the plunger into a clean 8-mL screwcap test tubes.

12.6.15 For 8330-HPLC/UV, dilute 1.0 mL of the filtered extract in an 8-mL screwcap test tube with 3.0 mL of the 1.3 g/L calcium chloride solution. Shake briefly to mix the contents.

12.6.15.1 Filter the extracts by transferring into a 10-cc disposable syringes fitted with 0.45 um PTFE 25-mm filters, and filter them with positive pressure using the plunger into a clean 4-mL screwcap vials.

12.6.15.2 Store the 8330-HPLC/UV extracts in the freezer and protect from light until analysis. The final sample concentration is 0.20 g/4.0 mL.

12.6.16 For nitroguanidine (NGu-S), dilute 2.0 mL of the filtered extract into an 8-mL test tube with 0.5 mL of distilled water to obtain a 80:20 acetonitrile:water composition. Mix the contents well.

12.6.16.1 Filter the extracts by transferring into 10-cc disposable syringes fitted with 0.45 um PTFE 25-mm filters, and filter them with positive pressure using the plunger into clean 4-mL screwcap vials.

12.6.16.2 Store the NGu-S extracts in the freezer and protect from light until analysis. The final concentration is 0.4g/2.5 mL.

12.6.17 For SPEX-LC/TSP/MS and 8330/SPEX-LC/TSP/MS, dilute 1.0 mL of the filtered extracts in an 8-mL test tube with 3.0 mL of distilled water (do NOT add calcium chloride solution!).

12.6.17.1 Add 20 uL of the internal standard mix d_3-2,6-DNT and d_4-1,3-DNB, and mix the contents well.

12.6.17.2 Filter the extracts with 10-cc disposable syringes fitted with 0.45 um PTFE 25-mm filters into clean 4-mL screwcap vials. The final extract concentration is 0.2g/4.0mL.

12.6.17.3 Store the extracts in the freezer and protect from light until ready for analysis by LC/TSP/MS.

12.6.18 For PA-S, the extracts may be filtered or decanted into an 8-mL test tube, and the extracts may be worked up immediately or stored in the freezer until ready for further prep.

Since the extracting solvent is 0.1% hydrochloric acid in 10% acetonitrile in water, the organic content of the extract is less than 20% in water, therefore no dilution with water is required. The final extract concentration is 2.0g/10mL.

12.6.18.1 If the extracts has not been filtered, then filter the required amount needed for analysis with 10-cc disposable syringes fitted with 0.45 um PTFE 25-mm filters into clean 8-mL screwcap test tubes.

12.6.19 Transfer 2.0 mL of the filtered extracts into 4-mL vials, and add 10 uL of the internal standard mix d_3-2,6-DNT and d_4-1,3-DNB to yield 50 ng/mL in the final extract. Mix the contents well, and the final extract concentration is 0.4g/2.0mL.

12.6.20 Store the extracts in the freezer and protect from light until analysis by LC/TSP/MS.

12.7 **INSTRUMENTATION for nitroguanidine by HPLC/UV**

The conditions listed below are the recommended analytical conditions for nitroguanidine by HPLC/UV. If changes to the HPLC conditions are necessary, they will be noted on the chromatogram.

12.7.1 Primary Column: Phenomenex Zorbax C_8, 250 x 4.6 mm, 5 um, or equivalent.

12.7.2 Column Temperature: 30 °C

12.7.3 Mobile Phase (Reservoir)
A = Water
B = Acetonitrile
C = Methanol

Gradient Program:

Time	A	B	C
0.0	100	0	0
8.0	100	0	0
9.0	0	50	50
15.0	0	50	50

12.7.4 Flow rate: 1.0 mL/min.

12.7.5 Injection volume: 25 uL

12.7.6 UV Detection at 250 nm.

12.7.7 Approximate retention times on C_8 column:

Nitroguanidine 4.3 minutes

12.7.8 Preventive Maintenance:

Change in-line-frit as needed to retain proper pressure, peak shape and response.

If nitroguanidine is not retained or the retention time is close to the void volume on the C8 or C18 column, replace with another column.

12.8 INSTRUMENTATION for 8330 by HPLC/UV

The conditions listed below are the recommended analytical conditions for 8330 by HPLC/UV. If changes to the HPLC conditions are necessary, they will be noted on the chromatogram.

12.8.1 Primary Column: Phenomenex Hypersil C_8, 150 x 4.6 mm, 5 um, or equivalent.

12.8.2 Column Temperature: 20 °C (Column kept cooled to a constant tempeature with a recirculated cooler).

12.8.3 Mobile Phase (Reservoir)

A = Water
B = 98:2 (v/v) methanol/tetrahydrofuran
C = 98:2 (v/v) methanol/acetonitrile

Gradient program:

Time	A	B	C
0.0	75	25	0
2.0	75	35	0
22.0	35	0	65
24.0	0	0	100

12.8.4 Flow rate: 1.5 mL/min.

12.8.5 Injection volume: 200 uL on UV-9050
250 uL on UV-200

12.8.6 UV Detection at 250 nm.

12.8.7 Elution order and approximate retention times on C_8 column:

HMX	3.90 minutes	
TNB	6.59	
RDX	7.16	
2,4-DNFB	8.58	(Surrogate)
DNB	9.43	
NB	10.95	
TNT	12.03	
TETRYL	13.09	
2,4-DNT	14.22	
2,6-DNT	14.92	
2-AM-4,6-DNT	15.47	
4-AM-2,6-DNT	15.75	
2-NT	16.24	
4-NT	16.52	
3-NT	17.09	

12.8.8 Preventive Maintenance:

Change in-line-frit as needed to retain proper pressure, peak shape and response. The use of guard column causes poor resolution between RDX and TNB; therefore, guard columns are not used.

Co-elution of RDX and TNB and of 2-AM and 4-AM on the primary column depends on the type and condition of the column and the solvent composition of THF in methanol.

Loss of resolution between TNB and RDX may be caused by column deterioration.

If the solvent composition of THF in methanol is changed, RDX and TNB elution order may be reversed or co-elution may occur.

12.9 **INSTRUMENTATION for Confirmation of 8330 by HPLC/UV**

The conditions listed below are the recommended analytical conditions for confirmation of 8330 by HPLC/UV. If changes to the HPLC conditions are necessary, they will be noted on the chromatogram.

12.9.1 Confirmation Column: Zorbax Cyano CN, 250 x 4.6 mm, 5 um, or equivalent.

12.9.2 Column Temperature: 20 °C (Column kept cooled to a constant temperature with a recirculated cooler)

12.9.3 Mobile Phase (Reservoir) and Gradient Program

OPTION 1

A = Water
B = 98:2 (v/v) methanol/THF
C = 98:2 (v/v) methanol/MeCN

Time	A	B	C
0.0	60	0	40
17.0	40	60	0
19.0	0	100	0
35.0	0	100	0

OPTION 2

A = water
B = acetonitrile
C = methanol

Time	A	B	C
0.0	80	8	12
20.0	60	28	12
35.0	0	100	0

12.9.4 Flow rate: 1.0 mL/min.

12.9.5 Injection volume: 500 uL on UV-9050
250 uL on UV-200

12.9.6 UV Detection at 250 nm.

12.9.7 Elution order and approximate retention times on CN column:

OPTION 1			OPTION 2		
NB	12.20		NB	18.52	
DNB	13.16		DNB	20.07	
TNB	13.63		2,4-DNFB	20.94	Surrogate
2-NT & 4-NT	15.19		TNB	21.57	
3-NT	15.59		2-NT & 4-NT	23.82	
1C3NB	15.62	Surrogate	3-NT	24.25	
2,6-DNT	16.23		RDX	24.71	
2,4-DNT	16.68		2,4-DNT	25.78	
TNT	17.96		2,6-DNT	26.37	
4-AM-2,6-DNT	18.19		2-AM-4,6-DNT	27.29	
2-AM-4,6-DNT	18.79		4-AM-2,6-DNT	27.50	
RDX	20.42		TNT	28.73	
TETRYL	23.14		HMX	29.48	
HMX	24.52		TETRYL	31.00	

3-NT and 1C3NB co-elute on option 1.
1C3NB and RDX co-elute on option 2.
2-NT and 4-NT co-elute on both options.

12.10 **INSTRUMENTATION for 8330 by LC/TSP/MS**

The conditions listed below are the recommended analytical conditions for the analysis of specialty explosives and for the analysis or confirmation of 8330 compounds. If changes to the LC/TSP/MS conditions are necessary, they will be noted on the chromatogram.

12.10.1 Primary Column: Novapak C-8, 3.9 X 150 mm, 5 um, or equivalent.

12.10.2 Temperature: Ambient

12.10.3 Mobile Phase (Reservoir)

A = 0.20 M Ammonium acetate (aqueous) with 0.10% acetic acid
B = N/A
C = n-Propanol

Gradient program:

Time	Flow	A	C	Grad
init	1.00	95	5	--
4.0	1.00	85	15	6
7.0	1.00	70	30	6
7.1	0.80	70	30	6
14.0	0.80	50	50	6
15.0	0.80	95	5	11
19.0	1.00	95	5	11

(flow rate in ml/min)

The post column pump flow rate of solution A is set at 0.40 mL/min throughout the run.

12.10.4 Injection volume: 150 uL

12.10.5 Thermospray (TSP) conditions

The following thermospray conditions must be optimized whenever a new thermospray vaporizer is installed in order to provide good performance.

Vaporizer Temperature:	80 to 120 °C
Source Temperature:	280 to 350 °C
Repeller Voltage:	0 to 50 Volts
Discharge Voltage:	1200 Volts
Filament Current:	0 to 400 uA
Electron Energy:	0 to 500 eV

(Note: Negative ion analysis will usually require discharge voltage of 800 to 1000 V, filament current of 400 uA, and electron energy of 500 eV).

12.10.6 Mass Spectrometer (MS) scanning conditions

Conditions below describe analysis in negative-ion selective-ion-monitoring (SIM) mode. Some compounds (e.g. HMX and RDX) also respond in positive-ion mode. These compounds may be analyzed using positive-ion selective-reaction-monitoring (SRM) for confirmation purposes or when interfering compounds are present in negative-ion SIM analysis. Ions used in positive-ion SRM analysis will differ from those listed below and must be established by analysis of standard reference materials.

Compound (in approximate order of elution)	Ion	Retention Time (approximated)
HMX	355	3:39
Trinitrobenzene	213	5:53
RDX	281	6:12
1,3-Dinitrobenzene	168	8:54
d_4-1,3-Dinitrobenzene	172	8:48 (Int. Std)
Picric Acid	228	8:50
Trinitrotoluene	227	9:52
TETRYL	241	10:12
d_5-Nitrobenzene	128	10:49 (Surrogate)
Nitrobenzene	123	10:57
Nitroglycerine	241	11:06
2,4-Dinitrotoluene	182	11:48
2-Amino-4,6-dinitrotoluene	197	11:56
d_3-2,6-Dinitrotoluene	185	12:12 (Int. Std)
4-Amino-2,6-dinitrotoluene	197	12:12
2,6-Dinitrotoluene	182	12:14
2- & 4-Nitrotoluene	137	13:08
PETN	330	13:14
3-Nitrotoluene	137	13:23

2-NT and 4-NT co-elute by this method.

12.10.7 Preventive Maintenance:

Change in-line-frit as needed to retain proper pressure, peak shape and response.

13. DATA ANALYSIS AND CALCULATIONS

13.1 Results are reported in ug/g or mg/Kg (ppm) for soil, unless otherwise instructed.

13.2 Results are reported as dry weight since soil samples are air dried, ground with motar and pestle, and sieved, unless specified by the client.

13.3 The concentration of each analyte and surrogate in a sample is calculated by using the appropriate formula:

Without using internal standard correction for HPLC/UV;

$$\text{Amount Found (ppb)} = \frac{CF_a \cdot A_x \cdot V_x}{W_s}$$

where CF_a = Average Calibration Factor in ng/mL.
A_x = Sample Total Area Counts.
V_x = Sample Extract Final volume in milliliters.
W_s = Initial Sample weight in grams.

Using internal standard correction for LC/TSP/MS;

$$\text{Amount Found (ppb)} = \frac{CF_a \cdot A_x \cdot V_x}{W_s} \times \frac{C_{is}}{A_{is}}$$

where C_{is} = Internal Standard Concentration in ng/mL.
A_{is} = Internal Standard Total Area Counts.

The results of soils in ppb (ng/g) are converted to ppm (ug/g) by dividing the result in ng/g by 1000;

$$\text{ug/g} = \frac{X \text{ ng/g}}{1000 \text{ ng/ug}}$$

14. METHOD PERFORMANCE

14.1 The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required expertise.

15. POLLUTION PREVENTION

NONE

16. WASTE MANAGEMENT

16.1 Flammable Waste

16.1.1 Acetone, acetonitrile, hexane, and methanol waste generated from solvent rinsing of glassware is collected as "Flammable Waste" in 1-gallon plastic bottles.

16.1.2 The unused portion of the acetonitrile extracts from soil extraction may be decanted from the soils and discarded as flammable waste. For the soil portion see "solid waste" below.

16.1.3 When the 1-gallon plastic bottle is full, the waste is transferred to the 55-gallon drum labelled "Flammable Waste" in the H-3 room.

16.1.4 Methylene chloride should be collected separately, see "DCM Waste" below. Any organic waste that is less than 50% methylene chloride is flammable waste.

16.2 Methylene Chloride (DCM) Waste

16.2.1 Methylene chloride, or dichloromethane (DCM), generated from solvent rinsing of glassware is collected separately from the Flammable Waste into "DCM Waste" in 1-gallon plastic bottles.

16.2.2 Any organic waste that is in excess of 50% methylene chloride is considered DCM waste.

16.2.3 When the 1-gallon plastic bottle is full, the waste is transferred to the 55-gallon drum labelled "DCM Waste" in the H-3 room.

16.3 Liquid-liquid extraction (LLE) Waste

16.3.1 Aqueous samples from high level and low level extraction (liquid-liquid extraction) after completion have been saturated with organic solvent (such as acetonitrile) is collected into 5-gallon carboy containers, or if the pH of the water samples were neutral (pH between 6 to 9) they can be collected directly into 55-gallon drum labelled "LLE Waste" in the H-3 room.

16.3.2 Aqueous samples collected in 5-gallon carboy containers are transferred to 55-gallon drum labelled "LLE Waste" in the H-3 room after the pH is check and ajusted if necessary to between 6 to 9.

16.4 High Performance Liquid Chromatography (HPLC) Effluent

16.4.1 Waste generated from the HPLC is collected into 1-L plastic bottles labelled "HPLC Waste", it consists primarily of methanol, acetonitrile, water, and ammonium acetate.

16.4.2 When the 1-gallon plastic bottle is full, the waste is transferred to the 55-gallon drum labelled "HPLC/ETHER Waste" in the H-3 room.

16.5 Solid Waste

16.5.1 Residual soil are allowed to air-dry in the hood or in well ventilated area. After the soils are dried, they are discarded into the "Dry Hazard" trash.

16.5.2 Dry Hazard trash consists of glass, gloves, papers, and plastics that were in contact with samples. When the trash is full, it is double bagged, secured, and disposed in the 55-gallon drum labelled "Dry Hazards" in the waste room where they are compacted in the 55-gallon drums.

16.5.3 Empty glass solvent bottles are disposed of in the waste room after they have been air dried. They are placed in the 55-gallon drum where they are compacted and crushed.

17. REFERENCES

17.1 SW-846 Draft Method 8330, "Nitroaromatics and Nitramines by High Pressure Liquid Chromatography (HPLC)", Revision 0, November 1992, U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington, D.C.

17.2 Thomas F. Jenkins, et. al., "Comparison of Cartridge and Membrane Solid-Phase Extraction with Salting-Out Solvent Extraction for Preconcentration of Nitroaromatic and Nitramine Explosives from Water", Special Report 92-95, December 1992, US Army Corps of Engineers, Cold Regions Research & Engineering Laboratory, Hanover, New Hampshire.

17.3 "Quanterra Quality Control Program", QA-003, Quanterra Environmental Services, Revision No. 0, April 4, 1995.

17.4 "Laboratory Documentation Practices", LP-CAL-0001, Quanterra Environmental Services, Revision 3.0, December 5, 1991.

17.5 "Calibration and Calibration Check of Balances", SAC-QA-0041, Quanterra Environmental Services, Revision 1.1, December 30, 1994.

18. MODIFICATIONS TO SOP

18.1 This method deviates from EPA method 8330, November 1992 revision on the following:

Acetonitrile is our preferred solvent instead of methanol in the preparation of standards.

The final solvent for extracts for HPLC/UV analysis should be no more than 25% acetonitrile in water. Higher percentages of acetonitrile causes poor chromatography.

Sodium chloride is used in the salting out method of aqueous samples instead of sodium sulfate.

The primary column used in this method is C-8 reverse phase instead of C-18, and the column length is 15 cm instead of 25 cm.

A gradient program is run using Water/Methanol with THF and Acetonitrile as modifiers instead of isocratic at 50/50 Methanol/Water.

The initial calibration is performed by injecting five concentration levels singly instead of triplicate injections of five levels randomly.

The amount of calcium chloride solution (5g/L aqueous) added to the soil extracts is 2:8 and is less than the original method of 1:1. Calcium chloride solution is a flocculating agent that aids in the filtration of soil extracts. Higher concentrations may cause clogging of the frits and over-pressurizing the HPLC.

The UV detector is set at 250 nm instead of 254 nm.

The injection volume is 200 uL instead of 100 uL.

18.2 Changes made on the original revision to revision 1.0.

Improved the stability of TETRYL in soil by the addition of acetic acid at 0.1% by volume in acetonitrile as the extraction solvent.

Included the component Nitroguanidine by HPLC/UV.

Included the components Nitroglycerine, PETN, and Picric Acid by LC/TSP/MS.

8330 components can also be analyzed by LC/TSP/MS.

Nitroglycerine, PETN, and Picric acid can be extracted using 0.1% hydrochloric acid in acetonitrile instead of acetic acid in acetonitrile; however, nitroglycerine and PETN can be extracted using acetic acid along with 8330 components.

18.3 Changes made on revision 1.0 to revision 2.0.

Added sections to include extraction of 8330 components at high levels by the low level extraction.

Included the extraction of Nitroguanidine in water and in soil.

Included the extraction of Specialty Explosives (Nitroglycerine, Picric Acid, and PETN) in water and in soil.

Included the LC/TSP/MS conditions for Specialty Explosives.

Analysis may be performed by LC/TSP/MS.

Confirmation on positives measured by HPLC/UV may be performed by LC/TSP/MS.

18.4 Changes made on revision 2.0 to revision 3.0

Component 1-Chloro-3-nitrobenzene is added as a surrogate for 8330 by HPLC/UV analysis.

Component d_5-Nitrobenzene is added as a surrogate for 8330 by LC/TSP/MS and component d_4-1,3-Dinitrobenzene is added as another internal standard with d_3-2,6-Dinitrotoluene for LC/TSP/MS.

Combining method 8330 and specialty explosives as a single methodology by LC/TSP/MS (8330/SPEX).

Included flow diagrams.

18.5 Changes made on revision 3.0 to revision 4.0.

Updated to Quanterra format.

Correction on the naming of TETRYL as Methyl-2,4,6-trinitrophenylnitramine, not Methyl-2,4,6-trinitrophenylamine.

Analytical curve and midpoint standards for 8330 by HPLC/UV are prepared in 25:75 0.1% acetic acid in acetonitrile:HPLC grade water, and is valid fon up to 45 days when properly stored in the refrigerator.

Addition of spiking soils for 8330-LCMS with 8330 mix at 2.0 ppm.

Changed final solvent composition of SPEX-A and 8330/SPEX-A to 20% acetonitrile in water (injection of samples greater than 20% acetonitrile in water adversely affects the chromatography).

Correction and clarity on the final prep of 8330/SPEX-LCMS-A.

Soil extracts are decanted into 8-cc test tubes for storage after extraction.

Addition of Method 8330 by SPE as Low Level and High Level in place of salting-out method.

Injection volume for cyano confirmation on UV 9050 to 250 uL and on UV 100 to 370 uL.

Injection volume for analytical column on UV 100 to 250 uL.

Separate extraction method for picric acid with 0.1% hydrochloric acid in 10% acetonitrile in water.

18.6 Changes made on revision 4.0 to revision 5.0.

SOP number changed from SAC-GC-0124 to SAC-LC-0001.

Other references related to quality added to References section.

Tetrahydrofuran added to list of flammable solvents.

Added TNT/RDX Soil Extrtaction Pac to test suspected soils before grinding.

New surrogate added, 2,4-dinitrofluorobenzene (2,4-DNFB) for HPLC/UV.

18.3 Changes made on revision 1.0 to revision 2.0.

Added sections to include extraction of 8330 components at high levels by the low level extraction.

Included the extraction of Nitroguanidine in water and in soil.

Included the extraction of Specialty Explosives (Nitroglycerine, Picric Acid, and PETN) in water and in soil.

Included the LC/TSP/MS conditions for Specialty Explosives.

Analysis may be performed by LC/TSP/MS.

Confirmation on positives measured by HPLC/UV may be performed by LC/TSP/MS.

18.4 Changes made on revision 2.0 to revision 3.0

Component 1-Chloro-3-nitrobenzene is added as a surrogate for 8330 by HPLC/UV analysis.

Component d_5-Nitrobenzene is added as a surrogate for 8330 by LC/TSP/MS and component d_4-1,3-Dinitrobenzene is added as another internal standard with d_3-2,6-Dinitrotoluene for LC/TSP/MS.

Combining method 8330 and specialty explosives as a single methodology by LC/TSP/MS (8330/SPEX).

Included flow diagrams.

18.5 Changes made on revision 3.0 to revision 4.0.

Updated to Quanterra format.

Correction on the naming of TETRYL as Methyl-2,4,6-trinitrophenylnitramine, not Methyl-2,4,6-trinitrophenylamine.

C4-121

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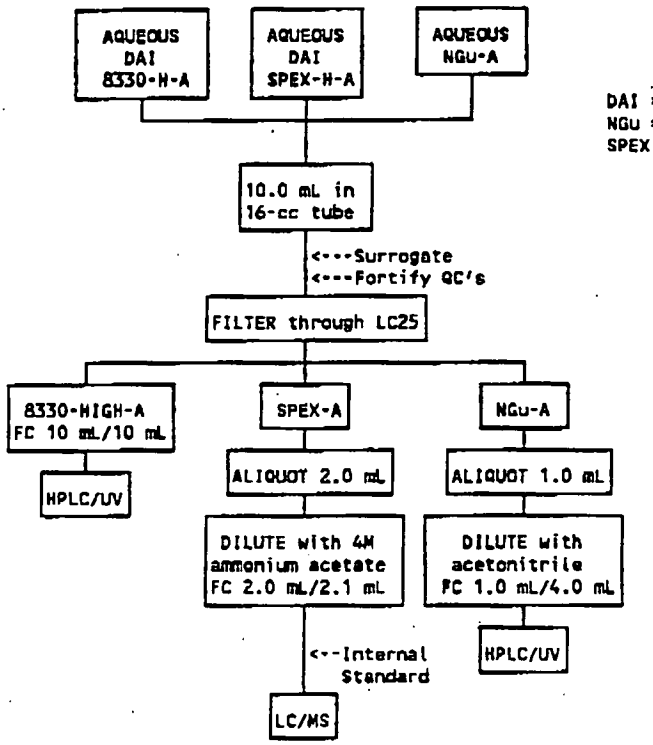
19. FLOW DIAGRAM

19.1 Aqueous by DAI for high levels.



DAI = Direct Aqueous Injection
NGu = Nitroguanidine
SPEX = Specialty Explosives (Nitroglycerine, Picric Acid, and PETN)

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SOP No. SAC-LC-0001
Revision No. 5.0
Date January 3, 1996
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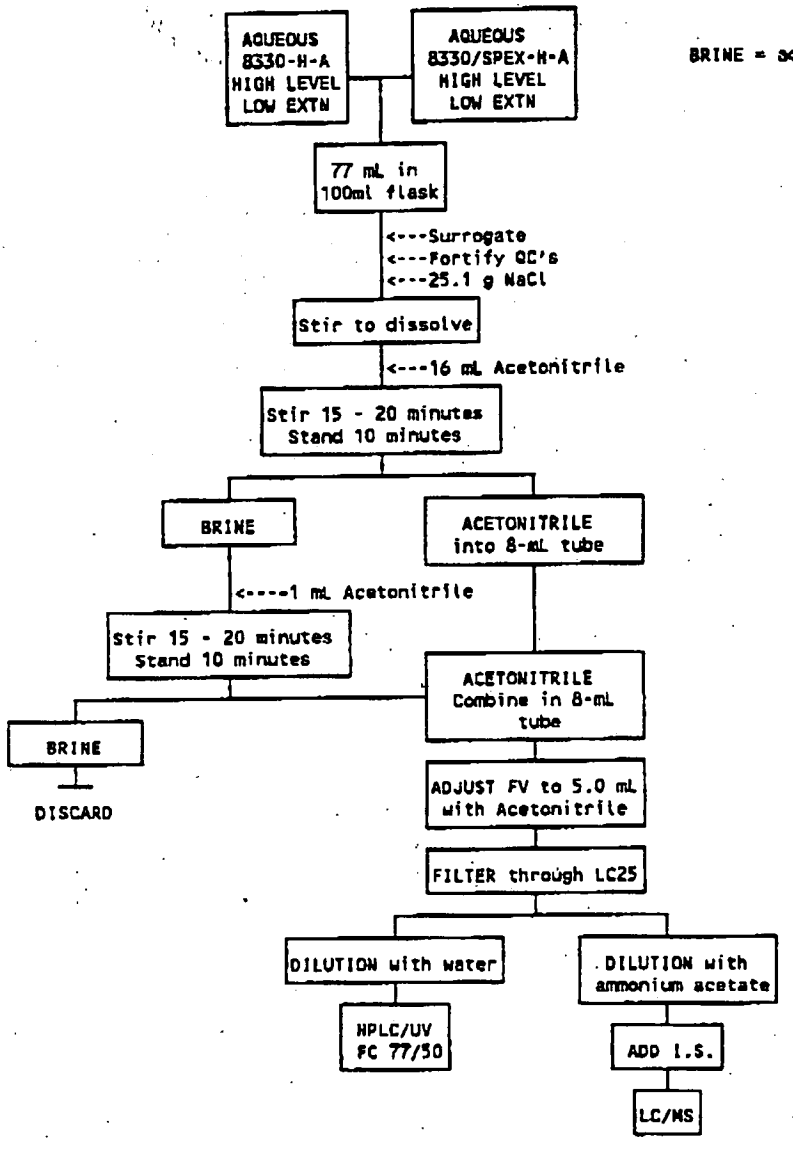
19.2 Aqueous by Salting Out for high levels.



CH-125

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Revision No. 5.0
Date January 3, 1996
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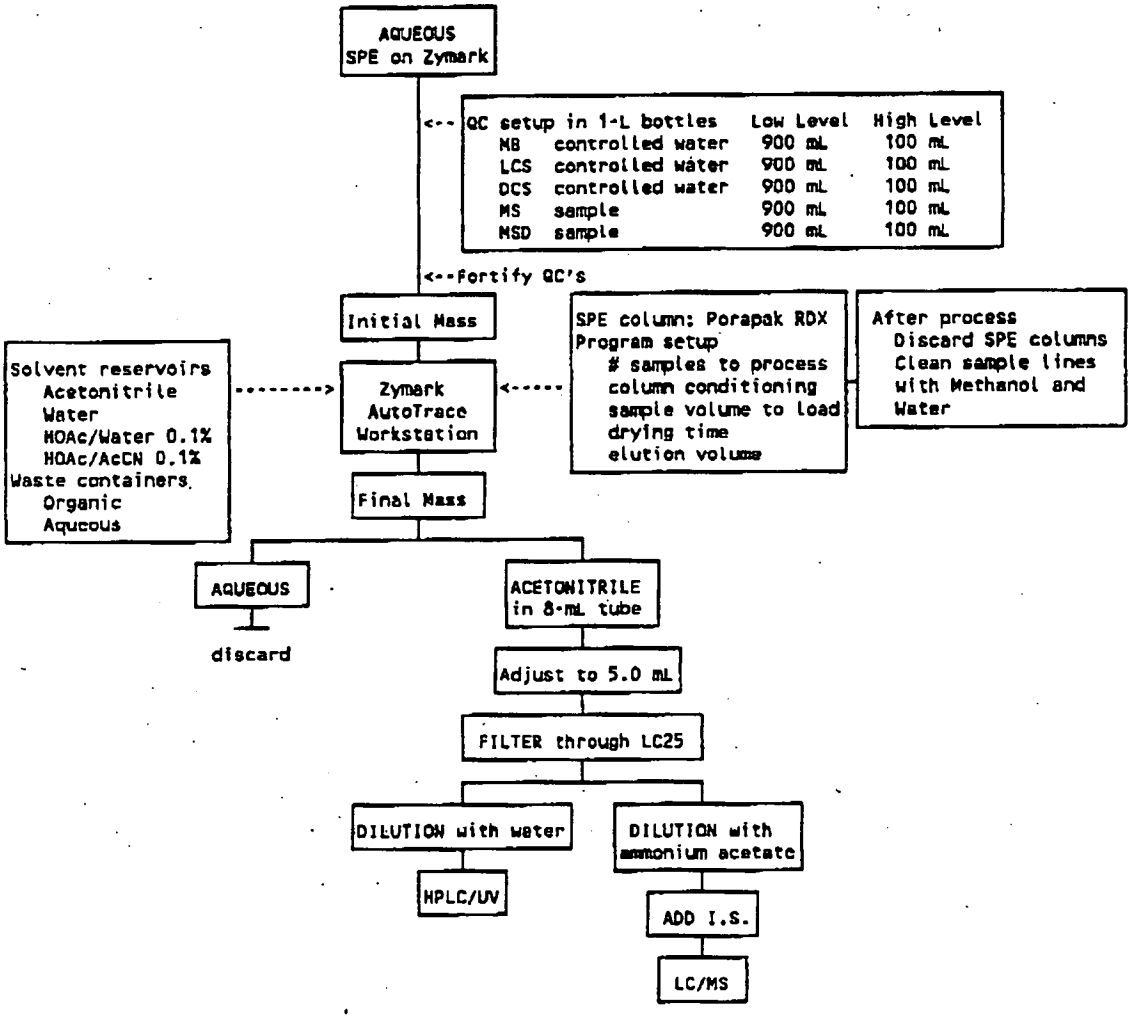
19.3 Aqueous by SPE using Zymark Autotrace Workstation for high levels or low levels.



C4-126

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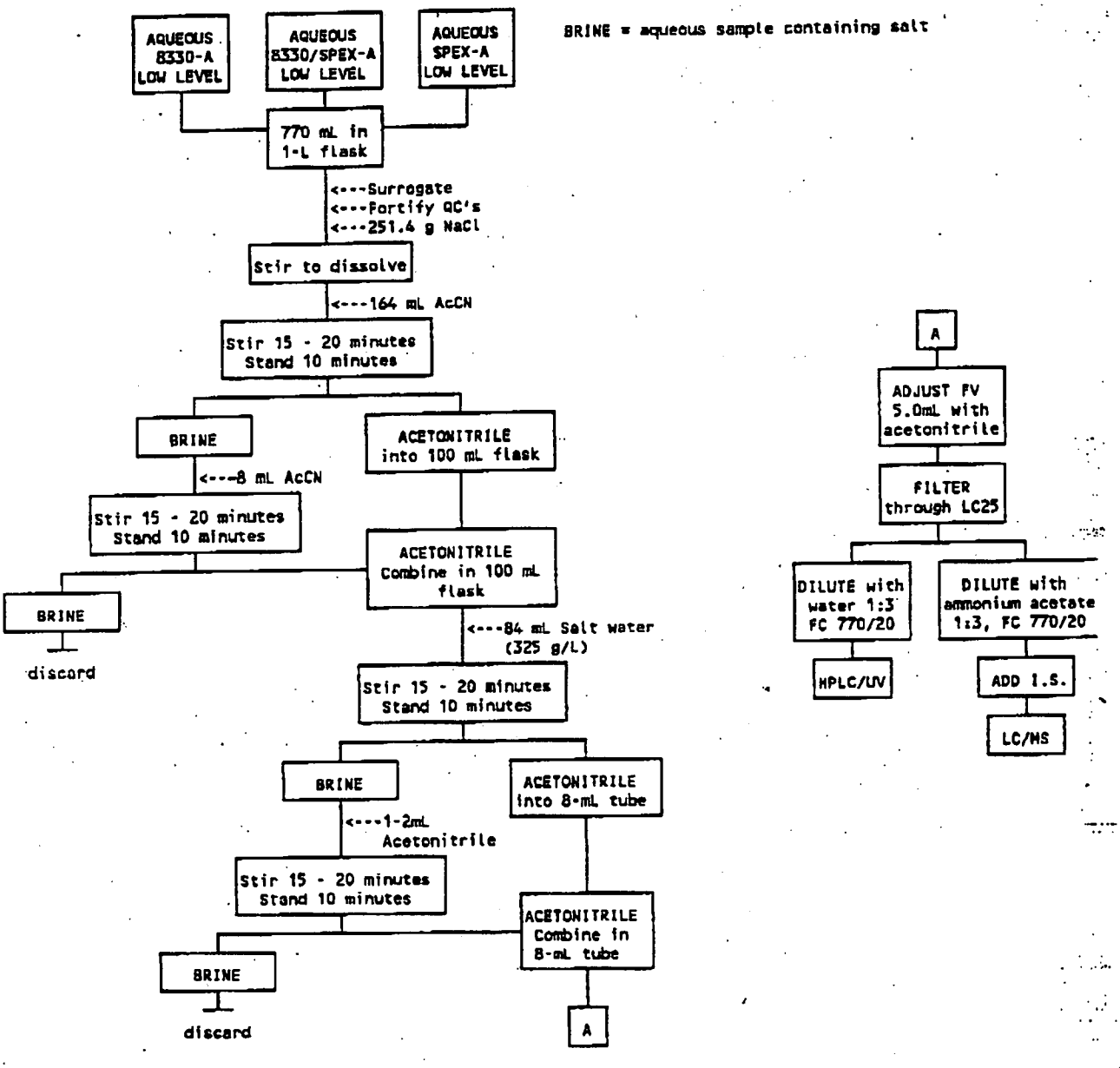
19.4 Aqueous by Salting Out for low levels.



C4-127

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SOP No. SAC-LC-0001
Revision No. 5.0
Date January 3, 1996
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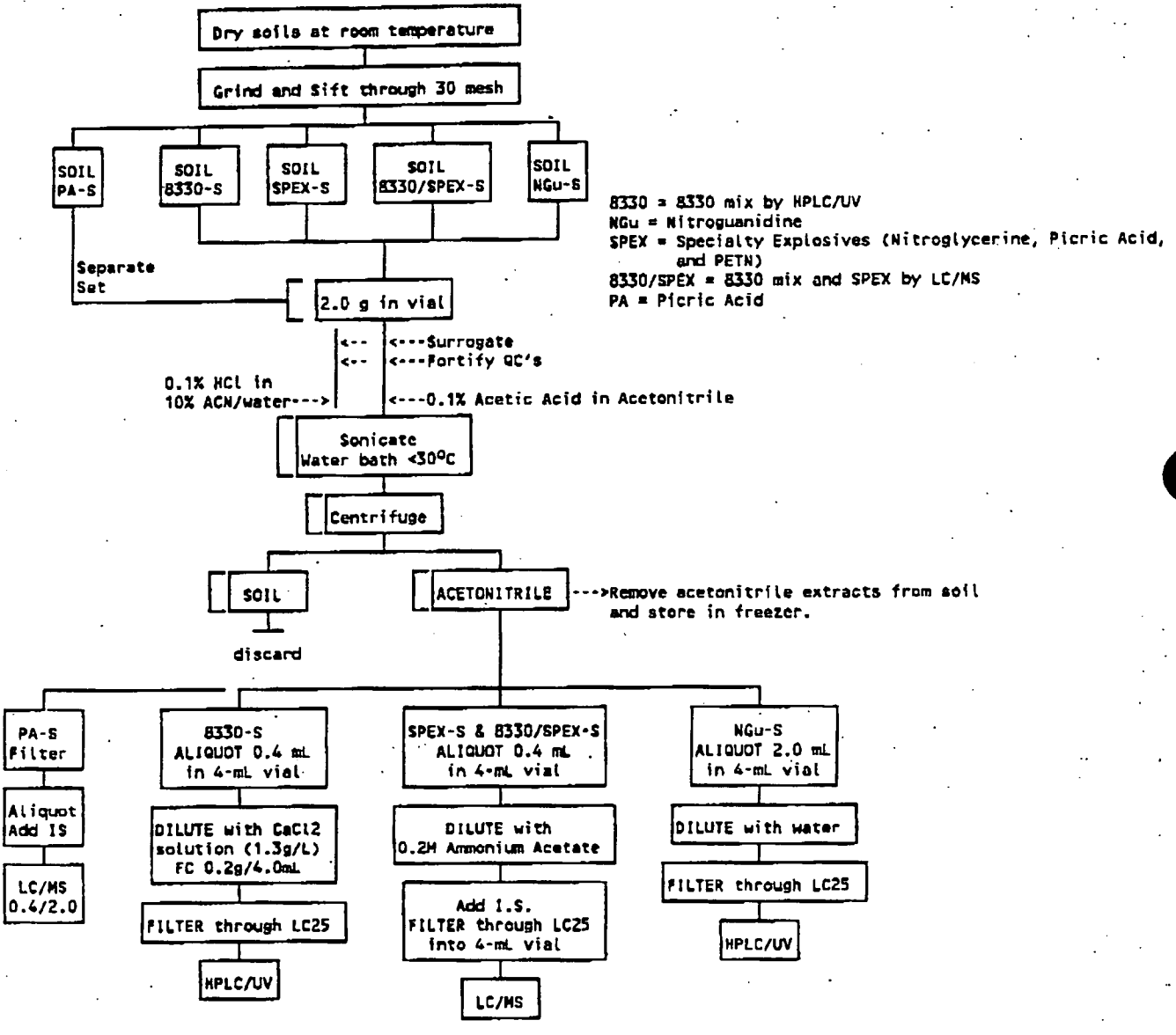
19.5 Soil for 8330 and specialty explosives.



C4-128

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Environmental Services

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20. APPENDIX

A copy of form - STAMDARD SOLUTION MADE FROM NEAT.

A copy of form - STANDARD SOLUTION MADE FROM SOLUTION(S).

A copy of form - MASTER SHEET

A copy of form - HPLC INSTRUMENT LOG

Quanterra / W. Sacramento

CHROMATOGRAPHY RUN LOG

Method: 8330_HY File: [MAY_SV] 4LC Inst: hplc #4 [109]

Calib: 4HY0513I 8330 Curve; 2.5 to 1000 ng/mL Hypersil C8 on 5/13/96

HPLC-UV #4 Phenomenex Hypersil C8 SN114198 150x4.6mm 5um, 8330 Analys

#	SAMPLE NAME	MULT	DIVS	DATE	TIME	LVL
1	Water Blank	1.00	/ 1.000	20-MAY-1996	13:35	
2						
3						
4						
5						
6						
7						
8						
9						
10						
11						
12						
13						
14						
15						
16						
17						
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35						
36						
37						
38						
39						
40						

COLUMN	A =	B =

Analyst: _____ Date: _____ Page: ____

Q/WS Elog 1.1 Printed: 05/20/96 15:14:59

C4-130

MASTER SHEET Holding time Due: _____ PROJECT Due: _____

PROJECT NO. : _____ Page ___ of ___ INITIATED BY: _____ DATE: _____

METHOD NO. : _____ COMPLETED BY: _____ DATE: _____

SAMPLE MATRIX: _____ REVIEWED BY : _____ DATE: _____

COMPOUND(S) : _____ APPROVED BY : _____ DATE: _____

STORAGE								

Comments: _____

CAL ID/SAMPLE DATA	SAMPLE SIZE	FORTIFICATION - STANDARD DATA PPB	COMPOUND(S)	ALIQUOT	STD. CONC.	STD. NO.			

DEQ: BOILER 5-26-94.

C4-131

MAY 05 '98 09:46 FR QUANTERRA 916 372 1059 TO.13606381685 P.63/66

PAGE # 96-_____

QUANTERRA ENVIRONMENTAL SERVICE, WEST SACRAMENTO

ENERGETICS AND SPECIALTY CHEMICALS GROUP (ESC)
STANDARD SOLUTION MADE FROM NEAT

SOLUTION ID#: _____

NAME OF STANDARD: _____

COMPONENT/SOURCE/STOCK#/LOT#/LOCATION/PURITY/EXP.	WEIGHT	FINAL CONC

(Additional components may be listed on back)

DESCRIPTION OF PREPARATION: BALANCE ID# _____

SPECIAL NOTES/COMMENTS:

ACCEPTABLE FOR USE? yes ___ no ___ QC'ed BY/DATE: _____

PREPARED BY/DATE: _____ REVIEWED BY/DATE: _____

STORAGE LOCATION: _____ EXPIRATION DATE: _____

DEG 2-1-96 / QA #359

C4-132

PAGE # 96-_____

QUANTERRA ENVIRONMENTAL SERVICE, WEST SACRAMENTO

ENERGETICS AND SPECIALTY CHEMICALS GROUP (ESC)
STANDARD SOLUTION MADE FROM SOLUTION(S)

SOLUTION ID#: _____

NAME OF MIX: _____

COMPONENT/ID#/LOCATION/CONCENTRATION/SOLVENT/ EXPIRATION	VOLUME USED	FINAL CONC

(Additional components may be listed on back)

DESCRIPTION OF PREPARATION:

_____

_____

_____

SPECIAL NOTES/COMMENTS:

_____

_____

ACCEPTABLE FOR USE? yes ___ no ___ QC'ed BY/DATE: _____

PREPARED BY/DATE: _____ REVIEWED BY/DATE: _____

STORAGE LOCATION: _____ EXPIRATION DATE: _____

DEG 2-1-96 / QA #360

C4-133

QUANTERRA ENVIRONMENTAL SERVICES (West Sacramento)
SOP CHANGE FORM

SOP Number:	SAC-LC-0001
SOP Title:	
SOP Sections Affected by Change:	SECTION 13.3
Reason for Addition or Change:	EQUATION WAS TYPED INCORRECTLY
Change Effective From [Date]:	SEPT. 3, 1997
Change or Addition (Specific Section; Use additional sheets if necessary):	HPLC/UV: AMOUNT FOUND (ppb) = $\frac{A_x \cdot V_x}{CF_c \cdot W_s}$

Submitted by/Date: Dennis E. Gall / 9-3-97
Approved by:*
Technical Reviewer: [signature] Date 9/3/97
EH&S Signature: [signature] for Cheryl [illegible] Date 9/3/97
QA Signature: [signature] Date 9/3/97
Management Signature: [signature] Date 9-3-97
* Must be same signature authorities of SOP being revised.

C4-134

QUANTERRA INC. (West Sacramento)

STANDARD OPERATING PROCEDURE CHANGE FORM

SOP Number:	SAC-LM-0001, Revision 5.0, 07/08/96.
SOP Title:	Determination of Nitroaromatics, Nitramines, and Specialty Explosives in Water and Soil/Sediments by HPLC/UV and by LC/TSP/MS
SOP Sections Affected by Change:	8.5, 12.1.4, 12.2.4, 12.4.4, 12.6.9, and 12.8.3.
Reason for Addition or Change:	Adding PETN with 8330 analytes by HPLC/UV.
Change Effective From [Date]:	May 4, 1998
Change or Addition (Specific Section; use additional sheets if necessary.):	8.5 Analytical Curve for 8330 by HPLC/UV. PETN is added to the 8330 Mix between 40 ng/mL to 1,000 ng/mL. 12.1.4 PETN is added to the LCS and MS/SD, or to the DCS, at 250 ppb by adding 25 uL of the 100 ug/mL standard to 10 mL water for DAI method 12.2.4 PETN is added to the LCS and MS/SD, or to the DCS, at 162 ppb by adding 125 uL of the 100 ug/mL standard to 77 mL water for high level salting method. 12.4.4 PETN is added to the LCS and MS/SD, or to the DCS, at 6.5 ppb by adding 50 uL of the 100 ug/mL standard to 770 mL water for low level salting method. 12.6.9 PETN is added to the LCS and MS/SD, or to the DCS at 5.0 ppm by adding 100 uL of the 100 ug/mL standard to 2.0 gram soil, and the sample is prep as per 8330. 12.8.3 The gradient program is modified for the elution of PETN. At 12 minutes, the solvent composition is 60:5:35, and at 15 minutes the solvent composition is 59:4:37, which is held till the end of the run. After the nitrotolunes elutes, the UV detection is switched at approximately 20.8 minutes to 214 nm to detect PETN at approximately 22.0 minutes.

Submitted by/Date:

C4-135

Approved by:*

Technical Reviewer: Karla Shreddler *Date:* 5/4/98

EH&S Signature: *Date:*

QA Signature: *Date:*

Management Signature: *Date*

**Must be same signature authorities of SOP being revised.*

d:\\qsacca01\sacqs\sop\amend.dot

C4-1369

TABLE 1 REPORTING LIMITS FOR QUANTERRA, WEST SACRAMENTO

Parameter	Method W=Water S=Soil	Analyte	Water (ug/L)	Soil (mg/kg)
SW8321	Explosives by HPLC			
		HMX	13.0	0.25
		sym-Trinitrobenzene	7.3	0.25
		RDX	14.0	0.25
		1,3-Dinitrobenzene	4.0	0.25
		Nitrobenzene	6.4	0.25
		2,4,6-Trinitrotoluene	6.9	0.25
		Tetryl	4.0	0.25
		2,4-Dinitrotoluene	5.7	0.25
		2,6-Dinitrotoluene	9.4	0.25
		2-Am-DNT	10.0	0.25
		4-Am-DNT	10.0	0.25
		2-Nitrotoluene	12.0	0.25
		3-Nitrotoluene	8.5	0.25
		4-Nitrotoluene	7.9	0.25

* The following additional compounds may be added to this method by client request.

		Nitroglycerine	50	0.5
		PETN	50	0.5

C4-136b

TABLE 2 QUANTERRA, WEST SACRAMENTO LABORATORY CONTROL LIMITS FOR MATRIX SPIKES, MATRIX SPIKE DUPLICATES, AND SURROGATE SPIKES [1]

Analytical Method	Spiking Compounds	Spike Concentration Water (ug/L)	Spike Concentration Soil/Sediments (mg/kg)	Laboratory-Established Control Limits Percent Recovery (%) Water	Percent Recovery (%) Soil/Sediments	Relative Percent Difference (%) Water	Relative Percent Difference (%) Soil/Sediments
SW8321	HMX	50	1.0	65-135	65-135	35	35
	sym-Trinitrobenzene	50	1.0	65-135	65-135	35	35
	RDX	50	1.0	65-135	65-135	35	35
	1,3-Dinitrobenzene	50	1.0	65-135	65-135	35	35
	Nitrobenzene	50	1.0	65-135	65-135	35	35
	2,4,6-Trinitrotoluene	50	1.0	65-135	65-135	35	35
	Tetryl	50	1.0	65-135	65-135	35	35
	2,4-Dinitrotoluene	50	1.0	65-135	65-135	35	35
	2.6-Dinitrotoluene	50	1.0	65-135	65-135	35	35
	2-Am-DNT	50	1.0	65-135	65-135	35	35
	4-Am-DNT	50	1.0	65-135	65-135	35	35
	2-Nitrotoluene	50	1.0	65-135	65-135	35	35
	3-Nitrotoluene	50	1.0	65-135	65-135	35	35
	4-Nitrotoluene	50	1.0	65-135	65-135	35	35
	Surrogate: Nitrobenzene-d5	200	5.0	40-135	40-135	35	35
	* The following additional compounds may be added to this method by client request.						
	Nitroglycerine	800	2.0	65-135	65-135	35	35
	PETN	400	1.0	65-135	65-135	35	35

(1) The control limits are subject to change at the discretion of the laboratory.

C4-136C

QUANTERRA STANDARD OPERATING PROCEDURE

TITLE: INDUCTIVELY COUPLED PLASMA-ATOMIC EMISSION SPECTROSCOPY, SPECTROMETRIC METHOD FOR TRACE ELEMENT ANALYSES, SW-846 METHOD 6010B AND EPA METHOD 200.7

(SUPERSEDES: REVISION 1)

Prepared by: Sevda Aleckson and Bill Nash

Reviewed by: _____
Director, Technology

Approved by: _____
Director, Quality Assurance,

Approved by: _____
Director, Environmental Health and Safety,

Approved by: _____
Management,

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C4-138

1. SCOPE AND APPLICATION

1.1. This procedure describes the analysis of trace elements including metals in solution by Inductively Coupled Plasma -Atomic Emission Spectroscopy (ICP-AES) using SW-846 Method 6010B and EPA Method 200.7 . Table I of Appendix A lists the elements appropriate for analysis by Methods 6010B and 200.7. Additional elements may be analyzed under Methods 6010B and 200.7 provided that the method performance criteria presented in Section 13.0 are met.

1.2. ICP analysis provides for the determination of metal concentrations over several orders of magnitude. Detection limits, sensitivity and optimum concentration ranges of the metals will vary with the matrices and instrumentation used. For instance, in comparison to conventional ICP technique, ICP-Trace can achieve detection levels comparable to those determined using the graphite furnace atomic absorption spectroscopy (GFAAS) technique.

1.3. Method 6010B is applicable to the determination of dissolved, suspended, total recoverable and total elements in ground water, aqueous samples, soils, sludges, wastes, sediments, and TCLP, EP and other leachates/extracts. All matrices require digestion prior to analysis with the exception of analyses for dissolved metals in filtered and acidified aqueous samples. Although digestion is not specifically required by the method, some clients and regulators may require digestion of **dissolved samples** and this must be clarified and documented before project initiation. Silver concentrations must be below 2.0 mg/L in aqueous samples and 100 mg/kg in solid matrix samples. Precipitation may occur in samples where silver concentrations exceed these levels and lead to the generation of erroneous data.

1.4. Method 200.7 is applicable to the determination of dissolved, suspended, total recoverable, and total elements in water, waste water, and solid wastes. All matrices require digestion prior to analysis with the exception of analyses for dissolved metals in filtered and acidified aqueous samples if the criteria in Section 11.1 are met. Silver concentrations must be below 0.1 mg/L in aqueous samples and 50 mg/kg in solid matrix samples.

1.5. State-specific requirements may take precedence over this SOP for drinking water sample analyses

2. SUMMARY OF METHOD

2.1. This method describes a technique for the determination of multi elements in solution using sequential or simultaneous optical systems and axial or radial viewing of the plasma. The basis of the method is the measurement of atomic emission by an optical spectroscopic technique. Samples are nebulized and the aerosol that is produced is

transported to the plasma torch where excitation occurs. Characteristic atomic-line emission spectra are produced by a radio frequency inductively coupled plasma (ICP). The spectra are dispersed by a grating spectrometer and the intensities of the emission lines are monitored by photomultiplier tubes. The photocurrents from the photomultiplier tubes are processed and controlled by a computer system. A background correction technique is required to compensate for variable background contribution to the determination of trace elements. Background must be measured adjacent to analyte lines during analysis. The position selected for the background intensity measurement, on either or both sides of the analytical line, will be determined by the complexity of the spectrum adjacent to the analyte line. The position used must be free of spectral interferences and reflect the same change in background intensity as occurs at the analyte wavelength measured. Background correction is not required in cases of line broadening where a background correction measurement would actually degrade the analytical result. The possibility of additional interferences should also be recognized and appropriate actions taken. Alternatively, multivariate calibration methods may be chosen for which point selection for background correction is superfluous since whole spectral regions are processed.

2.2. Refer to the appropriate SOPs for details on sample preparation methods.

3. DEFINITIONS

3.1. Dissolved Metals: Those elements which pass through a 0.45 um membrane. (Sample is acidified after filtration).

3.2. Suspended Metals: Those elements which are retained by a 0.45 um membrane.

3.3. Total Metals: The concentration determined on an unfiltered sample following vigorous digestion.

3.4. Total Recoverable Metals: The concentration determined on an unfiltered sample following treatment with hot, dilute mineral acid.

4. INTERFERENCES

4.1. Spectral, physical and chemical interference effects may contribute to inaccuracies in the determinations of trace elements by ICP. Spectral interferences are caused by:

- Overlap of a spectral line from another element.
- Unresolved overlap of molecular band spectra.
- Background contribution from continuous or recombination phenomena.

- Stray light from the line emission of high concentration elements.

4.1.1. A background correction technique is required to compensate for variable background contribution to the determination of trace elements. Background correction is not required in cases where a background corrective measurement would actually degrade the analytical result.

4.1.2. Inter-element correction factors (IECs) are necessary to compensate for spectral overlap. Inter-element interferences occur when elements in the sample emit radiation at wavelengths so close to that of the analyte that they contribute significant intensity to the analyte channel. If such conditions exist, the intensity contributed by the matrix elements will cause an excessively high (or sometimes low) concentration to be reported for the analyte. Inter-element corrections IECs must be applied to the analyte to remove the effects of these unwanted emissions.

4.1.3. Physical interferences are generally considered to be effects associated with sample transport, nebulization and conversion within the plasma. These interferences may result in differences between instrument responses for the sample and the calibration standards. Physical interferences may occur in the transfer of solution to the nebulizer (e.g., viscosity effects), at the point of aerosol formation and transport to the plasma (e.g., surface tension) or during excitation and ionization processes within the plasma itself. Changes in viscosity and surface tension can cause significant inaccuracies, especially in samples containing high dissolved solids or high acid concentrations. If physical interferences are present, dilution of the sample, use of a peristaltic pump, mass flow controller, use of an internal standard and/or use of a high solids nebulizer can reduce the effect.

4.1.4. Chemical interferences are characterized by molecular compound formation, ionization effects and solute vaporization effects. Normally these effects are not significant with the ICP technique but if observed can be minimized by buffering the sample, matrix matching or standard addition procedures.

5. SAFETY

5.1. Procedures shall be carried out in a manner that protects the health and safety of all Quanterra associates.

5.2. Eye protection that satisfies ANSI Z87.1 (as per the Chemical Hygiene Plan), laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.3. The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the Material Safety Data Sheets (MSDS) maintained in the laboratory.

5.3.1. The following materials are known to be **corrosive**:

sulfuric acid, hydrochloric acid, nitric acid and hydrofluoric acid. (NOTE: sulfuric and hydrofluoric acids are used in cleaning the ICP torch and hydrofluoric acid is also commonly used in air toxics preparations.).

~~1.1.2.~~5.3.2. The following materials are known to be **oxidizing agents**:

nitric acid and hydrogen peroxide.

~~1.1.3.~~5.3.3. The plasma emits strong UV light and is harmful to vision. **NOTE: AVOID looking directly at the plasma.**

5.3.4. The RF generator produces strong radio frequency waves, most of which are unshielded. People with pacemakers should not go near the instrument while in operation.

5.4. Exposure to chemicals must be maintained **as low as reasonably achievable**, therefore, unless they are known to be non-hazardous, all samples should be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Metals digestates can be processed outside of a fume hood. Solvent and waste containers will be kept closed unless transfers are being made.

5.5. The preparation of standards and reagents will be conducted in a fume hood or well ventilated area.

5.6. All work must be stopped in the event of a known or potential compromise to the health and safety of a Quanterra associate. The situation must be reported **immediately** to a laboratory supervisor.

5.7. The use of hydrofluoric acid requires special safety precautions. Consult the facility EH&S Manager and laboratory supervisor for guidance.

6. EQUIPMENT AND SUPPLIES

6.1. Inductively Coupled Plasma Atomic Emission Spectrometer equipped with autosampler and background correction.

6.2. Radio Frequency Generator.

6.3. Argon gas supply, welding grade or equivalent.

6.4. Coolflow or appropriate water cooling device.

6.5. Peristaltic Pump.

6.6. Calibrated automatic pipettes or Class A glass volumetric pipettes.

6.7. Class A volumetric flasks.

6.8. Autosampler tubes.

7. REAGENTS AND STANDARDS

7.1. Intermediate standards are purchased as custom Quanterra multi-element mixes or as single-element solutions. All standards must be stored in FEP fluorocarbon or unused polyethylene or polypropylene bottles. Intermediate standard solutions must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the intermediate solutions may be used for up to one year and must be replaced sooner if verification from an independent source indicates a problem. Expiration dates can be extended provided that the acceptance criteria described in laboratory-specific SOPs are met.

7.2. Working calibration and calibration verification solutions may be used for up to 3 months and must be replaced sooner if verification from an independent source indicates a problem. Standards should be prepared in a matrix of 5% hydrochloric and 5% nitric acids. An exception to this is in the event the Trace ICP is utilized without the internal standard. In this case, the standard acid matrix must be matched to the final preparation matrix as listed in Section 11.10.

7.3. Refer to Tables III, IV, IVA, V and VI (Appendix A) for details regarding the working standard concentrations for calibration, calibration verification, interference correction and spiking solutions.

7.4. Concentrated nitric acid (HNO_3), trace metal grade or better.

7.5. Concentrated hydrochloric acid (HCl), trace metal grade or better.

7.6. Reagent water must be produced by a Millipore DI system or equivalent. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Sample holding times for metals are six months from time of collection to the time of analysis.

8.2. Aqueous samples are preserved with nitric acid to a pH of <2 and may be stored in either plastic or glass. If boron or silica are to be determined, plastic containers are preferred. Refrigeration is not required. Preservation must be verified prior to analysis. For samples that will be analyzed by Method 200.7 for compliance with Safe Drinking Water regulations, the samples must be held for a minimum of 16 hours prior to verifying the pH.

8.3. Soil samples do not require preservation but must be stored at 4°C ± 2° until the time of preparation .

9. QUALITY CONTROL

Table VII (Appendix A) provides a summary of quality control requirements including type, frequency, acceptance criteria and corrective action.

~~1.1.~~9.1. Initial Demonstration of Capability

Prior to analysis of any analyte using either Method 200.7 or Method 6010B, the following requirements must be met.

~~1.1.1.~~9.1.1. Instrument Detection Limit (IDL) - The IDL for each analyte must be determined for each analyte wavelength used on each instrument. The IDL must be determined annually. If the instrument is adjusted in anyway that may affect the IDL, the IDL for that instrument must be redetermined. The IDL shall be determined by multiplying by 3, the standard deviation obtained from the analysis of a standard solution (each analyte in reagent water) at a concentration 3x - 5x the previously determined IDL, with seven consecutive measurements. Each measurement must be performed as though it were a separate analytical sample (i.e., each measurement must be followed by a rinse and/or any other procedure performed between the analysis of separate samples). The result of the IDL determination must be below the Quanterra reporting limit. The CLP IDL procedure can be used for this method.

9.1.2. Method Detection Limit (MDL) - An MDL must be determined for each analyte prior to the analysis of any client samples. The MDL is determined using seven replicates of reagent water, spiked with all the analytes of interest, that have been carried through the entire analytical procedure. MDLs must be redetermined on an annual basis in accordance with 40 CFR Part 136 Appendix B requirements as detailed in Quanterra QA Policy QA-005. The spike level must be between the calculated MDL and 10X the MDL to be considered valid. The result of the MDL determination must be below the Quanterra reporting limit (RL). MDL studies for the determination of metals in soil need not be performed; an appropriate soil MDL may be computed from the experimentally determined MDL for metals in aqueous solution.

9.1.3. Linear Range Verification (LR) - The linear range must be determined on an annual basis for each analyte wavelength used on each instrument. The linear range is the concentration above which results cannot be reported without dilution of the sample. The standards used to define the linear range limit must be analyzed during a routine analytical run. For the **initial** determination of the upper limit of the linear dynamic range (LDR) for each wavelength, determine the signal responses from a minimum of three to five different concentration standards across the estimated range. One standard should be near the upper limit of the estimated range. The concentration measured at the LDR must be no more than 10% less than the expected level extrapolated from lower standards. If the instrument is adjusted in any way that may affect the LRs, new dynamic ranges must be determined. The LR data must be documented and kept on file.

9.1.4. Background Correction Points - To determine the appropriate location for off-line background correction when establishing methods, the user must scan the area on either side adjacent to the wavelength and record the apparent emission intensity from all other method analytes. This spectral information must be documented and kept on file. The location selected for background correction must be either free of off-line interelement spectral interference or a

computer routine must be used for automatic correction on all determinations. Tests to determine spectral interference must be done using analyte concentrations that will adequately describe the interference. Background correction points must be set prior to determining IECs. Refer to the facility-specific instrument operation SOP and ICP instrument manual for specific procedures to be used in setting background correction points.

9.1.5. Inter-element Corrections (IECs) - ICP interelement correction factors must be determined prior to the analysis of samples and every six months thereafter. If the instrument is adjusted in any way that may affect the IECs, the IECs must be redetermined. When initially determining IECs for an instrument, wavelength scans must be performed to ensure that solutions in use are free from contaminants. If an IEC varies significantly from the previously determined IEC then the possibility of contamination should be investigated. The purity of the IEC check solution can be verified by using a standard from a second source or an alternate method (i.e., GFAA or ICP-MS). Published wavelength tables (e.g. MIT tables, Inductively Coupled Plasma-Atomic Spectroscopy: Prominent Lines) can also be consulted to evaluate the validity of the IECs. Refer to the facility specific instrument operation SOP and instrument manufacturer's recommendations for specific procedures to be used in setting IECs. An IEC must be established to compensate for any interelement interference which results in a false analyte signal greater than ± the RL as defined in Tables I, IA or II. To determine IECs, run a single element standard at the established linear range. To calculate an IEC, divide the observed concentration of the analyte by the observed concentration of the "interfering element."

Note: Trace ICP IECs are more sensitive to small changes in the plasma and instrument setup conditions. Adjustments in the IECs will be required on a more frequent basis for the Trace as reflected by the ICSA response.

~~1.1.6.~~9.1.6. Rinse Time Determination - Rinse times must be determined annually. To determine the appropriate rinse time for a particular ICP system, the linear range verification standard (see 9.1.3) should be aspirated as a regular sample followed by the analysis of a series of rinse blanks. The length of time required to reduce the analyte signals to < RL will define the rinse time for a particular ICP system. For some analytes it may be impractical to set the rinse time based on the linear range standard result (i.e., analyte not typically detected in environmental samples at that level and an excessive rinse time would be required at the linear range level). Until the required rinse time is established, the method recommends a rinse period of at least 60 seconds between samples and standards. If a memory effect is suspected, the sample must be reanalyzed after a rinse period of sufficient length. Rinse time studies can be conducted at additional concentration levels. These additional studies

must be documented and kept on file, if a concentration other than the linear range level is used to set the rinse time. The concentration levels used to establish the rinse time must be taken into consideration when reviewing the data.

9.2. Method Blank (MB) - One method blank must be processed with each preparation batch. The method blank consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. The method blank should not contain any analyte of interest at or above the reporting limit (exception: common laboratory contaminants, see below) or at or above 5% of the measured concentration of that analyte in associated samples, whichever is higher (sample result must be a minimum of 20x higher than the blank contamination level).

- If the analyte is a common laboratory contaminant (copper, iron, lead (Trace only) or zinc) the data may be reported with qualifiers if the concentration of the analyte in the method blank is less than two times the RL. **Such action must be taken in consultation with the client and must be addressed in the project narrative.**

- Repreparation and reanalysis of all samples associated with an unacceptable method blank is required when reportable concentrations are determined in the samples (see exception noted above).

- If there is no analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. **Such action must be taken in consultation with the client and must be addressed in the project narrative.**

- If the above criteria are not met and reanalysis is not possible, then the sample data must be qualified. **This anomaly must be addressed in the project narrative and the client must be notified.**

- For dissolved metals samples which have not been digested, a CCB result is reported as the method blank. The CCB run immediately prior to the start of the dissolved sample analyses must be used for this purpose. No more than 20 samples can be associated with one CCB.

9.3. Laboratory Control Sample (LCS) - One aqueous LCS must be processed with each preparation batch. The LCS must contain all analytes of interest and must be carried through the entire analytical procedure. Aqueous LCS spike levels are provided in Table III (Appendix A). The LCS is used to monitor the accuracy of the analytical process. On-going monitoring of the LCS results provides evidence that the

laboratory is performing the method within acceptable accuracy and precision guidelines.

- If any analyte is outside established control limits the system is out of control and corrective action must occur. Until in-house control limits are established, for method 6010B, a control limit of 80 - 120% (85-115% for 200.7) recovery must be applied.

- In the event that an MS/MSD analysis is not possible a Laboratory Control Sample Duplicate (LCSD) must be analyzed. The RPD of the LCS and LCSD must be compared to the matrix spike RPD limits.

- In the instance where the LCS recovery is greater than 120% (115% for 200.7) and the sample results are < RL, the data may be reported with qualifiers. **Such action must be taken in consultation with the client and must be addressed in the report narrative.**

- Corrective action will be repreparation and reanalysis of the batch unless the client agrees that other corrective action is acceptable.

- For dissolved metals samples which have not been digested, a CCV result is reported as the LCS. The CCV run immediately prior to the start of the dissolved sample analyses must be used for this purpose. No more than 20 samples can be associated with one CCV.

9.4. Matrix Spike/Matrix Spike Duplicate (MS/MSD) - One MS/MSD pair must be processed for each preparation batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Some client specific data quality objectives (DQO's) may require the use of sample duplicates in place of or in addition to MS/MSDs. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, these results may have immediate bearing only on the specific sample spiked. Samples identified as field blanks cannot be used for MS/MSD analysis. Spiking levels are provided in Tables III and VI (Appendix A).

- If any analyte recovery or RPD falls outside the acceptance range, the recovery of that analyte must be in control for the LCS. For both methods 200.7 and 6010B, control limits of 75 - 125% recovery and 20% RPD or historical acceptance criteria must be applied to the MS/MSD. If the LCS recovery is within limits, then the laboratory operation is in control and the results may be accepted. If the recovery of the LCS is outside limits corrective action must be taken. Corrective

action will include repreparation and reanalysis of the batch. MS/MSD results which fall outside the control limits must be addressed in the narrative.

- If the native analyte concentration in the MS/MSD exceeds 4x the spike level for that analyte, the recovery data are reported as NC (i.e., not calculated). If the reporting software does not have the ability to report NC then the actual recovery must be reported and narrated as follows: "Results outside of limits do not necessarily reflect poor method performance in the matrix due to high analyte concentrations in the sample relative to the spike level."

- If an MS/MSD is not possible due to limited sample volume then a laboratory control sample duplicate (LCSD) should be analyzed. The RPD of the LCS and LCSD must be compared to the matrix spike RPD limits.

- For dissolved metals samples which have not been digested, a MS/MSD must be performed per batch of up to 20 samples by spiking two aliquots of the sample at the levels specified in Table III (Appendix A).

9.5. Dilution test – A dilution test is performed to determine whether significant physical or chemical interferences exist due to the sample matrix. One sample per preparation batch must be processed as a dilution test. The test is performed by running a sample at a 5x (1:4) dilution. Samples identified as field blanks cannot be used for dilution tests. The results of the diluted sample, after correction for dilution, should agree within 10% of the original sample determination when the original sample concentration is greater than 50x the IDL. If the results are not within 10%, the possibility of chemical or physical interference exists.

9.6. Initial Calibration Verification (ICV/ICB) - Calibration accuracy is verified by analyzing a second source standard (ICV). For analyses conducted under Method 200.7, the ICV result must fall within 5% of the true value for that solution with relative standard deviation <3% from replicate (minimum of two) exposures. For Method 6010B, the ICV must fall within 10% of the true value for that solution with relative standard deviation <5% from replicate (minimum of two) exposures. An ICB is analyzed immediately following the ICV to monitor low level accuracy and system cleanliness. The ICB result must fall within +/- the RL from zero. If either the ICV or ICB fail to meet criteria, the analysis should be terminated, the problem corrected, the instrument recalibrated and the calibration reverified. (See Section 11.11 or 11.12 for required run sequence).

9.7. Continuing Calibration Verification (CCV/CCB) - Calibration accuracy is monitored throughout the analytical run through the analysis of a known standard after every 10 samples. The CCV is be a mid-range standard made from a dilution of the calibration standard. The CCV for both methods must fall within 10% of the true value for that solution with relative standard deviation <5% from replicate (minimum of two) exposures. A CCB is analyzed immediately following each CCV. (See

Section 11.11 or 11.12 for required run sequence.) The CCB result must fall within +/- RL from zero. If the blank is less than 1/10 the concentration of the action level of interest, and no sample is within 10% of the action limit, reanalysis and recalibration are not required before continuation of the run. Sample results may only be reported when bracketed by valid CCV/CCB pairs. If a mid-run CCV or CCB fails, the analysis for the affected element must be terminated, the problem corrected, the instrument recalibrated, the calibration verified and the affected samples reanalyzed. (Refer to Section 11.13 for an illustration of the appropriate rerun sequence).

9.8. Interference Check Analysis (ICSA/ICSAB) - The validity of the interelement correction factors is demonstrated through the successful analysis of interference check solutions. The ICSA contains only interfering elements, the ICSAB contains analytes and interferents. Refer to Table V (Appendix A) for the details of ICSA and ICSAB composition. Custom Quanterra multielement ICS solutions must be used. All analytes should be spiked into the ICSAB solution, therefore, if a non-routine analyte is required then it should be manually spiked into the ICSAB using a certified ultra high purity single element solution or custom lab-specific mix. If the ICP will display overcorrection as a negative number then the non-routine elements can be controlled from the ICSA as described in section 9.8.3. Elements known to be interferents on a required analyte must be included in the ICP run when that analyte is determined. Aluminum, iron, calcium and magnesium must always be included in all ICP runs.

9.8.1. The ICSA and ICSAB solutions must be run at the beginning of the run. (See Section 11.11 or 11.12 for required run sequence.)

9.8.2. The ICSAB results for the interferents must fall within 80 - 120% of the true value. If any ICSAB interferent result fails criteria, the analysis should be terminated, the problem corrected, the instrument recalibrated and the samples rerun.

9.8.3. ICSA results for the non-interfering elements with reporting limits ≤ 10 ug/L must fall within the Quanterra guidelines of ± 2x RL from zero. ICSA results for the non-interfering elements with RLs> 10 µg/L must fall within the Quanterra guidelines of ± 1x RL from zero. If the ICSA results for the non-interfering elements do not fall within +/- 2x RL (RL ≤10) or ± 1xRL (RL>10) from zero the field sample data must be evaluated as follows:

- If the non-interfering element concentration in the ICSA is the result of contamination versus a spectral interference, and this reason is documented, the field sample data can be accepted.
- If the affected element was not required then the sample data can be accepted.

- If the interfering elements are not present in the field sample at a concentration which would result in a false positive or negative result greater than +/- 2x RL from zero then the field sample data can be accepted.

- If the interfering element is present in the field sample at a level which would result in a false analyte signal greater than ± 2x RL from zero, the data can be accepted only if the concentration of the affected analyte in the field sample is more than 10x the analyte signal in the ICSA.

- If the data does not meet the above conditions then the IECs must be re-evaluated and corrected if necessary and the affected samples reanalyzed or the sample results manually corrected through application of the new IEC to the raw results. If the results are recalculated manually the calculations must be clearly documented on the raw data.

9.9. Method of Standard Addition (MSA) -This technique involves adding known amounts of standard to one or more aliquots of the processed sample solution. This technique compensates for a sample interferent that may enhance or depress the analyte signal, thus producing a different slope from that of the calibration standards. It will not correct for additive interferences which cause a baseline shift. Refer to Section 11.17 for additional information on when MSA is required as well as Appendix D for specific MSA requirements.

9.10. Quality Assurance/Project Summaries - Certain clients may require project- or program-specific QC which may supersede this SOP requirements. Quality Assurance Summaries (QASs) or equivalent documents providing project-specific requirements should be developed so that project staff clearly understand the special project requirements.

10. CALIBRATION AND STANDARDIZATION

10.1. Set up the instrument with the operating parameters recommended by the manufacturer. Allow the instrument to become thermally stable before beginning calibration (approximately 30 minutes of warm-up is required).

10.2. Profile and calibrate the instrument according to the instrument manufacturer's recommended procedures. Flush the system with the calibration blank between each standard or as the manufacturer recommends. The calibration curve must consist of a minimum of a blank and a standard. Refer to the facility-specific instrument SOP or ICP instrument manual for a detailed set up and operation protocols.

10.3. Calibration must be performed daily and each time the instrument is set up. Instrument runs may be continued over periods exceeding 24 hours as long as all

calibration verification (CCV) and interference check QC criteria are met. The instrument standardization date and time must be included in the raw data.

10.4. Refer to Section 9.0 for calibration verification procedures, acceptance criteria and corresponding corrective actions.

11. PROCEDURE

11.1. For 200.7 analyses, dissolved (preserved) samples must be digested unless it can be documented that the sample meets all of the following criteria:

A. Visibly transparent with a turbidity measurement of 1 NTU or less.

B. Is of one liquid phase and free of particulate or suspended matter following acidification.

C. Is NOT being analyzed for silver.

~~1.2.~~11.2. A minimum of two exposures for each standard, field sample and QC sample is required. The average of the exposures is reported. For Trace ICP analyses, the results of the sum channel must be used for reporting.

11.3. Prior to calibration and between each sample/standard the system is rinsed with the calibration blank solution. The minimum rinse time between analytical samples is 60 seconds unless following the protocol outlined in 9.1.6 it can be demonstrated that a shorter rinse time may be used. Triton-X can be added to the rinse solution to facilitate the rinse process.

11.4. The use of an autosampler for all runs is strongly recommended.

11.5. The use of automated QC checks through the instrument software is highly recommended for all calibration verification samples (ICV,CCV), blanks (ICB,CCB,PB), interference checks (ICSA,ICSAB) and field samples (linear range) to improve the data review process.

11.6. To facilitate the early identification of QC failures and samples requiring rerun it is strongly recommended that sample data be reviewed periodically throughout the run.

11.7. To facilitate the data review and reporting processes it is strongly recommended that all necessary dilutions be performed before closing out the instrument run.

11.8. For unattended overnight auto-runs it is strongly recommended that the frequency of ICSA/ICSAB analysis be increased to every 4 hours.

11.9. The use of an internal standard is recommended on the conventional, non-Trace ICPs as an alternative to using the method of standard additions. This technique is useful

in overcoming matrix interferences especially in high solids matrices. However, for conventional ICP techniques, internal standards may not be necessary provided that one of the following is performed to minimize physical interferences: (1) peristaltic pump is used, (2) high solids nebulizer is used, or (3) high solids samples are diluted and reanalyzed.

11.10. The use of an internal standard is **required** on the Trace ICP unless the calibration and QC standards are matrix matched to each digestion procedure used as follows:

Preparation Method	% HNO_3	% HCl
CLP Aqueous	1	5
CLP Soil	5	2.5
SW846 3050	5	10
SW846 3005	2	5
SW846 3010	3	5

The following procedural guidelines must be followed when using an internal standard:

~~1.1.1.~~11.10.1. Typically used internal standards are: yttrium or scandium. (Note: Any element can be used that is not typically found in environmental samples at a high rate of occurrence.)

11.10.2. The internal standard (IS) must be added to every sample and standard at the same concentration. It is recommended that the IS be added to each analytical sample automatically through use of a third pump channel and mixing coil. Internal standards should be added to blanks, samples and standards in a like manner, so that dilution effects resulting from the addition may be disregarded.

11.10.3. The concentration of the internal standard should be sufficiently high to obtain good precision in the measurement of the IS analyte used for data correction and to minimize the possibility of correction errors if the IS analyte is naturally present in the sample.

11.10.4. The internal standard raw intensity counts must be printed on the raw data.

11.10.5. The analyst must monitor the response of the internal standard throughout the sample analysis run. This information is used to detect potential problems and identify possible background contributions from the sample (i.e., natural occurrence of IS analyte).

11.10.5.1. If the internal standard counts fall within ±30% of the counts observed in the ICB then the data is acceptable.

11.10.5.2. If the internal standard counts in the field samples are more than ±30% higher than the expected level, the field samples must then be:

(1) Diluted and reanalyzed;

(2) The IS concentrations must be raised; or

(3) A different internal standard must be used.

~~1.11.~~11.11. The following analytical sequence must be used for Methods 6010B and 200.7:

Instrument Calibration
ICV
ICB
ICSA
ICSAB
8 samples
CCV
CCB
10 samples
CCV
CCB
Repeat sequence of up to 10 samples between CCV/CCB pairs as required to complete run
CCV
CCB

Refer to Quality Control Section 9.0 and Table VII (Appendix A) for Method 6010B and 200.7 quality control criteria.

~~1.12.~~11.12. Additional quality control analyses are necessary for analysis under the Contract Laboratory Program (CLP). If these are included then CLP, 6010 and 200.7 samples can be included in the same sequence. Refer to CORP-MT-002 for details.

11.13. Full method required QC must be available for each wavelength used in determining reported analyte results.

11.14. Guidelines are provided in the appendices on procedures to minimize contamination of samples and standards, preventive maintenance and troubleshooting.

11.15. All measurements must fall within the defined linear range where spectral interference correction factors are valid. Dilute and reanalyze all samples for required analytes that exceed the linear range or use an alternate wavelength for which QC data are established. If an interelement correction exists for an analyte which exceeds the linear range, the IEC may be inaccurately applied. Therefore, even if an overrange analyte may not be required to be reported for a sample, if that analyte is a interferent for any requested analyte in that sample, the sample must be diluted. Acid strength must be maintained in the dilution of samples.

11.16. For TCLP samples, full four-point MSA will be required if all of the following conditions are met:

1) recovery of the analyte in the matrix spike is not at least 50%,

2) the concentration of the analyte does not exceed the regulatory level, and,

3) the concentration of the analyte is within 20% of the regulatory level.

The reporting and regulatory limits for TCLP analyses as well as matrix spike levels are detailed in Table VI (Appendix A). Appendix E provides guidance on performing MSA analyses.

~~1.17.~~11.17. Any variation in procedure shall be completely documented using instrument run logs, maintenance logs, report narratives, a Nonconformance Memo, or an anomaly report and is approved by a Supervisor/Group Leader and QA Manager. If contractually required, the client shall be notified by the Project Manager.

11.18. Nonconformance documentation shall be filed in the project file.

11.19. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

12. DATA ANALYSIS AND CALCULATIONS

12.1. ICV percent recoveries are calculated according to the equation:

$$\%R = 100\left(\frac{Found(ICV)}{True(ICV)}\right)$$

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~~1.2.~~12.2. CCV percent recoveries are calculated according to the equation:

$$\%R = 100\left(\frac{Found(CCV)}{True(CCV)}\right)$$

~~1.3.~~12.3. Matrix Spike Recoveries are calculated according to the following equation:

$$\%R = 100\left(\frac{SSR - SR}{SA}\right)$$

Where:
SSR = Spike Sample Result
SR = Sample Result
SA = Spike Added

~~1.4.~~12.4. The relative percent difference (RPD) of matrix spike/matrix spike duplicates are calculated according to the following equations:

$$RPD = 100\left[\frac{|MSD - MS|}{\left(\frac{MSD + MS}{2}\right)}\right]$$

Where:
MS = determined spiked sample concentration
MSD = determined matrix spike duplicate concentration

~~1.5.~~12.5. The final concentration for a digested aqueous sample is calculated as follows:

$$mg/L = \frac{C \times V1 \times D}{V2}$$

Where:
C = Concentration (mg/L) from instrument readout
D = Instrument dilution factor
V1 = Final volume in liters after sample preparation
V2 = Initial volume of sample digested in liters

~~1.6.~~12.6. The final concentration determined in digested solid samples when reported on a dry weight basis is calculated as follows:

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$$mg / Kg, dry\ weight = \frac{C \times V \times D}{W \times S}$$

Where:

C = Concentration (mg/L) from instrument readout
D = Instrument dilution factor
V = Final volume in liters after sample preparation
W = Weight in Kg of wet sample digested
S = Percent solids/100

Note: A Percent Solids determination must be performed on a separate aliquot when dry weight concentrations are to be reported. If the results are to be reported on wet weight basis the "S" factor should be omitted from the above equation.

~~1.7.~~12.7. The LCS percent recovery is calculated according to the following equation:

$$\%R = 100\left(\frac{Found(LCS)}{True(LCS)}\right)$$

~~1.8.~~12.8. The dilution test percent difference for each component is calculated as follows:

$$\%Difference = \frac{|I - S|}{I} \times 100$$

Where:

I = Sample result (Instrument reading)
S = Dilution test result (Instrument reading × 5)

~~1.9.~~12.9. Appropriate factors must be applied to sample values if dilutions are performed.

12.10. Sample results should be reported with up to three significant figures in accordance with the Quanterra significant figure policy.

13. METHOD PERFORMANCE

13.1. Each laboratory must have initial demonstration of performance data on file for each analyte of interest as described in Section 9.0.

13.2. Refer to Tables I, IA & II in Appendix A for the list of Method 6010B and 200.7 analytes as well as additional analytes that may be analyzed using this SOP.

13.3. Method performance is determined by the analysis of MS and MSD samples as well as method blanks and laboratory control samples. The MS or MSD recovery should fall within +/- 25 % and the MS/MSD should compare within 20% RPD or within the laboratory's historical acceptance limits. These criteria apply to analyte concentrations greater than or equal to 10xIDL. Method blanks must meet the criteria specified in Section 9.2. The laboratory control samples should recover within 20% (15% for 200.7) of the true value or within the laboratory's historical acceptance limits.

13.4. Training Qualification:

The group/team leader or the supervisor has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience.

14. POLLUTION PREVENTION

14.1. This method does not contain any specific modifications that serve to minimize or prevent pollution.

15. WASTE MANAGEMENT

15.1. Waste generated in the procedure must be segregated and disposed of according to the facility hazardous waste procedures and per the local, state, and federal regulations. The Environmental Health and Safety Director should be contacted, if additional information is required.

15.2. Standards should be purchased and prepared in volumes consistent with laboratory use to minimize the volume of expired standards to be disposed.

16. REFERENCES

16.1. 40 CFR Part 136, Appendix B, 7-5-95, Determination of Method Detection Limits.

16.2. Test Methods for Evaluating Solid Waste , Physical/Chemical Methods, SW-846, 3rd Edition, Final Update III, Revision 2, December 1996. Method 6010B.

16.3. Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma-Atomic Emission Spectrometry, Revision 4.4, May 1994. Method 200.7.

16.4. CORP-MT-0002, Inductively Coupled Plasma-Atomic Emission Spectroscopy, Method 200.7 & CLP-M, SOW ILMO3.0.

16.5. QA-003, Quanterra QC Program.

16.6. QA-004, Rounding and Significant Figures.

16.7. QA-005, Method Detection Limits.

17. MISCELLANEOUS (TABLES, APPENDICES, ETC.)

17.1. Modifications/Interpretations from reference method

17.1.1. Modifications/interpretations from both Methods 6010B and 200.7.

17.1.1.1. Quanterra laboratories use mixed calibration standard solutions purchased from approved vendors instead of using individual mixes prepared in house as recommended by the subject methods.

17.1.1.2. The alternate run sequence presented in Section 11.12 is consistent with method requirements. Additional QC (i.e., ICSA) analyses were added to accommodate the CLP protocol requirements.

17.1.1.3. Methods 200.7 and 6010B state that if the correction routine is operating properly, the determined apparent analyte(s) concentration from analysis of each interference solution should fall within a specific concentration range around the calibration blank. In determining IECs, because of lack of definition clarification for "concentration range around the calibration blank," Quanterra has adopted the procedure in EPA CLP ILMO4.0.

17.1.1.4. Section 8.5 of Method 6010B and Section 9.5 of Method 200.7 recommend that whenever a new or unusual matrix is encountered, a series of tests be performed prior to reporting concentration data for that analyte. The dilution test helps determine if a chemical or physical interference exists. Because Quanterra laboratories receive no prior information from clients regarding when to expect a new or unusual matrix, Quanterra may select to perform a dilution test on one sample in each prep batch. According to the method, the post digestion spike (PDS) determines any potential matrix interferences. At Quanterra labs, matrix interference is determined by evaluating data for the LCS and MS/MSD. Quanterra requires documented, clear guidance when a new or unusual matrix will be received for a project and a request to perform the dilution test or PDS on a client-identified sample.

17.1.2. Modifications from Method 200.7.

17.1.2.1. Method 200.7 defines the IDL as the concentration equivalent to a signal, due to the analyte, which is equal to three times the standard deviation of a series of ten replicate measurements of the calibration blank signal at the same wavelength. Quanterra labs utilize the CLP IDL definition as defined in Section 9.1.1 of this SOP.

17.1.2.2. The calibration blank is prepared in an acid matrix of 5% HNO_3/5% HCl instead of the specified 2% HNO_3/10% HCl matrix as the former matrix provides for improved performance relative to the wide variety of digestate acid matrices which result from the various EPA preparation protocols applied.

17.1.2.3. Method section 9.3.4 specifies that "Analysis of the IPC (ICSA/AB) solution immediately following calibration must verify that the instrument is within ± 5% of calibration with a relative standard deviation <3% from replicate integrations ≥ 4." Quanterra uses a minimum of two exposures.

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17.1.2.4. Section 7.12 of 200.7 indicates that the QCS (ICV) should be prepared at a concentration near 1 ppm. The ICV specified in this SOP accommodates the 1 ppm criteria for the majority of analytes. For the remaining analytes, this SOP specifies ICV concentrations which are appropriate to the range of calibration. The intent of the ICV, verification of calibration standard accuracy, is independent of the ICV concentration used.

17.1.2.5. The ICS criteria applied by this SOP differ from those stated in the method. Method 200.7 section 10.4 states that results should fall within the established control limits of 3 times the standard deviation of the calibration blank for that analyte. The control limits listed in this SOP are those applicable to the EPA designed solution.

17.1.2.6. Method 200.7 section 9.3.4 states the CCB should be less than the IDL, but > the lower 3-sigma control limit of the calibration blank. The intent of this requirement is to ensure that the calibration is not drifting at the low end. Quanterra has adopted an absolute control limit of +/- RL from zero for calibration blank criteria. SOP section 9.7 provides the detailed corrective action criteria that must be followed.

17.1.3. Modifications from Method 6010B.

17.1.3.1. Chapter 1 of SW-846 states that the method blank should not contain any analyte of interest at or above the MDL. This SOP states that the method blank must not contain any analyte of interest at or above the reporting limit. Common lab contaminants are allowed up to two times the reporting limit in the blank following consultation with the client.

17.1.3.2. Method 6010B section 8.6.1.3 states that the results of the calibration blank are to agree within 3x the IDLIf not, repeat the analysis two or more times and average the results. If the average is not within three standard deviation of the background mean, terminate the analysis, correct the problem, recalibrate, and reanalyze the previous 10 samples. The intent of this requirement is to ensure that the calibration is not drifting at the low end. Quanterra has adopted an absolute control limit of +/- RL from zero for calibration blank criteria. See SOP Section 9.7 for a detailed description of the required corrective action procedures.

17.2. Modifications from previous SOP

Refer to revision 1 of this SOP.

~~1.3.~~17.3. Facility-Specific SOPs

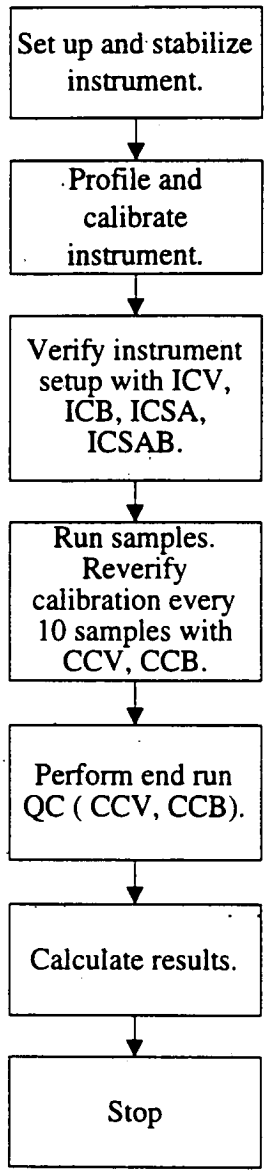
Each facility shall review and revise as appropriate this SOP to reflect any facility-specific requirementsIf no facility-specific amendments are required, the SOP can be adopted as is.

~~1.4.~~17.4. Documentation and Record Management

The following documentation comprises a complete ICP raw data package:

- Raw data (direct instrument printout).
- Relevant sample preparation benchsheets.
- Run log printout from instrument software where this option is available (TJA) or manually generated run log (i.e., Ward WSL printout).
- Data review checklist - See Appendix B.
- Standards documentation (including prep and expiration dates, source, and lot #).
- Nonconformance/anomaly documentation (if applicable).

17.5. Flow Diagram



APPENDIX A

TABLES

TABLE I. Method 200.7 and 6010B Target Analyte List

ELEMENT	Symbol	CAS #	6010B analyte	200.7 analyte	Reporting Limit (ug/L) Water	Reporting Limit (mg/kg) Soil
Aluminum	Al	7429-90-5	X	X	200	20
Antimony	Sb	7440-36-0	X	X	60	6
Arsenic	As	7440-38-2	X	X	300	30
Barium	Ba	7440-39-3	X	X	200	20
Beryllium	Be	7440-41-7	X	X	5.0	0.5
Boron	B	7440-42-8		X	200	20
Cadmium	Cd	7440-43-9	X	X	5.0	0.5
Calcium	Ca	7440-70-2	X	X	5000	500
Chromium	Cr	7440-47-3	X	X	10	1
Cobalt	Co	7440-48-4	X	X	50	5
Copper	Cu	7440-50-8	X	X	25	2.5
Iron	Fe	7439-89-6	X	X	100	10
Lead	Pb	7439-92-1	X	X	100	10
Lithium	Li	7439-93-2	X	X	50	5
Magnesium	Mg	7439-95-4	X	X	5000	500
Manganese	Mn	7439-96-5	X	X	15	1.5
Molybdenum	Mo	7439-98-7	X	X	40	4
Nickel	Ni	7440-02-0	X	X	40	4
Phosphorus	P	7723-14-0	X	X	300	30
Potassium	K	7440-09-7	X	X	5000	500
Selenium	Se	7782-49-2	X	X	250	25
Silicon	Si	7631-86-9		X	500	N/A
Silver	Ag	7440-22-4	X	X	10	1
Sodium	Na	7440-23-5	X	X	5000	500
Strontium	Sr	7440-24-6	X		50	5
Thallium	Tl	7440-28-0	X	X	2000	200
Vanadium	V	7440-62-2	X	X	50	5
Zinc	Zn	7440-66-6	X	X	20	2

TABLE IA. Method 200.7 and 6010B Trace ICP Target Analyte List

ELEMENT	Symbol	CAS #	Reporting Limit (ug/L) Water	Reporting Limit (mg/kg) Soil
Arsenic	As	7440-38-2	10	1.0
Lead	Pb	7439-92-1	3.0	0.3
Selenium	Se	7782-49-2	5.0	0.5
Thallium	Tl	7440-28-0	10	1.0
Antimony	Sb	7440-36-0	10	1.0
Cadmium	Cd	7440-43-9	2.0	0.2
Silver	Ag	7440-22-4	5.0	0.5
Chromium	Cr	7440-47-3	5.0	0.5

TABLE II. Non-Routine Analyte List

ELEMENT	Symbol	CAS #	Reporting Limit (ug/L) Water	Reporting Limit (mg/kg) Soil
Tin	Sn	7440-31-5	100	10
Titanium	Ti	7440-32-6	50	5
Bismuth	Bi	7440-06-99	200	20
Zirconium	Zr	7440-06-77	100	10
Tungsten	W	7440-03-37	500	50
Tellurium	Te	1349-48-09	500	50
Thorium	Th	7440-02-91	500	50
Uranium	U	7440-06-11	500	50
Palladium	Pd	7440-00-53	100	10

NOTE: Analysis of all elements listed may not be available at all Quanterra facilities.

TABLE III. Matrix Spike and Aqueous Laboratory Control Sample Levels

ELEMENT	LCS Level (ug/L)	Matrix Spike Level (ug/L)
Aluminum	2000	2000
Antimony	500	500
Arsenic	2000	2000
Barium	2000	2000
Beryllium	50	50
Cadmium	50	50
Calcium	50000	50000
Chromium	200	200
Cobalt	500	500
Copper	250	250
Iron	1000	1000
Lead	500	500
Lithium	1000	1000
Magnesium	50000	50000
Manganese	500	500
Molybdenum	1000	1000
Nickel	500	500
Phosphorous	10000	10000
Potassium	50000	50000
Selenium	2000	2000
Silver	50	50
Sodium	50000	50000
Strontium	1000	1000
Thallium	2000	2000
Vanadium	500	500
Zinc	500	500
Boron	1000	1000
Silicon	10000	10000
Tin	2000	2000
Titanium	1000	1000
Bismuth	1000	1000
Zirconium	1000	1000
Tellurium	1000	1000
Thorium	1000	1000
Uranium	1000	1000
Tungsten	1000	1000
Palladium	1000	1000

TABLE IV. ICP Calibration and Calibration Verification Standards

Element	Calibration Level	RL (ug/L)	ICV (ug/L)	CCV (ug/L)
Aluminum	100000	200	25000	50000
Antimony	10000	60	1000	5000
Arsenic	10000	300	1000	5000
Barium	10000	200	1000	5000
Beryllium	10000	5	1000	5000
Cadmium	10000	5	1000	5000
Calcium	100000	5000	25000	50000
Chromium	10000	10	1000	5000
Cobalt	10000	50	1000	5000
Copper	10000	25	1000	5000
Iron	100000	100	25000	50000
Lead	10000	100	1000	5000
Lithium	10000	50	1000	5000
Magnesium	100000	5000	25000	50000
Manganese	10000	15	1000	5000
Molybdenum	10000	40	1000	5000
Nickel	10000	40	1000	5000
Phosphorous	10000	300	1000	5000
Potassium	100000	5000	25000	50000
Selenium	10000	250	1000	5000
Silver	2000	10	500	1000
Sodium	100000	5000	25000	50000
Strontium	10000	50	1000	5000
Thallium	20000	2000	5000	10000
Vanadium	10000	50	1000	5000
Zinc	10000	20	1000	5000
Boron	10000	200	1000	5000
Silicon	10000	500	1000	5000
Tin	10000	100	1000	5000
Titanium	10000	50	1000	5000
Bismuth	10000	200	1000	5000
Zirconium	10000	100	1000	5000
Tellurium	10000	500	1000	5000
Thorium	10000	500	1000	5000
Uranium	10000	500	1000	5000
Tungsten	10000	500	1000	5000
Palladium	10000	100	1000	5000

TABLE IVA. Trace ICP Calibration and Calibration Verification Standards

Element	Calibration Level	RL (ug/L)	ICV (ug/L)	CCV (ug/L)
Aluminum	50000	200	12500	25000
Antimony	1000	10	250	500
Arsenic	1000	10	250	500
Barium	4000	10	1000	2000
Beryllium	4000	5	1000	2000
Cadmium	1000	2	250	500
Calcium	100000	5000	25000	50000
Chromium	4000	5	1000	2000
Cobalt	4000	50	1000	2000
Copper	4000	25	1000	2000
Iron	50000	100	12500	25000
Lead	1000	3	250	500
Magnesium	100000	5000	25000	50000
Manganese	4000	15	1000	2000
Molybdenum	4000	40	1000	2000
Nickel	4000	40	1000	2000
Potassium	100000	5000	25000	50000
Selenium	1000	5	250	500
Silver	2000	5	500	1000
Sodium	100000	5000	25000	50000
Thallium	2000	10	500	1000
Vanadium	4000	50	1000	2000
Zinc	4000	20	1000	2000

TABLE V. Interference Check Sample Concentrations*

Element	ICSA (ug/L)	ICSAB (ug/L)
Aluminum	500000	500000
Antimony	-	1000
Arsenic	-	1000
Barium	-	500
Beryllium	-	500
Cadmium	-	1000
Calcium	500000	500000
Chromium	-	500
Cobalt	-	500
Copper	-	500
Iron	200000	200000
Lead	-	1000
Magnesium	500000	500000
Manganese	-	500
Molybdenum	-	1000
Nickel	-	1000
Potassium	-	10000
Selenium	-	1000
Silver	-	1000
Sodium	-	10000
Thallium	-	10000**
Vanadium	-	500
Zinc	-	1000
Tin	-	1000

* Custom Quanterra solutions contain analytes common to all Quanterra facilities. Non-routine elements not listed above should be spiked into the ICSAB at 1000 ug/L.

** Thallium level for Trace ICP should be at 1000 ug/L.

TABLE VI. TCLP Reporting Limits, Regulatory Limits and Matrix Spike Levels

ELEMENT	Reporting Level (ug/L)	Regulatory Limit (ug/L)	Spike Level (ug/L)
Arsenic	500	5000	5000
Barium	10000	100000	50000
Cadmium	100	1000	1000
Chromium	500	5000	5000
Lead	500	5000	5000
Selenium	250	1000	1000
Silver	500	5000	1000

TABLE VII. Summary Of Quality Control Requirements

QC PARAMETER	FREQUENCY *	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Two-point Initial Calibration	Beginning of every analytical run, every 24 hours, whenever instrument is modified, or CCV criterion is not met	RSD between duplicate exposures ≤5%	Terminate analysis; Correct the problem; Prepare new standards; Recalibrate following system performance.
ICV	Beginning of every analytical run.	Method 200.7: 95 - 105 % recovery. Method 6010B: 90 - 110 % recovery.	Terminate analysis; Correct the problem; Recalibrate.
ICB	Beginning of every analytical run, immediately following the ICV.	The result must be within +/- RL from zero.	Terminate analysis; Correct the problem; Recalibrate.
CCV	Every 10 samples and at the end of the run.	Method 200.7 & 6010B: 90 - 110 % recovery.	Terminate analysis; Correct the problem; Recalibrate and rerun all samples not bracketed by acceptable CCV.
CCB	Immediately following each CCV.	The result must be within +/- RL from zero.	Terminate analysis; Correct the problem; Recalibrate and rerun all samples not bracketed by acceptable CCB.
ICSA	Beginning of every run	See Section 9.8.3	See Section 9.8.3.
ICSAB	Immediately following each ICSA.	Results must be within 80 - 120% recovery.	See Section 9.8.2.

* See Sections 11.11 and 11.12 for exact run sequence to be followed.

TABLE VII. Summary of Quality Control Requirements (Continued)

QC PARAMETER	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Dilution Test	One per prep batch.	For samples > 50x IDL, dilutions must agree within 10%.	Narrate the possibility of physical or chemical interference per client request.
Method Blank	One per sample preparation batch of up to 20 samples.	The result must be less than or equal to the RL. Common lab contaminants may be accepted up to 2x the RL after consultation with the client (See 9.2). Sample results greater than 10x the blank concentration are acceptable. Samples for which the contaminant is < RL may not require redigestion or reanalysis (see Section 9.2).	Redigest and reanalyze samples. Note exceptions under criteria section. See Section 9.2 for additional requirements.
Laboratory Control Sample (LCS)	One per sample preparation batch of up to 20 samples.	Aqueous LCS must be within 80 - 120% recovery or in-house control limits. (85-115% for 200.7) Samples for which the contaminant is < RL and the LCS results are > 120% (115% for 200.7) may not require redigestion or reanalysis (see Section 9.3)	Terminate analysis; Correct the problem; Redigest and reanalyze all samples associated with the LCS.

TABLE VII. Summary of Quality Control Requirements (Continued)

QC PARAMETER	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Matrix Spike	One per sample preparation batch of up to 20 samples.	75 - 125 % recovery or in-house control limits. I For TCLP See Section 11.17.	In the absence of client specific requirements, flag the data; no flag required if the sample level is > 4x the spike added. For TCLP see Section 11.17.
Matrix Spike Duplicate	See Matrix Spike	75 - 125 % recovery; RPD ≤ 20% .	See Corrective Action for Matrix Spike.

APPENDIX B

QUANTERRA ICP DATA REVIEW CHECKLIST

Quanterra ICP Data Review Checklist

Run/Project Information:

Run Date: _____ **Analyst:** _____ **Instrument:** _____
Prep Batches Run: _____

Circle Methods used: **6010B / 200.7: CORP-MT-0001 Rev 2**
CLP ILMO3.0/4.0 : CORP-MT-0002 Rev 1

Review Items

A. Calibration/Instrument Run QC	Yes	No	N/A	2nd Level
1. Instrument calibrated per manufacturer's instructions and at SOP specified levels ?				
2. ICV/CCV analyzed at appropriate frequency and within control limits ? (6010B, CLP = 90 - 110%, 200.7 = 95 -105%[ICV])		/		
3. ICB/CCB analyzed at appropriate frequency and within +/- RL or +/- CRDL (CLP) ?				
4. CRI analyzed? (for CLP only)				
5. ICSA/ICSAB run at required frequency and within SOP limits ?				
B. Sample Results				
1. Were samples with concentrations > the linear range for any parameter diluted and reanalyzed ?				
2. All reported results bracketed by in control QC ?				
3. Sample analyses done within holding time ?				
C. Preparation/Matrix QC				
1. LCS done per prep batch and within QC limits ?				
2. Method blank done per prep batch and < RL or CRDL (CLP) ?				
3. MS run at required frequency and within limits ?				
4. MSD or DU run at required frequency and RPD within SOP limits ?				
5. Dilution Test done per prep batch (or per SDG for CLP) ?				
6. Post digest spike analyzed if required (CLP only) ?				
D. Other				
1. Are all nonconformances documented appropriately ?				
2. Current IDL/LR/IEC data on file ?				
3. Calculations checked for error ?				
4. Transcriptions checked for error ?				
5. All client/project specific requirements met ?				
6. Date/time of analysis verified as correct ?				

Analyst: _____ **Date:** _____
Comments: _____

2nd Level Reviewer : _____ **Date:** _____
Comments: _____

APPENDIX C

CROSS REFERENCE OF TERMS USED IN METHODS 6010B, 200.7, AND BY QUANTERRA

CROSS REFERENCE OF TERMS COMMONLY USED IN METHODS EPA 200.7, SW6010B, AND QUANTERRA INC. SOP

EPA 200.7	SW6010B	Quanterra Inc. SOP
Calibration blank (CB)	Calibration blank	Initial and continuing calibration blanks (ICB/CCB)
Dilution test	Dilution test	Dilution Test
Instrument detection limit (IDL)	Instrument detection limit (IDL)	Instrument detection limit (IDL)
Instrument performance check (IPC)	Continuing calibration verification (CCV)	Continuing calibration verification (CCV)
Internal standard	Internal standard	Internal standard (IS)
Laboratory duplicates	n/a	n/a
Laboratory fortified blank (LFB)	n/a	Laboratory control sample (LCS)
Laboratory fortified sample matrix (LFM)	Matrix spike and matrix spike duplicate (MS/MSD)	Matrix spike and matrix spike duplicate (MS/MSD)
Laboratory reagent blank (LRB)	Method blank	Method or Prep blank (MB)
Linear dynamic range (LDR)	Linear dynamic range (LDR)	Linear dynamic range (LDR)
Method detection limit (MDL)	Method detection limit (MDL)	Method detection limit (MDL)
Quality control sample (QCS)	Check standard or Initial calibration verification (ICV)	Initial calibration verification (ICV)
Spectral interference check solution (SIC)	Interference check solution (ICS)	Interference check solution (ICSA/ICSAB)

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APPENDIX D

MSA GUIDANCE

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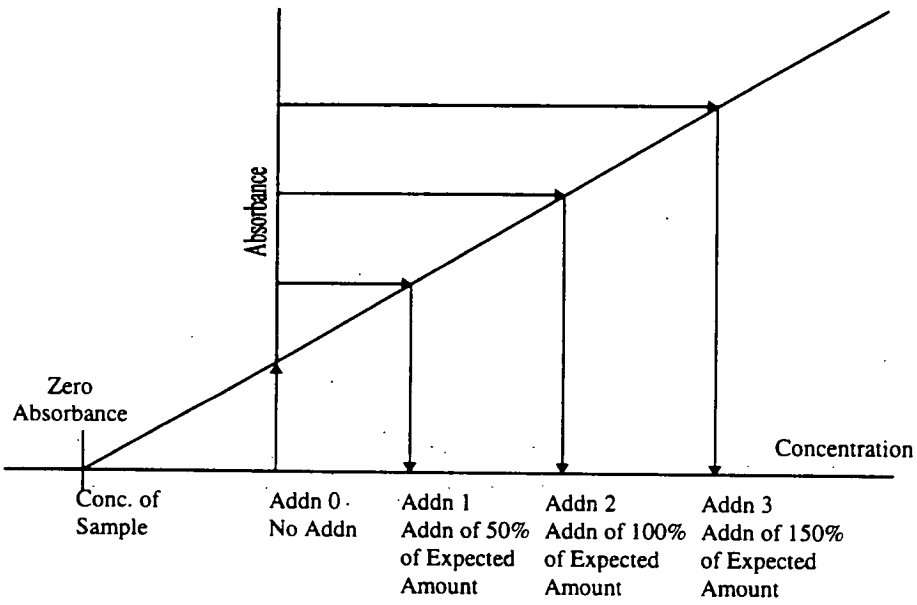
Appendix D. MSA Guidance

Method of Standard Addition

Four equal volume aliquots of sample are measured and known amounts of standards are added to three aliquots. The fourth aliquot is the unknown and no standard is added to it. The concentration of standard added to the first aliquot should be 50% of the expected concentration. The concentration of standard added to the second aliquot should be 100% of the expected concentration and the concentration of standard added to the third aliquot should be 150% of the expected concentration. The volume of the unspiked and spiked standard should be the same.

In order to determine the concentration of analyte in the sample, the analytical value of each solution is determined and a plot or linear regression performed. On the vertical axis the analytical value is plotted versus the concentrations of the standards on the horizontal axis. An example plot is shown in Figure 1. When the resulting line is extrapolated back to zero absorbance, the point of interception of the horizontal axis is the concentration of the unknown.



For the method of standard additions to be correctly applied, the following limitations must be taken into consideration:

- The plot of the sample and standards must be linear over the concentration range of concern. For best results, the slope of the curve should be similar to that of a plot of the aqueous standard curve.
- The effect of the interference should not vary as the ratio of the standard added to the sample matrix changes.

APPENDIX E

TROUBLESHOOTING GUIDE

APPENDIX E. TROUBLESHOOTING GUIDE

Problem	Possible Cause/ Solution
High Blanks	Increase rinse time Clean or replace tip Clean or replace torch Clean or replace sample tubing Clean or replace nebulizer Clean or replace mixing chamber Lower Torch
Instrument Drift	RF not cooling properly Vacuum level is too low Replace torch (Crack) Clean or replace nebulizer (blockage) Check room temperature (changing) Replace pump tubing Room humidity too high Clean torch tip (salt buildup) Check for argon leaks Adjust sample carrier gas Reprofile Horizontal Mirror Replace PA tube
Erratic Readings, Flickering Torch or High RSD	Check for argon leaks Adjust sample carrier gas Replace tubing (clogged) Check drainage(back pressure changing) Increase uptake time (too short) Increase flush time (too short) Clean nebulizer, torch or spray chamber Increase sample volume introduced Check that autosampler tubes are full Sample or dilution of sample not mixed Increase integration time (too short) Realign torch Reduce amount of tubing connectors
Cu/Mn Ratio Outside Limits or Low Sensitivity	Plasma conditions changed Clean nebulizer, torch or spray chamber Replace tubing (clogged) Realign torch Check IECs
Standards reading twice normal absorbance or concentration	Incorrect standard used Incorrect dilution performed

APPENDIX F

CONTAMINATION CONTROL GUIDELINES

APPENDIX F. CONTAMINATION CONTROL GUIDELINES

The following procedures are strongly recommended to prevent contamination:

All work areas used to prepare standards and spikes should be cleaned before and after each use.

All glassware should be washed with detergent and tap water and rinsed with 1:1 nitric acid followed by deionized water.

Proper laboratory housekeeping is essential in the reduction of contamination in the metals laboratory. All work areas must be kept scrupulously clean.

Powdered or Latex Gloves must not be used in the metals laboratory since the powder contains silica and zinc as well as other metallic analytes. Only vinyl or nitrile gloves should be used in the metals laboratory.

Glassware should be periodically checked for cracks and etches and discarded if found. Etched glassware can cause cross contamination of any metallic analytes.

Autosampler trays should be covered to reduce the possibility of contamination. Trace levels of elements being analyzed in the samples can be easily contaminated by dust particles in the laboratory.

The following are helpful hints in the identification of the source of contaminants:

Yellow pipet tips and volumetric caps can sometimes contain cadmium.

Some sample cups have been found to contain lead.

The markings on glass beakers have been found to contain lead. If acid baths are in use for glassware cleaning, they should be periodically checked for contaminants since contaminant concentrations will increase over time.

New glassware especially beakers can be a source of silica and boron.

Reagents or standards can contain contaminants or be contaminated with the improper use of a pipette.

Improper cleaning of glassware can cause contamination.

Latex gloves contain over 500 ppb of zinc.

APPENDIX G

PREVENTIVE MAINTENANCE

APPENDIX G. PREVENTIVE MAINTENANCE

A maintenance log is used to record when maintenance is performed on instruments. When an instrument problem occurs indicate the date, time and instrument number, then identify the problem and corrective action in the maintenance log.

The following procedures are required to ensure that that the instrument is fully operational.

Daily
- Change sample pump tubing and pump windings
- Check argon gas supply level
- Check rinse solution and fill if needed
- Check waste containers and empty if needed
- Check sample capillary tubing is clean and in good condition
- Check droplet size to verify nebulizer is not clogged.
- Check sample flow for cross flow nebulizer
- Check Cu/Mn ratio-should be 30% of value at date that IECs were performed
- Check pressure for vacuum systems

As Needed
- Clean plasma torch assembly to remove accumulated deposits
- Clean nebulizer and drain chamber; keep free-flowing to maintain optimum performance
- Replace peristaltic pump tubing, sample capillary tubing and autosampler sipper probe

Weekly
- Apply silicon spray on autosampler tracks
- Check water level in coolflow

Monthly
- Clean air filters on back of power unit to remove dust
- Check D mirror for air instruments

Bi-yearly
- Change oil for vacuum systems
- Replace coolant water filter (may require more or less frequently depending on quality of cooling water)

Quanterra
Environmental Services

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OPERATION-SPECIFIC STANDARD OPERATING PROCEDURE

TITLE: ANALYSIS OF METALS BY INDUCTIVELY COUPLED PLASMA/MASS SPECTROMETRY

(SUPERSEDES: LM-CAL-2041 ORIGINAL, NOVEMBER 1, 1993)

Prepared by: John Barnett

Reviewed by: Robert T. Dovi
Technical Specialist, Robert Dovi

Approved by: Gail Cho Celaschi
Quality Assurance Manager, Gail Cho Celaschi

Approved by: Cheryl Stith
Environmental Health and Safety Coordinator, Cheryl Ann Stith

Approved by: Roger A. Freize II
Laboratory Manager, Roger Freize II

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1. SCOPE AND APPLICATION

1.1. Analytes

1.1.1. This method is applicable to the determination of metals by inductively coupled plasma mass spectrometry (ICP-MS).

1.2. Applicable matrices.

1.2.1. This method is applicable to drinking, surface, and saline waters; soil and waste samples.

1.3. Reporting Limits

1.3.1. The Quanterra Inc. (West Sacramento) standard reporting limits for metals analyzed by ICP-MS are listed in Appendix A.

1.3.2. Upon client request, results below the standard reporting limit but above the current instrument detection limit (IDL) may be qualified using a "J" flag. The flag indicates that the flagged value is considered "estimated" as defined by U.S. EPA data validation guidelines.

1.4. Analysis Time

1.4.1. The analysis time varies depending on the number of analytes and the memory characteristics of those analytes quantitated in an analysis run.

2. SUMMARY OF METHOD

2.1. Aqueous samples, digestates or leachates are nebulized into a spray chamber where a stream of argon carries the sample aerosol through a quartz torch and injects it into a radio frequency plasma. There the sample is decomposed and desolvated. The ions produces are entrained in the plasma gas and by means of a water-cooled, differentially pumped interface, introduced into a high-vacuum chamber that houses a quadrupole mass spectrometer. The ions are sorted according to their mass-to-charge ratio and measured with a channel electron multiplier.

3. DEFINITIONS

4. INTERFERENCES

4.1.1. Isobaric interferences: Isobaric interferences associated with naturally occurring isotopes are automatically corrected by the instrument software.

4.1.2. Isobaric molecular interferences: Corrections for molecular interferences will be applied where appropriate based on known or suspected interferences.

4.1.2.1. See Appendix B for a list of common interferences and the isotopes affected.

Note: Not all possible interferences are listed.

4.1.3. Matrix interferences: Internal standards will be used to correct for some matrix interferences.

4.1.4. A representative sample(s) of the project or projects being analyzed will be screened to avoid using an analyte as an internal standard that is present in the samples at a level greater than 10% of the level in the internal standard.

4.1.5. Internal standards should be added at a level to give approximately 100,000 - 500,000 counts of raw signal intensity. The mass of the internal standard used should ideally be within ±50 amu of the mass of the affected analyte.

4.1.6. Severe matrix effects will be monitored by comparing the internal standard intensity in the sample to the internal standard intensity of the initial calibration blank (ICB). If the internal standard intensity is less than 30% or greater than 120% of the internal standard intensity of the ICB, a fivefold dilution will be performed on the sample to correct for severe matrix effects, and the sample reanalyzed.

5. SAFETY

5.1. Procedures shall be carried out in a manner that protects the health and safety of all Quanterra associates.

5.2. Eye protection that satisfied ANSI Z87.1 (as per the Chemical Hygiene Plan), laboratory coat, and chemically resistant gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been

contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.2.1. Neoprene, natural rubber, nitrile, and TRIonic® Cleanroom gloves provide varying degrees of protection against those chemicals listed. Refer to permeation/degradation charts for the actual data.

5.3. The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the Material Safety Data Sheets (MSDS) maintained in the laboratory.

5.3.1. The following materials are known to be **corrosive:**

sulfuric acid; hydrochloric acid; nitric acid; hydrofluoric acid (NOTE: sulfuric and hydrofluoric acids are used in cleaning the ICP torch and hydrofluoric acid is also commonly used in air toxics preparations.)

5.3.2. The following materials are known to be **oxidizing agents**:

nitric acid; hydrogen peroxide.

5.3.3. The plasma emits strong UV light and is harmful to vision. AVOID looking directly at the plasma.

5.3.4. The RF generator produces strong radio frequency waves, most of which are unshielded. People with pacemakers should not go near the instrument while in operation.

5.3.5. Refer to Perkin Elmer/Sciex ICP-MS Operator's Manual and the SOP LM-CAL-2000 ICP-MS Operation and Maintenance for information on instrument safety and precautions.

5.4. Exposure to chemicals must be maintained **as low as reasonable achievable**, therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.5. The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operation will permit.

5.6. All work must be stopped in the event of a known or potential compromise to the health and safety of a Quanterra associate. The situation must be reported **immediately** to a laboratory supervisor.

5.7. The use of hydrofluoric acid requires special safety precautions. Consult the facility EH&S Manager and laboratory supervisor for guidance.

NOTE: Contact may not be painful at first. The fluoride ion rapidly penetrates the skin and may cause delayed effects: lasting ulcerations, bone degeneration, pulmonary edema, muscle paralysis, and cardiac arrest. Any suspected exposure to HF liquid/fumes must be immediately evaluated by appropriate medical staff (i.e. U.C. Davis Medical Group Emergency Room.) Review Appendix G for a detailed first aid plan before work with HF begins.

6. EQUIPMENT AND SUPPLIES

6.1. Perkin Elmer/Sciex ELAN 5000 ICP-MS

6.2. Argon gas: High-purity grade (99.99%)

7. REAGENTS AND STANDARDS

7.1. Reagent grade water from a Milli-Q, Barnstead, or equivalent polishing system reading 18 Mohm or higher.

7.2. Calibration Standards: Prepare single or multi-element calibration standards as appropriate for the analysis.

7.3. ICV Stock Solution: Prepare calibration verification standards from a source other than that used for the calibration.

7.4. Calibration Check Standard (CCV): The CCV is a mutlielement solution and can be the either the ICV or one of the calibration standards.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Samples are to be collected in plastic or glass containers.

8.2. All soils must be refrigerated to 4°C ± 2°C.

8.3. The analytical holding time for metals is 6 months by ICP-MS.

8.4. Aqueous samples for total metals must be digested before analysis using an appropriate digestion procedure. The procedure in SOP LM-CAL-2060 for furnace analysis is the preferred digestion procedure. The ICP digestate may also be used, but this may increase the effect of interferences due to the presence of hydrochloric acid. SW846 Preparation Methods 3005, 3010, and 3020 and 3051 may also be used when appropriate. Upon consultation with the client very clean samples can forego digestion to help prevent contamintion when very low detection limits are required.

8.5. Soil or waste samples should be digested before analysis using an appropriate digestion procedure. The furnace digestion in SOP LM-CAL-2060 is preferred and generally gives acceptable results. SW846 Method 3050 and 3051 may also be used.

9. QUALITY CONTROL

9.1. Initial Demonstration of Capability

Prior to analysis of any analyte using Method 6020, the following requirements must be met.

9.1.1. Instrument Detection Limit (IDL) - The IDL for each analyte must be determined for each analyte wavelength used on each instrument. The IDL must be determined quarterly for the standard analytes listed in Appendix A. All other elements will be determined annually. If the instrument is adjusted in any way that may affect the IDL, the IDL for that instrument must be redetermined.

9.1.1.1. IDLs shall be determined by multiplying by three the average of the standard deviations obtained on three non-consecutive days from the analysis of a blank solution with seven consecutive measurements per day.

9.1.1.2. Each measurement must be performed as though it were a separate analytical sample.

9.1.1.3. Each measurement must be followed by a rinse and/or any other procedure normally performed between the analysis of separate samples.

9.1.1.4. The IDL measurement must consist of the same number of replicates used for analytical samples with the average result used for reporting.

9.1.2. Method Detection Limit (MDL) - An MDL must be determined for each analyte/matrix prior to the analysis of any samples. The MDL is determined using seven replicates of reagent water, spiked with all the analytes of interest, that have been carried through the entire analytical procedure. MDL's must be redetermined on an annual basis in accordance with 40 CFR Part 136 Appendix B requirements as detailed in Quanterra QA Policy QA-005. The spike level should be between the calculated MDL and 10X the MDL to be valid. At a minimum, the result of the MDL determination must be below the Quanterra reporting limit.

9.1.3. Linear Range Verification (LR) -The linear range is determined semi-annually (2x/year) for each element on the standard analyte list. Linearity is usually in the range of 0 to 1 million counts. For elements not on the standard analyte list, linearity will be from 0 to 1 million counts.

9.1.3.1. The linear range for routinely reported elements must be verified quarterly. Standards must be run at increasing concentration until elements are no longer within 10% of true value. The last concentration where the element was within 10 % of true value is considered the upper linear range. For those elements not analyzed routinely the upper linear range can be considered to be the concentration at 1,000,000 counts.

9.1.4. Interelement Corrections (IEC)-The ICSA and ICSAB solutions must be run daily. There are no published ICP-MS acceptance criteria for either the ICSA or the ICSAB. Analyses of these samples are used to assess the validity of the elemental equations used for the analysis.

9.2. Method Blank (MB) - One method blank must be processed with each preparation batch. The method blank consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The method blank is used to identify any system and

process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. The method blank should not contain any analyte of interest at or above the reporting limit (exception: common laboratory contaminants, see below) or at or above 5% of the measured concentration of that analyte in associated samples, whichever is higher (sample result must be a minimum of 20x higher than the blank contamination level).

- If the analyte is a common laboratory contaminant (copper, iron, or zinc) the data may be reported with qualifiers if the concentration of the analyte in the method blank is less than two times the RL. **Such action must be taken in consultation with the client and must be addressed in the project narrative.**

- Repreparation and reanalysis of all samples associated with an unacceptable method blank is required when reportable concentrations are determined in the samples (see exception noted above).

- If there is no analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. **Such action must be taken in consultation with the client and must be addressed in the project narrative.**

- If the above criteria are not met and reanalysis is not possible, then the sample data must be qualified. **This anomaly must be addressed in the project narrative and the client must be notified.**

- For dissolved metals samples which have not been digested or matrix matched, a CCB result is reported as the method blank. The CCB run immediately prior to the start of the dissolved sample analyses must be used for this purpose. No more than 20 samples can be associated with one CCB.

9.3. Laboratory Control Sample (LCS) - One aqueous LCS must be processed with each preparation batch. The LCS must contain all analytes of interest and must be carried through the entire analytical procedure. The LCS is used to monitor the accuracy of the analytical process. On-going monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines.

- If any analyte is outside established control limits the system is out of control and corrective action must occur. Until in-house control limits are established, a control limit of 80 - 120% recovery must be applied.

- In the event that an MS/MSD analysis is not possible a Laboratory Control Sample Duplicate (LCSD) must be analyzed. The RPD of the LCS and LCSD must be compared to the matrix spike RPD limits.

- In the instance where the LCS recovery is greater than 120% and the sample results are < RL, the data may be reported with qualifiers. **Such action must be taken in consultation with the client and must be addressed in the case narrative.**

- Corrective action will be repreparation and reanalysis of the batch unless the client agrees that other corrective action is acceptable.

- For dissolved metals samples which have not been digested or matrix matched, a CCV result is reported as the LCS. The CCV run immediately prior to the start of the dissolved sample analyses must be used for this purpose. No more than 20 samples can be associated with one CCV.

9.4. Matrix Spike/Matrix Spike Duplicate (MS/MSD) - One MS/MSD pair must be processed for each preparation batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Some client specific data quality objectives (DQO's) may require the use of sample duplicates in place of or in addition to MS/MSD's. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, these results may have immediate bearing only on the specific sample spiked. Samples identified as field blanks cannot be used for MS/MSD analysis.

- If any analyte recovery or RPD falls outside the acceptance range, the recovery of that analyte must be in control for the LCS. Control limits of 80 - 120 % recovery and 20% RPD (± RL if concentration is <10x the RL) must be applied to the MS/MSD. If the LCS recovery is within limits, then the laboratory operation is in control and the results may be accepted. If the recovery of the LCS is outside limits corrective action must be taken. Corrective action will include repreparation and reanalysis of the batch. MS/MSD results which fall outside the control limits must be addressed in the narrative.

- If the native analyte concentration in the MS/MSD exceeds 4x the spike level for that analyte, the recovery data are reported as NC (i.e., not calculated). If the

reporting software does not have the ability to report NC then the actual recovery must be reported and narrated as follows: "Results outside of limits do not necessarily reflect poor method performance in the matrix due to high analyte concentrations in the sample relative to the spike level."

- If an MS/MSD is not possible due to limited sample volume then a laboratory control sample duplicate (LCSD) should be analyzed. The RPD of the LCS and LCSD must be compared to the matrix spike RPD limits.

- For dissolved metals samples which have not been digested or matrix matched, a MS/MSD must be performed per batch of up to 20 samples by spiking two aliquots of the sample.

9.5. Initial Calibration Verification (ICV/ICB) - Calibration accuracy is verified by analyzing a second source standard (ICV). The ICV must fall within 10% of the true value for that solution. An ICB is analyzed immediately following the ICV to monitor low level accuracy and system cleanliness. The ICB result must fall within +/- the reporting limit (RL) from zero. If either the ICV or ICB fail to meet criteria, the analysis should be terminated, the problem corrected, the instrument recalibrated and the calibration reverified.

9.6. Continuing Calibration Verification (CCV/CCB) - Calibration accuracy is monitored throughout the analytical run through the analysis of a known standard after every 10 samples. The CCV must fall within 10% of the true value for that solution. A CCB is analyzed immediately following each CCV. The CCB result must fall within +/- RL from zero. Sample results may only be reported when bracketed by valid ICV/CCV and ICB/CCB pairs. If a mid-run CCV or CCB fails, the CCV or CCB may be reanalyzed once to and accepted if there is a reason for the initial out-of-control event such as carryover from a high concentration sample. Otherwise, if the CCV or CCB fails, the analysis for the affected element must be terminated, the problem corrected, the instrument recalibrated, the calibration verified and the affected samples reanalyzed. (Refer to Section 11.9 for an illustration of the appropriate rerun sequence).

9.7. The calibration curve must have a correlation coefficient of 0.995 or greater (assuming that more than 2 points are used to generate the linear curve).

9.8. The internal standard intensities must be 30% < [IS intensities for the ICB] < 120%. If this criteria is not met, the sample will be diluted and reanalyzed until the IS recoveries are within the limits. If the upper control limit is exceeded, the analyst

should review the data for the presence of the out-of-control internal standard in the native sample. Narrate any findings.

9.8.1. The internal standard intensity in the ICV and ICB should be within 20% of the IS intensity in the instrument standardization solution. If not, the analyst should check for any instrument anomalies and continue if none are noted.

9.9. Analytical matrix spikes on samples after preparation may be done at the discretion of a senior ICP-MS operator or Technical Manager to further assess the effect of the matrix on the results or as an indication of the performance of the interference equations used in the sample matrix.

9.10. Quality Assurance Summaries - Certain clients may require specific project or program QC which may supersede the SOP requirements. Quality Assurance Summaries (QAS) should be developed to address these requirements.

10. CALIBRATION AND STANDARDIZATION

10.1. Instrument start-up

10.1.1. Follow the instrument start-up procedure outlined in the ICP-MS OPERATION AND MAINTENANCE SOP LP-CAL-2000).

10.2. Instrument Tuning

10.2.1. Follow the instrument tuning procedure outlined in the ICP-MS OPERATION AND MAINTENANCE SOP (LP-CAL-2000).

10.2.2. Mass calibration and resolution checks must be documented and included as part of the raw data package.

10.2.2.1. Resolution must be < 1.0 amu at 10% peak height.

10.2.2.2. Mass calibration must be within ± 0.1 amu from the actual value or the mass calibration must be adjusted.

10.3. Standardization

10.3.1. From the APPLICATIONS menu load the QUANTITATIVE ANALYSIS application.

10.3.2. Load the appropriate parameter set if one already exists or create a new one for the analytes to be quantitated in the run. Solicit the assistance of a senior ICP-MS operator in creating a new parameter file.

10.3.3. See Appendix B for recommended isotopes and interference equations for commonly analyzed elements.

10.3.4. If no recommended isotopes are given for the element to be analyzed, consult a senior ICP-MS operator or appropriate reference (see Section 13.2).

10.3.5. See Appendix C or commonly used internal standards.

10.3.6. All masses which could affect data quality should be monitored to determine potential interferences either simultaneously during an analytical run or in a separate scan.

10.3.7. Open a new data set for a new analytical run. Title the data set with an appropriate title such as the date.

10.3.8. In order to simplify data archival, data set names should be limited to eight characters if possible.

10.3.9. Under the data set description enter the statement "ANALYST: (your initials)" and any other necessary information.

10.3.10. Two standards are routinely used for instrument calibration using a simple linear calibration curve.

- Low standard - Reagent water matching the sample digestate and standards, i.e. 1% HNO_3/0.5% HCl for water samples.
- High standard - the level should be appropriate for the analysis being performed.

10.3.11. If the use of more than two standards is desired, either the weighted linear or blank subtracted calibration types should be selected. The use of a simple

linear curve with more than two standards may cause a positive y-intercept leading to artificially elevated blank levels.

10.3.12.The first line in the data set should be used for the Low Standard. The standard name must be the same as what the standard concentration file was named. The Low Standard must be classified as a "standard" (as opposed to a "blank" which would signal the software to perform blank subtraction, which is not routinely used). The concentrations entered in the concentration file should be zero for all elements. Based on this standardization technique a Simple Linear curve is the most appropriate.

10.3.13.The second line should be used for the high standard. Again, the standard name must match the standard file name. The appropriate concentrations should be entered in the standard concentration file under the same name and saved.

10.3.14.Add internal standard to all standards and samples either by spiking or pumping. Both methods are acceptable.

10.3.14.1.If the pumping method is used, an additional line for the pump and a mixing tee are required. The internal standard line must be placed in the internal standard solution before any analyses are started.

10.3.15.A read delay of 45 seconds (60 seconds or longer if using the autosampler) should be entered in the configuration portion of the software which will allow the sample to travel through the sample uptake tubing and equilibrate through the plasma into the vacuum chamber before the read process is initiated. This allows all standards and samples to be processed in an identical manner.

10.3.16.Insert the sampler tube into the blank solution and press **PF12** (READ) to begin the analysis. After the "Measurements Complete" message appears at the top left-hand corner of the screen remove the sample uptake tube and return it to the rinse solution.

10.3.17.After the standard data has been collected and processed the data will automatically be stored and printed.

10.3.18.Repeat step 10.3.16 for the high standard. When complete the instrument is

standardized

11. PROCEDURE

11.1. One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and is approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

11.2. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

11.3. An instrument or team backlog is generated from the LIMS data system. This backlog lists the Quanterra (WSAC) project numbers that were assigned to each batch or QC lot of samples (a batch or QC lot consists of a maximum of 20 customer samples). The backlog also shows project due date, number of samples and sample matrix.

11.4. An analytical run will consist of all customer samples and quality control samples analyzed under a single standardization. Each new standardization will begin a new analytical run.

11.5. A new ANALYSIS RUN LOG will be filled out for each analytical run. An alternative is to print out the "Sample Entry Report" from the Elan computer and include that.

11.6. Type in the QC and sample information into the sample ID column. You can change the default dilution factor (1) and enter any additional sample information (such as QC lots) by pressing **SHIFT F5** and filling out the detailed sample information.

11.7. In order to use the ICP-MS data upload program into LIMS, the following naming conventions must be followed:

11.7.1. Samples are identified by project number-sample number and aliquot. For example, Sample 1 from project 28977 is identified as: 28977-01.

11.7.2. Matrix spikes, duplicates, and matrix spike duplicates are identified as 28977-

01MS, 28977-01DU, and 28977-01SD, respectively.

11.7.3. Prep Blanks are identified by BLANK.

11.7.4. LCSs are identified as LCS, LCS DUP.

11.8. The order of analysis for the initial QC samples should be:

- ICV
- ICB
- Elemental Equation verification or interference check solution (if required).
- Prep Blank
- LCS
- LCSDUP (if required).
- Samples

11.9. Analysis sequence when out-of-control run QC is observed:

- CCV
- CCB
- Restandardize (see 10.3.16 through 10.3.18)
- CCV
- CCB
- Samples

12. DATA ANALYSIS AND CALCULATIONS

12.1. All pertinent calculations are performed by the ELAN software.

12.2. Reporting Requirements

12.2.1. Units equal mg\L for aqueous samples and mg\kg for soil samples.

12.2.2. If dilutions were required due to insufficient sample interferences, or other problems, the reporting limits are multiplied by the dilution factor.

12.2.3. For results less than 10 mg\L, two significant figures will be reported. For results greater than or equal to 10 mg\L, three significant figures will be reported. Refer to Quanterra Policy QA-0004 for additional information on significant figures and rounding.

12.2.4. Document any non-standard procedures or anomalies by using the anomaly program in LIMS.

12.3. Data Package Requirements

12.3.1. A complete data package consists of: the initial masscal, resolution, and sensitivity checks; run log, copy of parameter file used, level 1 checklist, and all raw data.

12.3.2. Level I review will be completed by the analyst.

12.3.3. Level II review will be completed by a senior level laboratory analyst familiar with the technical aspects of ICP-MS and in accordance with the ICP-MS DATA REVIEW checklists. The instrument operator of an analytical run may not perform the Level II review for that run.

13. METHOD PERFORMANCE

13.1. Each laboratory must have initial demonstration of performance data on file for each analyte of interest as described in Section 9.0.

13.2. Refer to Appendix A for the list of analytes using this SOP.

13.3. Method performance is determined by the analysis of matrix spike and matrix spike duplicate samples as well as method blanks and laboratory control samples. The matrix spike recovery should fall within ± 20% and the matrix spike duplicates should

compare within ± 20% RPD (± RL if concentration is < 10x RL). Method blanks must meet the criteria specified in Section 9.2. The laboratory control samples should recover within 20% of the true value until in-house control limits are established.

13.4. Training Qualification

13.4.1. The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience.

14. POLLUTION PREVENTION

14.1. Standards or solutions are not approved for disposal to the sink.

15. WASTE MANAGEMENT

15.1. Waste generated in the procedure must be segregated and disposed according to the facility hazardous waste procedures. The Environmental Health and Safety Director should be contacted if additional information is required.

15.2. Standards should be purchased and prepared in volumes consistent with laboratory use to minimize the volume of expired standards to be disposed.

15.3. All waste must be disposed of according to the facility hazardous waste managment procedures, Attachment C of the Quanterra Inc. Chemical Hygiene Plan, Section WS002, Table 1.

15.4. Samples and other solutions containing high concentrations of toxic materials must be disposed of according to the facility hazardous waste management procedures, Attachment C of the Quanterra Inc. Chemical Hygiene Plan, Section WS003, Disposal of Samples After Analysis.

16. REFERENCES

16.1. Source Method: EPA Method 6020 CLP-M, Version 8.

16.2. Quanterra Inc., Quality Assurance Management Plan, Revision 1.0, May 1996.

16.3. Quanterra Corporate Policy QA-003 QC Program

16.4. LS-CAL-2002, Standards Preparation-Metals.

16.5. Perkin Elmer/Sciex ELAN model 5000 Users Manual

16.6. SW846 Method 6020, Revision 0 (3rd Update, September 1994).

16.7. EPA Method 200.8 EMSL office of Research & Development, Cincinnati, OH (Draft Method, Revision 4.3, August 1990).

16.8. Recommended ICP-MS References

16.8.1. K. E. Jarvis, A. L. Gray, and R. S. Honk, Handbook of Inductively Coupled Plasma Mass Spectrometry; Chapman & Hall, New York 1992.

16.8.2. A. R. Date and A. L. Gray, Applications of Inductively Coupled Mass Spectrometry, Chapman & Hall, New York 1989. ISBN # 0-41201721-0

17. MISCELLANEOUS (TABLES, APPENDICES, ETC...)

17.1. Deviations From Source Method and Rationale

17.1.1. As a conservation of space, the raw data will only contain the mean and standard deviation results on standard ELAN summary report. As long as standard deviation data is shown the analyst can make decisions on replicate quality on standard ELAN summary report.

17.1.2. The ICP-MS is not required to be restandardized every 8 hours as long as all continuing calibration criteria continue to be met.

17.1.3. The continuing calibration standards are not required to be reanalyzed within 2 hours as long as they continue to meet acceptability criteria.

17.1.4. Each CCV and CCB may be reanalyzed once to verify the instrument is not out of calibration providing that the next sample has not yet been analyzed and the analyst has an indication that the CCV or CCB result could have been affected by air bubbles, sample carryover, or other problems.

17.1.5. A serial dilution is not performed. The internal standard is used to monitor matrix interference.

17.1.6. This SOP may be used to analyze for elements not included in Method 6020, as long as appropriate QC samples are run with acceptable results.

17.1.7. For reporting consistency, an ICB/CCB is acceptable if the result is <RL.

17.2. Modifications from Previous SOP

17.2.1. Format was updated to be consistent with Quanterra Inc. QA-001.

17.2.2. ICSA/ICSAB analysis frequency was increased to "daily." Section 9.1.4.

17.2.3. Linear range studies are conducted semiannually instead of quarterly. Section 9.1.3.

17.2.4. Control limits and corrective actions for the MBLK, LCS and MS/MSD were updated to reflect Quanterra Inc. QA Policy QA-003. Sections 9.2, 9.3, 9.4.

17.2.5. Safety sections were updated to reflect the Quanterra Inc. Chemical Hygiene Plan. Sections 5.0, 14.0, 15.0.

17.2.6. Acceptance criteria for the internal standard was updated. Section 9.8.

APPENDICES

Appendix A	Standard Analyte List and Reporting Limits
Appendix B	Common Molecular Ion Interferences-Background and Matrix
Appendix C	Recommended Isotopes and Additional Masses Which May be Monitored
Appendix D	Elemental Equations for Data Calculations
Appendix E	Internal Standards and Limitations for Use
Appendix F	Forms and Documentation
Appendix G	Hydrofluoric Acid First Aid Plan
Appendix H	Flow Diagram

Appendix A

Standard Analyte List and Reporting Limits

Note: These are the routine reporting limits for most sample types. Lower reporting limits may be achievable for special projects. Difficult sample matrices may cuase reporting limits to be raised.

Element	Symbol	CAS #	Aqueous Report Limit, mg/L	Soil Reporting Limit, mg/Kg
Aluminum	Al	7429-90-5	0.05	5.0
Antimony	Sb	7440-36-0	0.002	0.2
Arsenic	As	7440-38-2	0.002	0.2
Barium	Ba	7440-39-3	0.001	0.1
Beryllium	Be	7440-41-7	0.001	0.1
Cadmium	Cd	7440-43-9	0.001	0.1
Chromium	Cr	7440-47-3	0.001	0.1
Cobalt	Co	7440-48-4	0.001	0.1
Copper	Cu	7440-50-8	0.001	0.1
Iron	Fe	7439-89-6	0.05	5.0
Lead	Pb	7439-92-1	0.001	0.1
Magnesium	Mg	7439-95-4	0.01	1.0
Manganese	Mn	7439-96-5	0.001	0.1
Molybdenum	Mo	7439-98-7	0.001	0.1
Nickel	Ni	7440-02-0	0.001	0.1

Element	Symbol	CAS #	Aqueous Report Limit, mg/L	Soil Reporting Limit, mg/Kg
Potassium	K	7440-09-7	0.05	5.0
Selenium	Se	7782-49-2	0.002	0.2
Silver	Ag	7440-22-4	0.001	0.1
Sodium	Na	7440-23-5	0.05	5.0
Tin	Sn	7440-31-5	0.002	0.2
Titanium	Ti	7440-03-26	0.001	0.1
Thallium	Tl	7440-28-0	0.001	0.1
Vanadium	V	7440-62-2	0.005	0.5
Zinc	Zn	7440-66-6	0.002	0.2

APPENDIX B (FROM 13.2.6, METHOD 200.8)

COMMON MOLECULAR ION INTERFERENCES IN ICP-MS

BACKGROUND MOLECULAR IONS

Molecular Ion	Mass	Element Interferences*
NH^+	15	
OH^+	17	
OH_2^+	18	
C_2^+	24	
CN^+	26	
CO^+	28	
N_2^+	28	
N_2H^+	29	
NO^+	30	
NOH^+	31	
O_2^+	32	
O_2H_+	33	
$^{36}ArH^+$	37	
$^{38}ArH^+$	39	

C4-208

Molecular Ion	Mass	Element Interferences*
$^{40}ArH^+$	41	
CO_2^+	44	
CO_2H^+	45	Sc
ArC^+, ArO^+	52	Cr
ArN^+	54	Cr
$ArNH^+$	55	Mn
ArO^+	56	
$ArOH^+$	57	
$^{40}Ar^{36}Ar^+$	76	Se
$^{40}Ar^{38}Ar^+$	78	Se
$^{40}Ar_2^+$	80	Se

* Method elements or internal standards affected by the molecular ions.

MATRIX MOLECULAR IONS

CHLORIDE

Molecular Ion	Mass	Element Interference
$^{35}ClO^+$	51	V
$^{35}ClOH^+$	52	Cr
$^{37}ClO^+$	53	Cr
$^{37}ClOH^+$	54	Cr
$Ar^{35}Cl^+$	75	As
$Ar^{37}Cl^+$	77	Se

SULFATE

Molecular Ion	Mass	Element Interference
$^{32}SO^+$	48	
$^{32}SOH^+$	49	
$^{34}SO^+$	50	V, Cr
$^{34}SOH^+$	51	V
SO_2^+, S_2^+	64	Zn
$Ar^{32}S^+$	72	
$Ar^{34}S^+$	74	

PHOSPHATE

Molecular Ion	Mass	Element Interference
PO^+	47	
POH^+	48	
PO_2^+	63	Cu
ArP^+	71	

GROUP I, II METALS

Molecular Ion	Mass	Element Interference
$ArNa^+$	63	Cu
ArK^+	79	
$ArCa^+$	80	

MATRIX OXIDES*

Molecular Ion	Mass	Element Interference
TiO	62-66	Ni, Cu, Zn
ZrO	106-112	Ag, Cd
MoO	108-116	Cd

* Oxide interferences will normally be very small and will only impact the method elements when present at relatively high concentrations. Some examples of matrix oxides are listed of which the analyst should be aware. It is recommended that Ti and Zr isotopes are monitored in solid waste samples, which are likely to contain high levels of these elements. Mo is monitored as a method analyte.

RECOMMENDED ANALYTICAL ISOTOPES AND ADDITIONAL MASSES WHICH MAY BE MONITORED *

Isotope	Element of Interest
27	Aluminum (1)
121,123	Antimony (1)
75	Arsenic (1)
138,137,136,135,134,132,130	Barium (1)
9	Beryllium (1)
114,112,111,110,113,116,106,108	Cadmium (1)
42,43,44,46,48	Calcium (1)
52,53,50,54	Chromium (1)
59	Cobalt (1)
63,65	Copper (1)
56,54,57,58	Iron (1)
206,207,208	Lead (1)
24,25,26	Magnesium (1)
55	Manganese (1)
202,200,199,201	Mercury
98,96,92,97,94	Molybdenum

Isotope	Element of Interest
58,60,62,61,64	Nickel (1)
39	Potassium (1)
80,78,82,76,77,74	Selenium
107,109	Silver (1)
23	Sodium (1)
203, 205	Thallium (1)
51,50	Vanadium (1)
66, 67, 68	Zinc (1)
83	Krypton
72	Germanium
139	Lanthanum
140	Cerium
129	Xenon
118	Tin
105	Palladium
47,49	Titanium
125	Tellurium
69	Gallium
35,37	Chlorine

*FROM Table 9 of Method 6020 CLP-M)

(1) Element approved for ICP-MS determination by SW846 Method 6020 CLP-M

NOTE: Isotopes recommended for analytical determination are underlined.

APPENDIX D

RECOMMENDED ELEMENTAL EQUATIONS FOR DATA CALCULATIONS

Element	Elemental Equation	Note
Al	(1.000) (^{27}C)	
Sb	(1.000) (^{121}C)	
As	(1.000) (^{75}C) - (3.1278)[^{77}C) - (1.0177)(^{78}C)]	(1)
Ba	(1.000) (^{137}C)	
Be	(1.000) (^{9}C)	
Cd	(1.000) (^{111}C) - (1.073) [(^{108}C) - (0.712) (^{106}C)]	(2)
Cr	(1.000) (^{52}C)	(3)
Co	(1.000) (^{59}C)	
Cu	(1.000) (^{63}C)	
Pb	(1.000) (^{206}C) + (1.000) (^{207}C) + (1.000) (^{208}C)	(4)
Mn	(1.000) (^{55}C)	
Mo	(1.000) (^{98}C) - (0.146) (^{99}C)	(5)
Ni	(1.000) (^{60}C)	

Element	Elemental Equation	Note
Se	(1.000) (^{82}C)	(6)
Ag	(1.000) (^{107}C)	
Tl	(1.000) (^{205}C)	
Th	(1.000) (^{232}C)	
U	(1.000) (^{238}C)	
V	(1.000) (^{51}C) - (3.127) [(^{53}C) - (0.113) (^{52}C)]	(7)
Zn	(1.000) (^{66}C	

* Method elements or internal standards affected by the molecular ions.

INTERNAL STANDARDS

Element	Elemental Equation	Note
Bi	(1.000) (^{209}C)	
In	(1.000) (^{115}C) - (0.0149) (^{118}C)	(8)
Sc	(1.000) (^{45}C)	
Tb	(1.000) (^{159}C)	
Y	(1.000) (^{89}C)	

C Calibration blank subtracted counts at specified mass.

(1) Correction for chloride interference with adjustment for Se77. ArCl 75/77 ratio may be determined from the reagent blank.

(2) Correction of MoO interference. An additional isobaric elemental correction should be made if palladium is present.

(3) In 0.4% v/v HCl, the background from ClOH will normally be small. However the contribution may be estimated from the reagent blank.

(4) Allowance for isotopic variability of lead isotopes.

(5) Isobaric elemental correction for ruthenium.

(6) Some argon supplies contain krypton as an impurity. Selenium is corrected for Kr82 by background subtraction.

(7) Correction of chloride inference with adjustment for Cr53. Cl0 51/53 ration may be determined from the reagent blank.

(8) Isobaric elemental correction for tin.

APPENDIX E

INTERNAL STANDARDS AND LIMITATIONS OF USE

Internal Standard	Mass	Possible Limitation
^{6}Lithium	6	a
Scandium	45	Polyatomic Ion Interference
Germanium	72	
Yttrium	89	a, b
Rhodium	103	
Indium	115	Isobaric Interference by Sn
Terbium	159	
Holmium	165	
Lutetium	175	
Bismuth	209	a

a May be present in environmental samples.

b In some instruments Yttrium may form measurable amounts of YO^+ (105 amu) and YOH^+ (106 amu). If this is the case, care should be taken in the use of the cadmium elemental correction equation.

APPENDIX F

FORMS AND DOCUMENTATION

QUANTERRA INC.
West Sacramento

SOP#_____ **ICP-MS ANALYSIS RUN LOG** **METHOD**_____

ANALYST: **FILENAME:** **DATE:**

NO.	SAMPLE I.D.	D.F.	NO.	SAMPLE I.D.	D.F.	NO.	SAMPLE I.D.	D.F.
1			26			51		
2			27			52		
3			28			53		
4			29			54		
5			30			55		
6			31			56		
7			32			57		
8			33			58		
9			34			59		
10			35			60		
11			36			61		
12			37			62		
13			38			63		
14			39			64		
15			40			65		
16			41			66		
17			42			67		
18			43			68		
19			44			69		
20			45			70		
21			46			71		
22			47			72		
23			48			73		
24			49			74		

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25			50			75		

Standard Verifications: Cal Stock: # ICV/DCS Stock #:

Deviations from SOP:_____

_____Approved:_____

QUANTERRA INC. (West Sacramento)

Anomalies

TEST	SAMPLE(s)	ANOMALIES

1. Prep blank contamination for _____.
2. MDL's used to meet required reporting limits.
3. QC out of limits for non-required elements.
4. Furnace results taken from ICP data.
5. Sample(s) is/are hazardous due to (*characteristic*) for (*element*).
6. Sample(s) diluted due to matrix interference, detection limit raised.
7. Detection limit raised due to matrix interference (no dilution required).
8. Total and dissolved analysis not consistent.
9. Other (explain): _____

_____

_____

_____

Comments: _____

_____

_____

_____

Approved:_____ Date:_____

QUANTERRA INC. (West Sacramento)

ICP-MS DATA REVIEW CHECKLIST

LEVEL I

FILE NO.	PROJECT NOS.	ANALYST:	DATE:	YES	NO	NA

	YES	NO	NA
1. Copy of analysis protocol used included?			
2. ICVs & CCVs within 10% of true value?			
3. ICB & CCBs < reporting limit?			
4. 10 samples or less analyzed between calibration checks?			
5. All parameters within linear range. No results reported from intensity reading with a > sign?			
6. LCS/DCS within limits?			
7. Prep blank value < reporting limit or all samples >10x blank?			
8. Internal standard intensities for samples (unless followed by dilution) are > 30% and < 120% of the ICB intensities?			
9. Appropriate dilution factors applied to data?			
10. Matrix spike and spike dup within customer defined limits			
11. Each batch checked for presence of internal standard in samples?			
12. Anomalies entered using LIMS anomaly program.			

COMMENTS: _____

REVIEWED BY:	DATA ENTERED BY:
DATE:	DATE:

QAQC/BENCHSHEETS/ICPMS

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QUANTERRA INC. (West Sacramento)

PREVIEW DATA REVIEW

LEVEL II

FILE NO.	PROJECT NOS.	ANALYST:	DATE:	YES	NO	NA

	YES	NO	NA
1. Prep and analysis anomalies acceptable?			
2. Bench sheets complete?			
3. Anomalies noted on Camera Ready Report Cover Sheet?			
4. Reanalysis data included?			
5. Error corrections made in appropriate manner?			
6. QC checked and acceptable?			
7. PJ report checked for transcription errors?			
8. Reporting limits correct?			
9. Appropriate flags entered?			
10. Analysis date reflects date of accepted data?			
11. Corrections entered and verified?			
12. Data released?			

COMMENTS: _____

_____

_____

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LEVEL II REVIEWED BY:	CORRECTIONS ENTERED BY:
DATE:	DATE:

Quanterra Inc.-WSAC

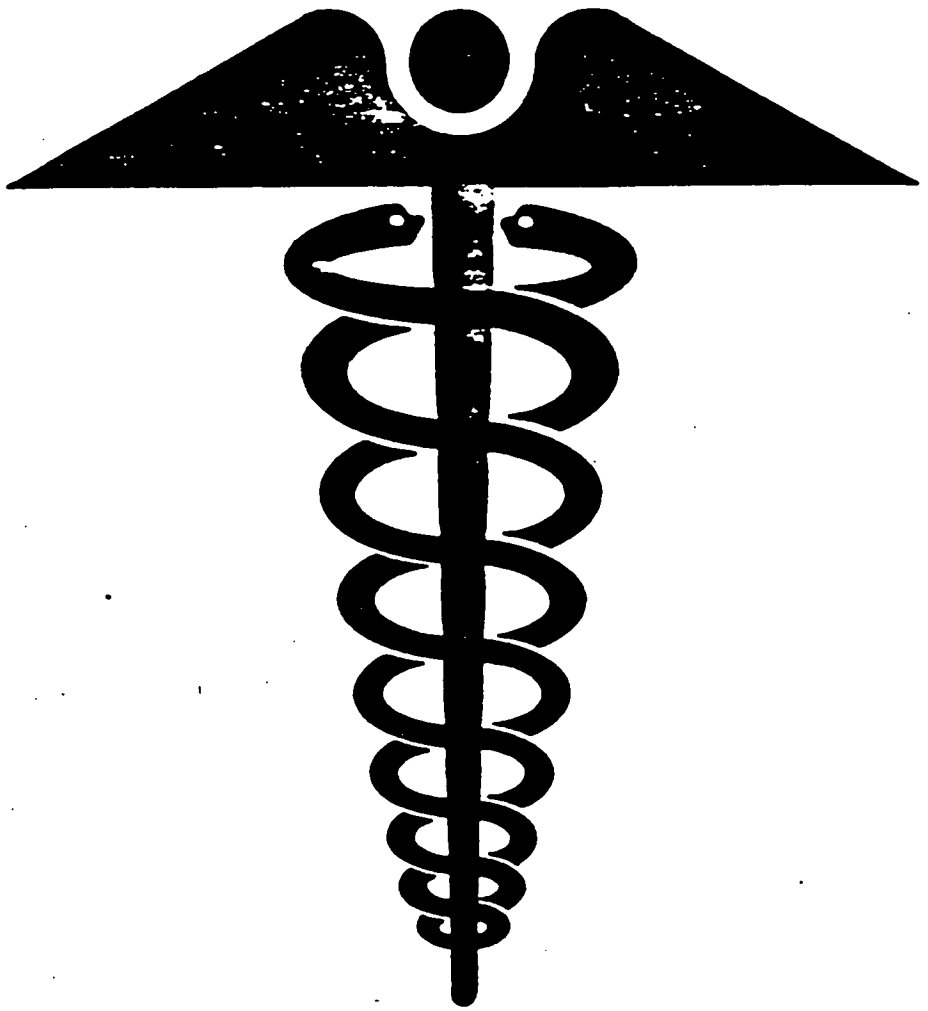
QAQC/BENCHSHEETS/PREVIEW

APPENDIX G

HYDROFLUORIC ACID FIRST AID PLAN

~~Appendix 1~~ 3 1-15-96

Recommended Medical Treatment for Hydrofluoric Acid Exposure



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The HF Products Group

Allied Signal

The HF Products Group

Allied Signal

For more information including technical information on any aspect of hydrofluoric acid write: Allied-Signal Inc., Hydrofluoric Acid Products Group, 101 Columbia Road, P.O. Box 1053, Morristown, NJ 07962-1053.

In Canada, write: Allied-Signal Canada Inc., Manager, Hydrofluoric Acid Products Group, 201 City Centre Drive, Suite 205, Mississauga, Ontario L5B 2T4.

To place an order, obtain prices or availability information from any location in the continental United States, call toll free 800-522-8001.

Your HF Resource

All statements, information, and data given herein are believed to be accurate and reliable but are presented without guaranty, warranty, or responsibility of any kind, express or implied. Statements or suggestions concerning possible use of our products are made without representation or warranty that any such use is free of patent infringement and are not recommendations to infringe any patent. The user should not assume that all medical and first aid measures are indicated or that other measures may not be required.

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Hydrofluoric acid is a very strong inorganic acid. Both anhydrous hydrofluoric acid (hydrogen fluoride) and its solutions are clear, colorless liquids. When exposed to air, concentrated solutions and anhydrous hydrofluoric acid produce pungent fumes which are especially dangerous. Unless heated, dilute concentrations of hydrofluoric acid in water do not produce vapors.

NOTE: Persons unfamiliar with hydrofluoric acid often mistake it or confuse it with hydrochloric acid. Although hydrofluoric acid (HF) and hydrochloric acid (HCl) sound similar, the toxicity of these two acids is very different. To decrease or avoid confusion, we recommend that *HYDROFLUORIC ACID*, and *HYDROGEN FLUORIDE* be referred to as "HF".

HF is primarily an industrial raw material. It is used in stainless steel manufacturing, iron and steel foundries, metal finishing, aluminum manufacturing, inorganic and organic chemical manufacturing, petroleum refining, mineral processing, glassmaking and electronic components manufacturing. It is also used in certain industrial and consumer cleaning compounds. However, its use in consumer products is discouraged because of the hazards described herein.

Most non-industrial burns are caused by dilute concentrations of HF. Most of the HF used in the electronics industry is less than 50% concentration. However, many industrial uses of HF involve concentrated (50-100%) HF.

WARNING: BURNS WITH CONCENTRATED HF ARE USUALLY VERY SERIOUS, WITH THE POTENTIAL FOR SIGNIFICANT COMPLICATIONS DUE TO FLUORIDE TOXICITY. CONCENTRATED HF, LIQUID OR VAPOR, MAY CAUSE SEVERE BURNS, METABOLIC IMBALANCES, PULMONARY EDEMA AND LIFE THREATENING CARDIAC ARRYTHMIAS. EVEN *MODERATE* EXPOSURES TO CONCENTRATED HF MAY RAPIDLY PROGRESS TO A FATALITY IF LEFT UNTREATED.

Allied-Signal is the world's leading supplier of hydrofluoric acid. The recommended medical procedures described below are based on many years experience in dealing with the unique hazards of this product, in addition to a review of the medical literature. Every effort must be made to prevent exposure to hydrofluoric acid. If exposure does occur, the specialized procedures which follow are recommended to avoid the very serious consequences that might otherwise occur.

Because the medical treatment of hydrofluoric acid exposure is so specialized and differs from the treatment of other inorganic acid exposures, not all physicians may be aware of appropriate treatment measures. It is recommended that HF users make arrangements ahead of time with local medical resources to be sure that users are familiar with first aid measures and that professional personnel are familiar with the toxicity of HF and the treatment of HF exposure. This would include, at a minimum, thoroughly reviewing this booklet and making sure that treatment facilities and supplies are available.

Hydrofluoric acid (HF) can cause serious, painful burns of the skin. Specialized first aid and medical treatment is required. Burns larger than 25 square inches (160 square cm) may result in serious systemic toxicity.

Hydrofluoric acid is a highly corrosive acid which can severely burn skin, eyes, and mucous membranes. The *vapors* from anhydrous hydrofluoric acid or its concentrated solutions can also burn these tissues.

Hydrofluoric acid is similar to other acids in that the initial extent of a burn depends on the concentration, the temperature and the duration of contact with the acid. Hydrofluoric acid differs, however, from other acids because the fluoride ion readily penetrates the skin, causing destruction of deep tissue layers including bone. Unlike other acids which are rapidly neutralized, this process may continue for days.

Strong acid concentrations (over 50%), and particularly anhydrous HF (AHF or 100% HF), usually cause immediate, severe, burning pain and a whitish discoloration of the skin which usually proceeds to blister formation. Exposure to HF vapors can also result in similar burns.

In contrast to the immediate effects of concentrated HF, the effects of contact with more dilute hydrofluoric acid or its vapors may be delayed, and this is one of the problems with the recognition of some HF burns. Contact with acid concentrations in

tracheostomy. It is recommended that all patients with such exposures be hospitalized for observation.

Because of the strong irritant nature of hydrofluoric acid, an individual inhaling HF vapors or fumes will usually experience upper respiratory injury, with mucous membrane irritation and inflammation as well as cough. All individuals suspected of inhalation should be observed for pulmonary effects. This would include those individuals with significant upper respiratory irritation, broncho-constriction by pulmonary auscultation or spirometry, and any individual with HF exposure to the head, chest or neck areas. It has been reported that pulmonary edema may be delayed for several hours and even up to two days. If there is no initial upper respiratory irritation, significant inhalation exposure can generally be ruled out.

The Permissible Exposure Limit (PEL) set by the U.S. Occupational Safety and Health Administration (OSHA) is a ceiling level of 3 ppm and a 15 minute short term exposure limit (STEL) of 6 ppm (24). The National Institute for Occupational Safety and Health (NIOSH) has established a level that is immediately dangerous to life and health (IDLH) at 30 ppm (25). The American Industrial Hygiene Association has published Emergency Response Planning Guidelines setting 50 ppm as the maximum level below which nearly all individuals could be exposed for one hour without experiencing or developing life-threatening health effects (ERPG-3), 20 ppm as the maximum level below which nearly all individuals could be exposed for one hour without developing irreversible health effects or symptoms which would impair taking protective action (ERPG-2), and 5 ppm as the maximum level below which nearly all individuals could be exposed up to one hour without experiencing other than mild, transient adverse health effects (ERPG-1) (26).

If hydrofluoric acid is ingested, severe burns to the mouth, esophagus and stomach may occur. Severe systemic effects usually also occur. Ingestion of even small amounts of dilute HF have resulted in death (27).

Chronic toxicity from overexposure to fluoride ion has been reported to result in tooth mottling in children, bone fluorosis and sometimes osteosclerosis in adults and children.

Skeletal fluorosis is known to be associated with excessive exposure to fluoride compounds. Cases of skeletal fluorosis have been reported in populations exposed to naturally occurring drinking water containing greater than 10 ppm of fluoride ion and in individuals exposed to high levels of fluoride containing dusts. However, skeletal fluorosis has not been reported as a consequence of hydrofluoric acid exposure.

Fluorides are not carcinogenic, and have not been reported to cause male or female reproductive effects. Fluoride exposures should be kept below recommended levels to assure no adverse effects to the developing fetal skeletal system.

Monitoring of urine for fluorides is an accepted method of determining exposure (28). Urine fluoride levels above 3 mg/liter at the beginning of a workshift, or above 10 mg/liter at the end of a workshift, may indicate excessive absorption of fluoride. It should be noted that fluorides are often present in significant amounts in persons not occupationally exposed (because of dietary sources of fluoride such as tea), and that the urine fluoride determination is not specific for HF (29, 30).

In Case of Contact or Suspected Contact with Hydrofluoric Acid:

1. Move victim immediately under safety shower or other water source and flush affected area thoroughly with large amounts of cool running water. Speed in washing off the acid is of primary importance.
2. Remove all contaminated clothing while flushing with water.
3. Rinse with large amounts of cool running water. If 0.13% ZEPHIRAN® solution or 2.5% calcium gluconate gel are available, the rinsing may be limited to 5 minutes, with the soaks or gel applied as soon as the rinsing is stopped. If ZEPHIRAN® or calcium gluconate gel are not available, rinsing must continue until medical treatment is rendered.
4. While the victim is being rinsed with water, someone should alert first aid or medical personnel and arrange for subsequent treatment.

3. If a physician is not immediately available, apply one or two drops of 0.5% PONTOCAINE® hydrochloride solution or other aqueous, topical ophthalmic anesthetic and continue irrigation. Use no other medications unless instructed to do so by a physician. Rubbing of the eyes is to be avoided.

1. Immediately move victim to fresh air and get medical attention.
2. Keep victim warm, quiet and comfortable.
3. If breathing has stopped, start artificial respiration at once. Make sure mouth and throat are free of foreign material.
4. Oxygen should be administered as soon as possible by a trained individual. Continue oxygen while awaiting medical attention unless instructed otherwise by a physician.
5. A nebulized solution of 2.5% calcium gluconate may be administered with oxygen by inhalation.
6. Do not give stimulants unless instructed to do so by a physician.
7. The victim should be examined by a physician and held under observation for at least a 24 hour period.
8. Vapor exposures can cause skin mucous membrane burns as well damage to pulmonary tissue. Vapor burns to the skin are treated the same as liquid HF burns.

1. Have the victim drink large amounts of water as quickly as possible to dilute the acid. Do not induce vomiting. Do not give emetics or baking soda. Never give anything by mouth to an unconscious person.
2. Give several glasses of milk or several ounces of milk of magnesia, MAALOX®, MYLANTA® etc. The calcium or magnesium in these compounds may act as an antidote.
3. Get immediate medical attention.

Burns from dilute acid are difficult to distinguish from other chemical burns and usually appear as areas of erythema. However, they may progress, if not treated, to areas of blistering, necrosis or ulceration. Burns from more concentrated acid have a rather characteristic appearance and present as severely reddened, swollen areas with blanched, whitish regions which rapidly progress to blistering and necrosis. A thick granular exudate usually appears under these blisters which requires debridement and removal.

Hydrofluoric acid burns cause extreme pain. The pain is thought to result from nerve ending irritation due to increased levels of potassium ions in extracellular spaces to compensate for the reduced levels of calcium ions which have been bound by the fluoride. Relief of pain is an excellent indication of the success of treatment and, therefore, local anesthetics should be avoided.

Many different types of therapies have been suggested for HF burns. The aim of all treatment is to chemically sequester the fluoride ion and to prevent extensive, deep-tissue destruction (31).

After treatment of burned areas is begun, the victim should be carefully examined to insure there are no other burned areas which may have been overlooked.

Most HF burns can be satisfactorily treated by immersion of the burned part in an iced, aqueous aqueous-alcohol) solution of a quaternary ammon compound. Two solutions have been clinically successful, 0.13% ZEPHIRAN® (benzalkonium chloride) or 0.2% HYAMINE® 1622 (benzethonium chloride). *Because of its availability as a non-prescription drug, ZEPHIRAN® is recommended in the United States.*

The solutions should be cooled with ice cubes. (Shaved or crushed ice may cause excessive cooling, with the danger of frostbite.)

If immersion in the solution is not practical, soaked compresses of the same iced solution should be applied to the burned area. The immersion or compresses should be used for at least two hours. Compresses should be changed or soaked with additional solutions approximately every two to four minutes.

If blisters are present, they should be opened and drained and necrotic tissue should be debrided prior to use of ZEPHIRAN® immersion or compresses.

Prolonged immersion in the iced ZEPHIRAN® bath may result in discomfort due to excess chilling; relief may be obtained by removing the part from the bath every ten to fifteen minutes for a few minutes and then reimmersing it. After the initial 30-60 minutes of treatment, less ice can be used so the bath is cool rather than cold.

treatment to be effective. One author has cautioned that removal of the nail should rarely be necessary in the case of dilute HF acid (less than 10%) burns (32). The treating physician must consider the morbidity associated with removal of the nail versus the need to treat the HF exposure.

If immersion in ZEPHIRAN® solution is started immediately, it may be possible to avoid removing the nail. Sometimes better penetration under the nail can be successfully accomplished by splitting the nail or by drilling several burr holes in the nail using a large gauge needle or a nail drill. If calcium gluconate injection is used as treatment, the nail may still need to be split or removed. Some authorities recommend the use of general anesthesia or a regional nerve block, rather than local anesthesia, to remove the nail so that pain relief by calcium gluconate injections may be used as an indicator of effective treatment. When using calcium gluconate injections in the digits, care must be taken to inject the solution slowly so as to avoid any compromise to the circulation in these areas.

Reports in the literature have described the use of intra-arterial injection or infusion of dilute calcium gluconate solutions to treat HF burns of the hand and digits which do not respond to other methods, either due to inadequate or improper treatment, or in cases where treatment has been greatly delayed. The method is described as follows:

> "A long catheter was inserted percutaneously into the radial artery using standard aseptic technique. Intra-arterial catheter placement was confirmed by pressure transducer and oscilloscope. If the burn involved only the thumb, index, or long fingers, the catheter was advanced only a few centimeters proximally in preparation for digital subtraction arteriography. If the burn involved the ring or small fingers, the catheter was advanced proximally into the brachial artery because access to the ulnar circulation was necessary.
>
> Following satisfactory placement of the arterial catheter, we performed digital subtraction arteriography on all patients in our series to identify the origin of vascular supply to digits involved.
>
> Once the tip of the arterial catheter was in the desired location, a dilute preparation of calcium [gluconate] (10 ml of a 10% solution mixed in 40 to 50 ml 5% dextrose) was infused with a pump apparatus into the catheter over four hours. We generally have used calcium gluconate.... Each patient was observed closely during the infusion period for progression of symptoms and potential complications of the procedure, such as alterations of distal vascular supply.
>
> Following the four-hour infusion, the arterial catheter was maintained in place in the usual manner while the patient underwent an observation period. If typical HF pain returned within four hours, a second calcium infusion was repeated until the patient was pain free four hours following completion of the calcium infusion. (13)"

This method, although rather involved, should be considered in selected cases, especially where inadequate or delayed treatment has occurred.

Where blistering and/or necrosis occur, early debridement may facilitate healing.

In instances of extensive burns, skin grafting has occasionally been required, but the need for this treatment should be markedly reduced by immediate and aggressive primary treatment.

Follow-up care requires monitoring to prevent secondary infections, and the use of antibiotic creams such as SILVADENE® or GARAMYCIN® has proven effective. HF burns may heal slowly, but if properly treated most heal with little or no scarring in 14 to 28 days.

Significant amounts of fluoride ion may be absorbed by skin contact, inhalation, or by ingestion. If systemic absorption of fluoride occurs, hypocalcemia, hypomagnesemia and hyperkalemia may also occur. All of these parameters need to be monitored and appropriate therapeutic measures instituted. The patient should be observed for clinical signs of hypocalcemia following ingestion or inhalation or following extensive burns greater than 25 square inches. Serum calcium determinations must be performed immediately and periodically to monitor and treat hypocalcemia. Severe lowering of serum calcium levels can occur within one to two hours even with HF burns covering less than 2.5% of body surface area (7). Continuous EKG monitoring to observe prolongation of the Q-T interval may be useful to detect early changes in serum calcium, although profound hypocalcemia following HF exposure has been reported in the absence of EKG changes.

The fall in serum calcium may occur precipitously following HF exposure. In two reported cases of exposure to anhydrous HF, the serum calcium fell to

Pulmonary function testing may be helpful in assessing the degree and progress of pulmonary injury.

Specific measures may be needed to treat pulmonary edema. High doses of parenteral steroids may be needed along with the administration of appropriate diuretics. Caution should be taken not to administer excessive fluid. Hemoconcentration may require treatment by phlebotomy. The management of pulmonary edema may result in renal failure due to reduced fluid volume, and this may be another indication for renal dialysis.

If it is necessary to relieve anxiety, use general measures and do not use sedatives which could cause central nervous system depression or hypoventilation. Although right heart failure is uncommon in chemically-induced pulmonary edema, monitoring of pulmonary pressure, arterial pressure, and central venous pressure may be indicated.

Secondary infections must be treated. It is preferable to start antibiotics at the first signs of infection such as fever or tachycardia. Periodic blood cultures may be advisable. Prophylactic use of antibiotics is not advised.

Ingestion Injuries

After first aid is completed (drinking several glasses of water followed by two glasses of milk or two ounces of milk of magnesia, MYLANTA®, or other calcium or magnesium containing antacids), the stomach may be lavaged with lime water. The Levin tube must be passed with care to prevent perforation. Treatment is the same as for ingestion of other strong acids. Systemic toxicity is very likely to occur and may require aggressive treatment.

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drug. Care should be taken that HYAMINE® 1622 is used, not HYAMINE® with other numeric or alphanumeric modifiers.

CALCIUM GLUCONATE GEL is not available commercially in the United States as a pharmaceutical agent. It is hoped that a commercial preparation of calcium gluconate gel will be approved by the U.S. Food and Drug Administration, and become available in the U.S. as it now is in Canada, Great Britain, and other countries. At the present time in the U.S., however, calcium gluconate gel requires a prescription either for the compounded gel or for the 10% solution. The shelf life is uncertain, but it is recommended that calcium gluconate gel be replaced on an annual basis.

CALCIUM GLUCONATE INJECTION, USP (one gram in 10 ml, 10% solution) is labelled for intravenous use only. Experience has shown that when diluted to 5% with normal saline, and used as described in this booklet, it is a safe and effective treatment for HF skin exposure. When diluted to 2.5% and used as described, it is safe for nebulization and inhalation, and when diluted to 1.0% and used as described, it is safe for eye irrigation.

GARAMYCIN® is a Registered Trademark of Schering Corporation, Kenilworth, NJ 07033

HYAMINE® 1622 is a Registered Trademark of Lonza, Inc., Fairlawn, NJ 07410

K-Y® Jelly is a Registered Trademark of Johnson & Johnson Products, Inc., New Brunswick, NJ 08903

MAALOX® is a Registered Trademark of Rorer Pharmaceutical Corporation, Fort Washington, PA 19034

MORGAN THERAPEUTIC LENS® is a Registered Trademark of MorTan, Inc., Missoula, MT 59806

MYLANTA® is a Registered Trademark of Stuart Pharmaceuticals, Wilmington, DE 19897

PONTOCAINE® is a Registered Trademark of Winthrop Laboratories, New York, NY 10016

SILVADENE® is a Registered Trademark of Marion Laboratories, Kansas City, MO 64137

ZEPHIRAN® is a Registered Trademark of Winthrop Laboratories, New York, NY 10016

24.337

	Skin Burns		Eye Exposure	Inhalation		Ingestion
	Conc. HF	Dilute HF	All HF	Conc. HF	Dilute HF	All HF
First Aid	Water Wash THEN ZEPHIRAN® [A] 0.13% soaks OR Calcium Gluconate 2.5% Gel	Water Wash THEN ZEPHIRAN® [A] 0.13% soaks OR Calcium Gluconate 2.5% Gel	Water Wash THEN 1% Calcium Gluconate Irrigation	Oxygen AND 2.5% Calcium Gluconate by Nebulizer	Oxygen AND 2.5% Calcium Gluconate by Nebulizer	Do Not Induce Vomiting Milk or Water THEN Milk of Magnesia or MYLANTA®®
Medical Treatment	Debride THEN Continue Soaks or Calcium Gluconate 2.5% Gel OR Calcium Gluconate 5% Injection[1] AND Observe For/And Treat Systemic Effects[3] (especially if > 25sq. in)	Debride THEN Continue Soaks or Calcium Gluconate 2.5% Gel OR Calcium Gluconate 5% Injection[1] Systemic Effects[3] Unlikely	Topical Tetracaine Hydrochloride THEN 1% Calcium Gluconate Irrigation AND Consult Ophthalmologist	Observe AND Treat Bronchoconstriction, Pulmonary Edema and Systemic Effects[3]	Observe (Serious Effects Unlikely)	Lavage with Limewater AND Treat Systemic Effects[3]

1) This is a brief summary of First Aid and Treatment measures. The text of the booklet "RECOMMENDED MEDICAL TREATMENT FOR HYDROFLUORIC ACID EXPOSURE" must be consulted for more complete information.
2) 5% Calcium gluconate injections must be used if the gel does not significantly relieve pain in 30-60 minutes. Injections may also be used as the primary treatment, especially for larger and/or deeper burns.
3) Systemic effects include hypocalcemia, hypomagnesemia, hyperkalemia, cardiac arrhythmias, and altered pulmonary hemodynamics. TREATMENT includes cardiac monitoring, monitoring serum calcium, magnesium, and electrolytes; administration of intravenous calcium gluconate, correcting magnesium and electrolyte imbalance, and dialysis.

NOTE: In addition to the usual medical history, the physician will find it helpful to obtain the following information: concentration of Hydrofluoric Acid, date and time of exposure, duration of exposure, how exposure occurred, body parts exposed/affected, first aid measures contacted (when, where, how long), injuries due to dilute Hydrofluoric Acid solutions or low concentrations of vapors may result in delays in clinical presentation up to 24 hours following exposure.

[A] Registered Trademark, Winthrop Laboratories, New York, NY 10016

® Registered Trademark, Dow Pharmaceuticals Wilmington, DE 19897

For additional reference charts or information on properties, storage and handling, or medical treatment for hydrofluoric acid, contact:

The HF Products Group
Allied-Signal Inc.
P.O. Box 1053
Morristown, NJ 07962-1053

In the event of an emergency with this product, call the 24-hour Allied-Signal emergency telephone number: (201) 455-3000.

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"Topical Calcium Gluconate as an efficacious treatment for dermal hydrofluoric acid burns."

(Ann. Emerg. Med. Jun '94, Vol. 24, No. 1, pg. 9)

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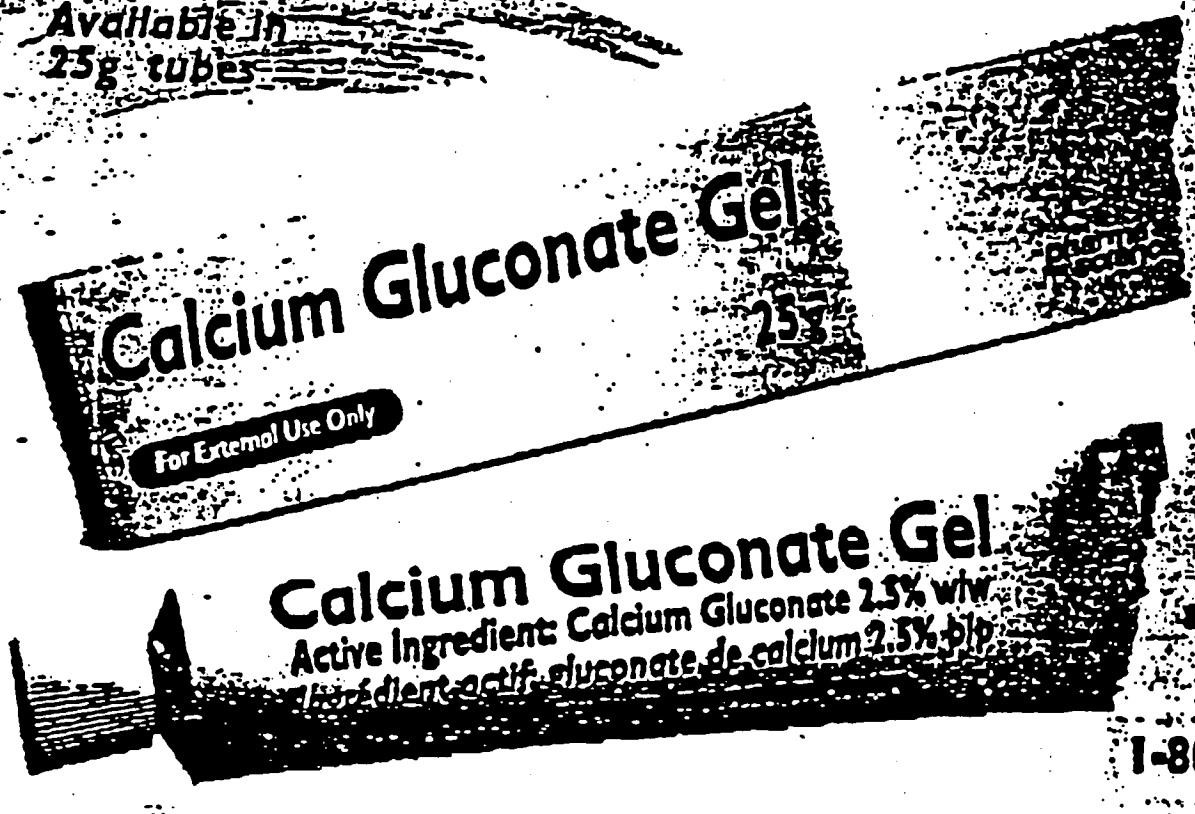

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C4-238

Coping with HF

Reports on the Internet mailing list SAFETY tell of the extreme hazards associated with hydrofluoric acid. They also provide some valuable insights into dealing with HF hazards.

A cautionary story appeared in *The West Australian* last year. Exposure to hydrofluoric acid killed an employee of a small laboratory in Western Australia. The victim accidentally splashed about 100 mL of a 70% solution on his leg. Though the leg was amputated, the acid had poisoned the man's blood and he died agonizingly. According to a health expert on the case, the lab worker would have had little chance of surviving an HF spill as big as the one that killed him even if every precautionary measure had been taken. The acid has to come in contact with only 2% of the body to be fatal.

HF exposure causes profound hypocalcemia, a lowering of blood calcium. HF burns are deep and excruciatingly painful (though burns from exposure to dilute solutions of less than 25% may not be felt for more than an hour). They can be treated through intravenous administration of calcium gluconate solutions. Immediate attention in the laboratory should entail flushing with water followed by soaking in 0.13% Zephiran solution or topical application of calcium gluconate gel which ties up the fluoride ion and thus reduces its damaging effect. Lab personnel should not use HF without Zephiran (Winthrop Laboratories) or calcium gluconate gel being readily available. Calcium gluconate gel can be prepared by mixing calcium gluconate with a water-soluble lubricant such as Johnson & Johnson's K-Y Jelly. Tubes of prepared gel are available commercially; one supplier is Pharmaciences, Inc., in Montreal).

Labs that use HF also should inform local medical facilities and make sure that they have in place a protocol and materials for dealing with patients exposed to HF. For more information on first aid and medical treatment of HF exposures, request a copy of AlliedSignal's publication "Recommended Medical Treatment for Hydrofluoric Acid Exposure" by calling 800/522-8001. This booklet is a must for labs using HF! MSDSs for HF-containing products may provide first aid information as well.

Labs with HF on hand should also be prepared for a spill. Silicate-based absorbents (such as kitty litter) are not useful, because they can react with HF to form SiF_4, a toxic and corrosive gas. Instead, stock a supply of gypsum ($CaSO_4$) to absorb HF spills.

Check around to see if HF is employed in your lab. Also, check old chemical supplies and discard HF solutions that are not in use. Finally, review the MSDSs for such lab supplies as commercial cleaners; HF may turn up as an ingredient.

Personnel using HF should understand the hazards associated with exposure and know the steps to take in the event exposure occurs. The OSHA Lab Standard (29 CFR 1910.1450) mandates that such a hazardous chemical be mentioned in a lab's chemical hygiene plan and discussed in the required annual training.

LSEM has a new editorial office. See staff box on page 2 for new mail and E-mail addresses. Direct comments, inquiries and contributions there, and subscription requests to Burbank.

Waste Management Guide for Clinical Labs

The National Committee for Clinical Laboratory Standards (NCCLS) offers a collection of documents on issues in lab safety and environmental management. One of these, "Clinical Laboratory Waste Management; Approved Guideline" (Document GP5-A, December 1993), provides much important information on waste management in less than 100 pages. According to Gerald A. Hoskyn, MD, of the Cleveland [Ohio] Clinic Foundation, who chairs the NCCLS Subcommittee on Laboratory Waste, the guideline will be revised periodically to keep it current.

NCCLS describes the document thus: "GP5-A, Clinical Laboratory Waste Management; Approved Guideline, was written for use by laboratory managers and is intended to provide approaches to controlling laboratory-generated hazardous and nonhazardous waste. A brief summary of the relevant federal regulations and laws is included. The types of waste addressed include chemical, infectious, radioactive, sharps, and nonhazardous. Options for handling, packing, labeling, storing, treating, transporting, and disposal of each type of waste are also described."

To learn more about the lab waste subcommittee, contact Dr. Hoskyn at 216/444-2830. For a copy of the guideline or list of other relevant publications, write NCCLS at 771 E. Lancaster Ave., Villanova, PA 19085.

Free NSC Training

The National Safety Council is offering a two-day course covering health and safety standards applicable to small-business owners. The training is funded through a grant received by OSHA and, therefore, is free. Topics include ergonomics, personal protective equipment, recordkeeping, incident reporting and program development. While not lab-specific, the course still can benefit lab personnel. The training is given each month through February in Itasca, IL. To learn more, or to register, call NSC at 800/621-7615, ext. 2356.

FR REVIEW

An overview of relevant notices and proposed and final rules published in the Federal Register (60 FR) August 31–October 24, 1995.

Aug. 31: EPA proposed streamlined procedures for revising stationary source operating permits issued under Title V of the CAAA (45530). Contact Michael Trutna, EPA, 919/541-5435. See story on page 1.

Sept. 1: EPA finalized its NESHAP for aerospace manufacturing and rework facilities (45948). Contact Vickie Bootle, EPA, 919/541-0164. See story on page 2.

Sept. 5: EPA proposed to delete diethyl phthalate from the list of chemicals subject to reporting requirements under Section 313 of the Emergency Planning and Community Right-to-Know Act (46076). Contact the EPCRA Hotline, 800/535-0202.

Sept. 5: EPA rescinded Subpart I of 40 CFR 61 (radionuclide air emissions limits) as it applies to nuclear power reactors (46206). Contact Fran Jones, EPA, 202/233-9300.

Sept. 7: EPA issued a rule limiting regulatory obligations of lending institutions or other persons holding a security interest in property containing an underground storage tank (46692). Contact the RCRA Hotline, 800/424-9346.

Sept. 8: NRC proposed a one-time 5-year extension of certain by-product, source, and special nuclear materials licenses (46784). Contact John Patchet, NRC Region II, 404/331-5083.

Sept. 20: EPA terminated the reporting requirements for 234 chemicals under the TSCA Section 8(d) model health and safety data reporting rule (48660). Contact the TSCA Hotline, 202/554-0551.

Sept. 20: DOT's Research and Special Programs Administration (RSPA) revised its regulations pertaining to infectious substances, including regulated medical waste (48780). Contact Jennifer Antonetti, RSPA, 202/366-8553.

Sept. 27: EPA announced that revisions to North Carolina's hazardous waste program satisfy requirements necessary to qualify for final authorization under RCRA (49800). Contact Al Hanke, EPA Region 4, 404/347-2234.

Sept. 28: EPA proposed rescinding 40 CFR 61, Subpart I (radionuclide NESHAP), as it applies to NRC and Agreement State licensees other than nuclear power reactors (50161). It believes rescission is acceptable since NRC has committed to propose a rule to constrain air emissions from such licensees to a level that would result in a dose of no

Continued on page 7

QUANTERRA INC. (West Sacramento)

STANDARD OPERATING PROCEDURE CHANGE FORM

SOP Number:	LM-CAL-2041
SOP Title:	Analysis of Metals by Inductively Coupled Plasma/Mass Spectrometry
SOP Sections Affected by Change:	9.3.2
Reason for Addition or Change:	Limits were updated to reflect current method requirements.
Change Effective From [Date]:	
Change or Addition (Specific Section; use additional sheets if necessary.):	9.3.2 Average DCS recovery must be within ± 20% of the expected value.

Submitted by/Date: John Barnett

Approved by:*

Technical Reviewer: *Date:*

EH&S Signature: *Date:*

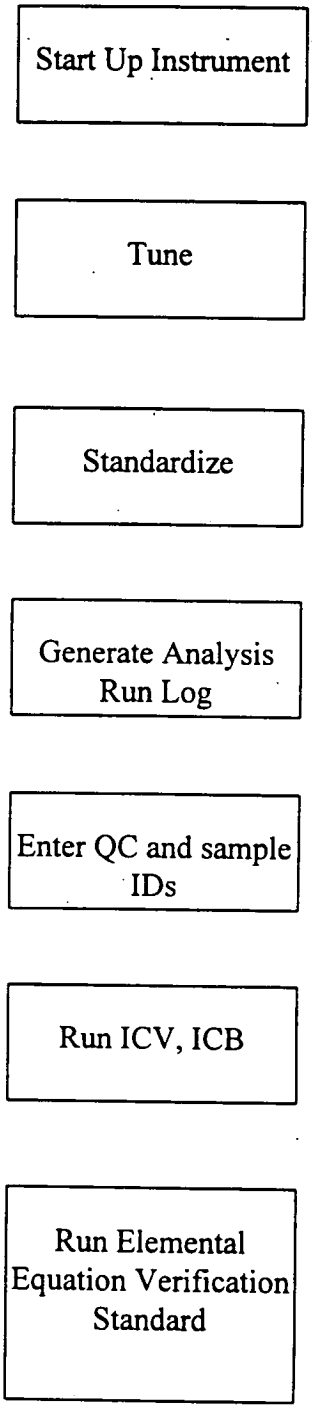
QA Signature: *Date:*

Management Signature: *Date*

Must be same signature authorities of SOP being revised.
d:\\qsacca01\sacqa\sop\amend.dot

C4-240

Flow Diagram



C4-241

Run batch QC and samples.

Run CCV, CCB

Flow Diagram

Start Up Instrument

Tune

Standardize

Generate Analysis Run Log

Enter QC and sample IDs

Run ICV, ICB

Run Elemental Equation Verification Standard

Run batch QC and samples.

Run CCV, CCB

Quanterra Environmental Services

QUANTERRA ENVIRONMENTAL SERVICES (West Sacramento)
SOP CHANGE FORM

SOP Number:	
SOP Title:	
SOP Sections Affected by Change:	
Reason for Addition or Change:	
Change Effective From [Date]:	
Change or Addition (Specific Section; Use additional sheets if necessary):	_____

Submitted by/Date: _____

Approved by:*

Technical Reviewer: _____ Date _____

EH&S Signature: _____ Date _____

QA Signature: _____ Date _____

Management Signature: _____ Date _____

* Must be same signature authorities of SOP being revised.

C4-245

Controlled Copy
Copy No._____

QUANTERRA STANDARD OPERATING PROCEDURE

TITLE: PREPARATION AND ANALYSIS OF MERCURY IN SOLID SAMPLES BY COLD VAPOR ATOMIC ABSORPTION SPECTROSCOPY, SW846 7471A AND MCAWW 245.5

(SUPERSEDES: REVISION 0)

Prepared by: _____
Principal Scientist, Inorganics

Reviewed by: _____
Technology Standardization Committee

Approved by: _____
Director, Quality Assurance

Approved by: _____
Director, Environmental Health and Safety

Approved by: _____
Management

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C4-246

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C4-247

1. SCOPE AND APPLICATION

1.1. This procedure describes the preparation and analysis of mercury (Hg, CAS # 7439-97-6) by Cold Vapor Atomic Absorption Spectroscopy (CVAA) using SW-846 Method 7471A and MCAWW Method 245.5.

1.2. CVAA analysis provides for the determination of total mercury (organic and inorganic). The combination of the oxidants, potassium permanganate and potassium persulfate, has been found to give 100% recovery with both types of compounds. Detection limits, sensitivity and optimum concentration ranges for mercury analysis will vary with the matrices, instrumentation and volume of sample used.

1.3. Methods 7471A and 245.5 are applicable to the preparation and analysis of mercury in soils, sediments, bottom deposits and sludge-type materials. All matrices require sample preparation prior to analysis.

1.4. The Quanterra reporting limit for mercury in solid matrices is 0.1 mg/kg based a 0.2 g sample aliquot (wet weight).

2. SUMMARY OF METHOD

2.1. This SOP describes a technique for the determination of mercury in solution. The procedure is a physical method based on the absorption of radiation at 253.7 nm by mercury vapor. A representative portion of the sample is digested in sulfuric and nitric acids. Organic mercury compounds are oxidized with potassium permanganate and potassium persulfate and the mercury reduced to its elemental state with stannous chloride and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer. Absorbance is measured as a function of mercury concentration. Concentration of the analyte in the sample is determined by comparison of the sample absorbance to the calibration curve (absorbance vs. concentration).

3. DEFINITIONS

3.1. Total Metals: The concentration determined on an unfiltered sample following digestion.

4. INTERFERENCES

Chemical and physical interferences may be encountered when analyzing samples using this method.

4.1. Potassium permanganate which is used to breakdown organic mercury compounds also eliminates possible interferences from sulfide. Concentrations as high as 20 mg/L of sulfide as sodium sulfide do not interfere with the recovery of inorganic mercury from reagent water.

4.2. Copper has also been reported to interfere; however, copper concentrations as high as 10 mg/L had no effect on the recovery of mercury from spiked samples.

4.3. Chlorides can cause a positive interference. Samples high in chlorides require additional permanganate (as much as 25 mL) because, during the oxidation step, chlorides are converted to free chlorine, which also absorbs radiation at 253.7 nm. Care must be taken to ensure that free chlorine is absent before the mercury is reduced and swept into the cell. This is accomplished by adding excess hydroxylamine reagent (25 mL) and purging the sample headspace before stannous chloride is added. Both inorganic and organic mercury spikes have been quantitatively recovered from seawater using this technique.

Note: Sufficient addition of permanganate is apparent when the purple color persists at least 15 minutes. Some samples may require dilution prior to digestion due to extremely high concentrations of chloride.

4.4. Interference from certain volatile organic materials that absorb at this wavelength may also occur. If suspected, a preliminary run without stannous chloride can determine if this type of interference is present. While the possibility of absorption from certain organic substances present in the sample does exist, this problem is not routinely encountered. This is mentioned only to caution the analyst of the possibility. If this condition is found to exist, the mercury concentration in the sample can be determined by subtracting the result of the sample run without the reducing reagent (stannous chloride) from that obtained with the reducing reagent.

4.5. Samples containing high concentrations of oxidizable organic materials, as evidenced by high COD levels, may not be completely oxidized by this procedure. When this occurs the recovery of mercury will be low. The problem can be eliminated by reducing the volume of original sample used.

4.6. The most common interference is laboratory contamination which may arise from impure reagents, dirty glassware, improper sample transfers, dirty work areas, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them.

5. SAFETY

5.1. Procedures shall be carried out in a manner that protects the health and safety of all Quanterra associates.

5.2. Eye protection that satisfies ANSI Z87.1 (as per the Chemical Hygiene Plan), laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.3. The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the Material Safety Data Sheets (MSDS) maintained in the laboratory. The following specific hazards are known:

5.3.1. The following materials are known to be **corrosive:**

hydrochloric acid, nitric acid and sulfuric acid.

5.3.2. The following materials are known to be **oxidizing agents**:

nitric acid, potassium permanganate, potassium persulfate and magnesium perchlorate.

5.3.3. Mercury is a highly toxic element that must be handled with care. The analyst must be aware of the handling and clean-up techniques before working with mercury. Since mercury vapor is toxic, precaution must be taken to avoid its inhalation, ingestion or absorption through skin. All lines should be checked for leakage and the mercury vapor must be vented into a hood or passed through a mercury absorbing media such as:

5.3.3.1. Equal volumes of 0.1 M $KMnO_4$ and 10% H_2SO_4, or

5.3.3.2. Iodine, 0.25%, in a 3% KI solution.

5.3.4. Magnesium sulfate is known to be a reproductive toxin (mutagen).

5.4. Exposure to chemicals must be maintained **as low as reasonably achievable.** Therefore, unless they are known to be non-hazardous, all samples should be opened, transferred and prepared in a fume hood, or under other means of mechanical

ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.5. The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operation will permit.

5.6. All work must be stopped in the event of a known or potential compromise to the health and safety of a Quanterra associate. The situation must be reported **immediately** to a laboratory supervisor.

5.7. Do not look directly into the beam of the Hg lamp. The UV light that these lamps radiate is harmful to the eyes.

5.8. Cylinders of compressed gas must be handled with caution, in accordance with local regulations. It is recommended that, wherever possible, cylinders be located outside the laboratory and the gas led to the instrument through approved lines.

5.9. The CVAA apparatus must be properly vented to remove potentially harmful fumes generated during sample analysis.

6. EQUIPMENT AND SUPPLIES

6.1. Temperature controlled water bath (capable of maintaining temperature of 90- 95 °C) or autoclave capable of obtaining 15 lbs., 120 °C.

6.2. Atomic Absorption Spectrophotometer equipped with:

6.2.1. Absorption Cell with quartz end windows perpendicular to the longitudinal axis. Dimensions of the cell must result in sufficient sensitivity to meet the SOP defined reporting limit. The quartz windows must be maintained to provide accurate measurements. Any scratches or fingerprints can alter the absorption of UV radiation.

6.2.2. Mercury specific hollow cathode lamp (HCL) or electrodeless discharge lamp (EDL).

6.2.3. Peristaltic pump which can deliver 1 L/min air.

6.2.4. Flowmeter capable of measuring an airflow of 1 L/min.

6.2.5. Recorder or Printer.

6.2.6. Aeration Tubing: A straight glass frit having a course porosity and Tygon tubing is used for the transfer of mercury vapor from the sample bottle to the absorption cell and return.

6.2.7. Drying device (a drying tube containing magnesium perchlorate or magnesium sulfate and/or a lamp with a 60 W bulb) to prevent condensation in cell. The lamp is positioned to shine on the absorption cell maintaining the air temperature in the cell about 10 °C above room temperature. Other drying devices that acheive the same purpose are also acceptable (i.e., Gortex filter).

Note: Instruments designed specifically for the measurement of mercury using the cold vapor technique may be substituted for the atomic absorption spectrophotometer.

6.3. BOD bottles or equivalent.

6.4. Nitrogen or argon gas supply, welding grade or equivalent.

6.5. Calibrated automatic pipettes or Class A glass volumetric pipettes.

6.6. Class A volumetric flasks.

6.7. Top-loading balance, capable of reading up to two decimal places.

6.8. Thermometer (capable of accurate readings at 95 °C).

6.9. Disposable cups or tubes.

7. REAGENTS AND STANDARDS

7.1. Reagent water must be produced by a Millipore DI system or equivalent. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

7.2. Stock (1000 ppm) mercury standards (in 10% HNO_3) are purchased as custom Quanterra solutions. All standards must be stored in FEP fluorocarbon or previously unused polyethylene or polypropylene bottles. Stock standard solutions must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the stock solutions may be used for up to one year and must be replaced sooner if verification from an independent source indicates a problem.

7.3. Intermediate mercury standard (10 ppm): Take 1 mL of the stock mercury standard (7.2) and dilute to 100 mL with reagent water. The intermediate standard must be made monthly and must be prepared in a matrix of 2% HNO_3. This acid (2 mL of concentrated HNO_3) must be added to the flask/bottle before the addition of the stock standard aliquot.

7.4. Working mercury standard (0.1 ppm): Take 1 mL of the intermediate mercury standard (7.3) and dilute to 100 mL with reagent water. The working mercury standard must be made daily and must be prepared in a matrix of 0.15% HNO_3. This acid (150 uL of concentrated HNO_3) must be added to the flask/bottle before the addition of the stock standard aliquot.

7.5. The calibration standards must be prepared fresh daily from the working standard (7.4) by transferring 0, 0.2, 0.5, 1.0, 2.0, 5.0 and 10.0 mL aliquots of the working mercury standard into sample prep bottles and proceeding as specified in Section 11.1

Note: Alternate approaches to standard preparation may be taken and alternate volumes of standard may be prepared as long as the accuracy and final standard concentrations as detailed in Table I are maintained. For example, automated mercury systems do not require 100 mL of standard and therefore smaller volumes may be generated to reduce waste generation.

7.6. The initial calibration verification standard must be made from a different stock solution than that of the calibration standards.

7.7. Refer to Table I (Appendix A) for details regarding the working standard concentrations for calibration, calibration verification and spiking solutions. All standards must be processed through the entire analytical procedure including sample preparation.

7.8. Nitric acid (HNO_3), concentrated, trace metal grade or better.

Note: If a high reagent blank is obtained, it may be necessary to distill the nitric acid.

7.9. Sulfuric acid (H_2SO_4), concentrated, trace metal grade or better.

7.9.1. Sulfuric acid, 0.5 N: Dilute 14.0 mL of concentrated H_2SO_4 to 1 liter with reagent water.

7.10. Hydrochloric acid (HCl), concentrated, trace metal grade or better.

7.11. Aqua Regia: Prepare immediately before use by carefully adding three volumes of concentrated HCl to one volume of concentrated HNO_3.

7.12. Stannous sulfate solution: Add 25 g of stannous sulfate to 250 mL of 0.5 N sulfuric acid. This mixture is a suspension and should appear cloudy. This solution should be made daily and should be stirred continuously during use.

Note: Stannous chloride may be used in place of stannous sulfate. Prepare the stannous chloride solution according to the recommendations provided by the instrument manufacturer.

7.13. Sodium chloride-hydroxylamine hydrochloride solution: Add 12 g of sodium chloride and 12 g of hydroxylamine hydrochloride to every 100 mL of reagent water.

Note: Hydroxylamine sulfate may be used in place of hydroxylamine hydrochloride.

7.14. Potassium permanganate, 5% solution (w/v): Dissolve 5 g of potassium permanganate for every 100 mL of reagent water.

7.15. Potassium persulfate, 5% solution (w/v): Dissolve 5 g of potassium persulfate for every 100 mL of reagent water.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Sample holding time for mercury is 28 days from time of collection to the time of sample analysis.

8.2. Soil samples do not require preservation but must be stored at 4° C ± 2° C until the time of analysis.

9. QUALITY CONTROL

Table II (Appendix A) provides a summary of quality control requirements including type, frequency, acceptance criteria and corrective action.

9.1. Initial Demonstration of Capability

Prior to the analysis of any analyte using 7471A or the 245.5, the following requirements must be met.

9.1.1. Method Detection Limit (MDL) - An MDL must be determined for each analyte/matrix prior to the analysis of any samples. The MDL is determined using seven replicates of reagent water, spiked with all the analytes of interest,

that have been carried through the entire analytical procedure. MDLs must be redetermined on an annual basis in accordance with 40 CFR Part 136 Appendix B requirements. The spike level must be between the calculated MDL and 10X the MDL to be valid. The result of the MDL determination must be below the Quanterra reporting limit.

9.1.2. Initial Demonstration Study - This requires the analysis of four QC check samples. The QC check sample is a well characterized laboratory generated sample used to monitor method performance. The results of the initial demonstration study must be acceptable before analysis of samples may begin.

9.1.2.1. Four aliquots of the check sample (LCS) are prepared and analyzed using the procedures detailed in this SOP and the determinative SOPs.

9.2. Preparation Batch - A group of up to 20 samples that are of the same matrix and are processed together using the same procedures and reagents. The preparation batch must contain a method blank, a LCS and a matrix spike/matrix spike duplicate. In some cases, at client request, it may be appropriate to process a matrix spike and sample duplicate in place of the MS/MSD. If clients specify specific samples for MS/MSD, the batch may contain multiple MS/MSD pairs.

9.3. Sample Count - Laboratory generated QC samples (method blanks, LCS) are not included in the sample count for determining the size of a preparation batch. MS/MSD are not included in the sample count unless there are multiple sets of MS/MSD per batch. In other words, the first MS/MSD are not counted; all additional MS and MSDs are counted as samples.

9.4. Method Blank (MB) - One method blank must be processed with each preparation batch. The method blank consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. The method blank should not contain any analyte of interest at or above the reporting or at or above 5% of the measured concentration of that analyte in associated samples, whichever is higher (sample result must be a minimum of 20 times higher than the blank contamination level).

- Repreparation and reanalysis of all samples associated with an unacceptable method blank is required when reportable concentrations are determined in the samples (see exception noted above).

- If there is no analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. **Such action must be taken in consultation with the client and must be addressed in the project narrative.**

- If the above criteria are not met and reanalysis is not possible, then the sample data must be qualified. **This anomaly must be addressed in the project narrative and the client must be notified.**

9.5. Laboratory Control Sample (LCS) - One LCS must be processed with each preparation batch. The LCS is used to monitor the accuracy of the analytical process. On-going monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines. The LCS must be carried through the entire analytical procedure. The CCV results can be reported as the LCS results since all CCVs (as well as all other standards) are processed through the sample preparation step with the field samples. No more than 20 samples can be associated with one CCV used for the purpose of reporting LCS data.

- If the LCS is outside established control limits the system is out of control and corrective action must occur. Until in-house control limits are established, a control limit of 80 - 120% recovery must be applied.

- In the instance where the LCS recovery is > 120% and the sample results are < RL, the data may be reported with qualifiers. Such action must be taken in consultation with the client and must be addressed in the case narrative."

- In the event that an MS/MSD analysis is not possible, a Laboratory Control Sample Duplicate (LCSD) must be analyzed. The RPD of the LCS and LCSD must be compared to the matrix spike RPD limits.

- Corrective action will be repreparation and reanalysis of the batch unless the client agrees that other corrective action is acceptable.

9.6. Matrix Spike/Matrix Spike Duplicate (MS/MSD) - One MS/MSD pair must be processed for each preparation batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Some client specific data quality objectives (DQO's) may require the use of sample duplicates in place of or in addition to MS/MSD's. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, these results may have immediate bearing

only on the specific sample spiked. Samples identified as field blanks cannot be used for MS/MSD analysis. Spiking levels are provided in Table I (Appendix A).

- If analyte recovery or RPD falls outside the acceptance range, the recovery of that analyte must be in control for the LCS. Until in-house control limits are established, a control limit of 75 - 125 % recovery and 20% RPD must be applied to the MS/MSD. If the LCS recovery is within limits, then the laboratory operation is in control and the results may be accepted. If the recovery of the LCS is outside limits, corrective action must be taken. Corrective action will include repreparation and reanalysis of the batch. MS/MSD results which fall outside the control limits must be addressed in the narrative.

- If the native analyte concentration in the MS/MSD exceeds 4 times the spike level for that analyte, the recovery data are reported as NC (i.e., not calculated). If the reporting software does not have the ability to report NC then the actual recovery must be reported and narrated as follows: "Results outside of limits do not necessarily reflect poor method performance in the matrix due to high analyte concentrations in the sample relative to the spike level."

- If an MS/MSD is not possible due to limited sample volume, then a laboratory control sample duplicate (LCSD) should be analyzed. The RPD of the LCS and LCSD must be compared to the matrix spike RPD limits.

9.7. Initial Calibration Verification (ICV/ICB) - Calibration accuracy is verified by analyzing a second source standard (ICV). The ICV result must fall within 20% of the true value for that solution. An ICB is analyzed immediately following the ICV to monitor low level accuracy and system cleanliness. The ICB result must fall within +/- the reporting limit (RL) from zero. If either the ICV or ICB fail to meet criteria, the analysis should be terminated, the problem corrected and the instrument recalibrated. (See Section 11.2.10 and Section 11.2.11 for required run sequence). If the cause of the ICV or ICB failure was not directly instrument related the corrective action will include repreparation of the associated samples.

9.8. Continuing Calibration Verification (CCV/CCB) - Calibration accuracy is monitored throughout the analytical run through the analysis of a known standard after every 10 samples. The CCV must be a mid-range standard at a concentration other than that of the ICV. The CCV result must fall within 20% of the true value for that solution. A CCB is analyzed immediately following each CCV. (See Section 11.2.10 and 11.2.11 for required run sequence.) The CCB result must fall within +/- RL from zero. Each CCV and CCB analyzed must reflect the conditions of analysis of all associated samples. Sample results may only be reported when bracketed by valid ICV/CCV

and ICB/CCB pairs. If a mid-run CCV or CCB fails, the analysis must be terminated, the problem corrected, the instrument recalibrated, the calibration verified and the affected samples reanalyzed. If the cause of the CCV or CCB failure was not directly instrument related the corrective action will include repreparation of the associated samples.

9.9. Method of Standard Addition (MSA) -This technique involves adding known amounts of standard to one or more aliquots of the sample prior to preparation. This technique compensates for a sample interferent that may enhance or depress the analyte signal, thus producing a different slope from that of the calibration standards. It will not correct for additive interferences which cause a baseline shift. Refer to Section 11.2.12 for additional information on when full 4 point MSA is required as well as Appendix C for specific MSA requirements.

10. CALIBRATION AND STANDARDIZATION

10.1. Calibration standards must be processed through the preparation procedure as described in Section 11.1.

10.2. Due to the differences in preparation protocols separate calibration and calibration verification standards must be prepared for aqueous and solid matrices.

10.3. Calibration must be performed daily (every 24 hours) and each time the instrument is set up. The instrument calibration date and time must be included in the raw data.

10.4. Set up the instrument with the operating parameters recommended by the manufacturer. Allow the instrument to become thermally stable before beginning calibration (approximately 30 minutes of warm-up is required). Refer to the facility specific instrument SOP and CVAA instrument manual for detailed setup and operation protocols.

10.5. Calibrate the instrument according to instrument manufacturer's instructions, using a minimum of five standards and a blank. One standard must be at the Quanterra reporting limit. Analyze standards in ascending order beginning with the blank. Refer to Section 7.5 and Table I for additional information on preparing calibration standards and calibration levels.

10.6. The calibration curve must have a correlation coefficient of ≥0.995 or the instrument shall be stopped and recalibrated prior to running samples. Sample results can not be reported from a curve with an unacceptable correlation coefficient.

10.7. Refer to Section 9.0 for calibration verification procedures, acceptance criteria and corrective actions.

11. PROCEDURE

11.1. Standard and Sample Preparation:

11.1.1. All calibration and calibration verification standards (ICV, ICB, CCV, CCB) are processed through the digestion procedure as well as the field samples.

11.1.2. Transfer 0, 0.2, 0.5, 1.0, 2.0, 5.0 and 10.0 mL aliquots of the working standard (7.5) into a series of sample digestion bottles.

Note: Alternate volumes of standard may be prepared as long as the accuracy and final standard concentrations as detailed in Table I are maintained.

11.1.3. Add reagent water to each standard bottle to make a total volume of 23.5 mL. Continue preparation as described under 11.1.5 or 11.1.6 below.

11.1.4. Transfer triplicate 0.2 g or 0.6g total portions of a well mixed sample into a clean sample digestion bottle. Continue preparation as described under 11.2.2 or 11.2.3 below.

11.1.5. Water Bath protocol:

11.1.5.1. To each **standard** bottle: Add 5 mL of aqua regia.
To each **sample** bottle: Add 5 mL of reagent water and 5 mL of aqua regia.

11.1.5.2. Heat for 2 minutes in a water bath at 90 - 95 ° C.

11.1.5.3. Cool.

11.1.5.4. Add 50 mL of distilled water.

11.1.5.5. Add 15 mL of potassium permanganate solution.

11.1.5.6. Add 8 mL of potassium persulfate solution, mix thoroughly

11.1.5.7. Heat for 30 minutes in the water bath at 90 - 95 °C.

11.1.5.8. Cool.

11.1.5.9. Add 6 mL of sodium chloride-hydroxylamine sulfate solution to reduce the excess permanganate.

11.1.5.10. To each **standard** bottle: Add 50 mL of reagent water. To each **sample** bottle: Add 55 mL of reagent water.

11.1.5.11. Continue as described under Section 11.2.

11.1.6. Autoclave protocol:

11.1.6.1. Add 5 mL concentrated of H_2SO_4 and 2.5 mL of concentrated HNO_3.

11.1.6.2. Add 5 mL of saturated potassium permanganate solution.

11.1.6.3. Add 8 mL of potassium persulfate solution.

11.1.6.4. Cover digestion bottle with aluminum foil or screw cap loosely applied.

11.1.6.5. Heat samples at 121 °C and 15 lbs. pressure for 15 minutes.

11.1.6.6. Cool.

11.1.6.7. At this point, there should be 44ml total volume in each standard and sample bottle.

Note: Alternate final volumes may be used as long as the standards and sample are treated the same way.

11.1.6.8. Add 6 mL of sodium chloride-hydroxylamine sulfate solution to reduce excess permanganate.

11.1.6.9. Continue as described under Section 11.2 .

11.2. Sample Analysis:

11.2.1. Because of differences between various makes and models of CVAA instrumentation, no detailed operating instructions can be provided. Refer to

the facility specific instrument operating SOP and the CVAA instrument manual for detailed setup and operation protocols.

11.2.2. All labs are required to detail the conditions/programs utilized for each instrument within the facility specific instrument operation SOP.

11.2.3. Manual determination:

11.2.3.1. Treating each sample individually, purge the head space of the sample bottle for at least one minute.

11.2.3.2. Add 5 mL of stannous chloride solution and immediately attach the bottle to the aeration apparatus.

11.2.3.3. Allow the sample to stand quietly without manual agitation while the sample is aerated (1 L/min flow). Monitor the sample absorbance during aeration. When the absorbance reaches a maximum and the signal levels off, open the bypass valve and continue aeration until the absorbance returns to its baseline level. Close the bypass valve and remove the aeration device.

11.2.3.4. Place the aeration device into 100 mLs of 1% HNO_3 and allow to bubble rinse until the next sample is analyzed.

11.2.4. Automated determination: Follow instructions provided by instrument manufacturer.

11.2.5. Perform a linear regression analysis of the calibration standards by plotting maximum response of the standards vs. ug of mercury. Determine the mercury concentration in the samples from the linear regression fit of the calibration curve. Calibration using computer or calculation based regression curve fitting techniques on concentration/response data is acceptable.

11.2.6. All measurements must fall within the defined calibration range to be valid. Dilute and reanalyze all samples for analytes that exceed the highest calibration standard.

11.2.7. If the sample results are negative and the absolute value of the negative result is greater than the reporting limit, the sample must be diluted and reanalyzed.

11.2.8. The samples must be allowed to cool to room temperature prior to analysis or a decrease in the response signal can occur.

11.2.9. Baseline correction is acceptable as long as it is performed after every sample or after the CCV and CCB; resloping is acceptable as long as it is immediately preceded and followed by a compliant CCV and CCB.

11.2.10. The following analytical sequence must be used with 7471A and 245.5:

Instrument Calibration
ICV
ICB
Maximum 10 samples
CCV
CCB
Repeat sequence of 10 samples between CCV/CCB pairs as required to complete run
CCV
CCB

Refer to Quality Control Section 9.0 and Table II (Appendix A) for quality control criteria to apply to Methods 7471A and 245.5.

Note: Samples include the method blank, LCS, MS, MSD, duplicate, field samples and sample dilutions.

11.2.11. The following run sequence is consistent with 7471A, CLP and 245.5 and may be used as an alternate to the sequence in 11.2.10. This run sequence is recommended if multiple method requirements must be accommodated in one analytical run:

Instrument Calibration
ICV
ICB
CRA*
CCV
CCB
10 samples
CCV
CCB
Repeat sequence of 10 samples between CCV/CCB pairs as required to complete run.

CCV
CCB

Refer to the appropriate CLP SOP (CORP-MT-0008) for quality control requirements for QC samples.

* Refer to the CLP SOP for information on the CRA.

11.2.12. TCLP samples are always prepped as waters:

1) recovery of the analyte in the matrix spike is not at least 50%,

2) the concentration of the analyte does not exceed the regulatory level, and,

3) the concentration of the analyte is within 20% of the regulatory level.

Appendix E provides guidance on performing MSA analyses. For TCLP mercury determinations, MSA spikes must be added prior to sampe preparation.

11.3. To facilitate the early identification of QC failures and samples requiring rerun it is strongly recommended that sample data be reviewed periodically throughout the run.

11.4. Guidelines are provided in the appendices on procedures to minimize contamination of samples and standards, preventive maintenance and troubleshooting.

11.5. One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and is approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

11.6. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

12. DATA ANALYSIS AND CALCULATIONS

12.1. ICV percent recoveries are calculated according to the equation:

$$\%R = 100\left(\frac{Found(ICV)}{True(ICV)}\right)$$

12.2. CCV percent recoveries are calculated according to the equation:

$$\%R = 100\left(\frac{Found(CCV)}{True(CCV)}\right)$$

12.3. Matrix spike recoveries are calculated according to the following equation:

$$\%\ R = 100\left(\frac{SSR - SR}{SA}\right)$$

Where:
SSR = Spike Sample Result
SR = Sample Result
SA = Spike Added

12.4. The relative percent difference (RPD) of matrix spike/matrix spike duplicates or sample duplicates are calculated according to the following equations:

$$RPD = 100\left[\frac{|MSD - MS|}{\left(\frac{MSD + MS}{2}\right)}\right]$$

Where:
MS = determined spiked sample concentration
MSD = determined matrix spike duplicate concentration

$$RPD = 100\left[\frac{|DU1 - DU2|}{\left(\frac{DU1 + DU2}{2}\right)}\right]$$

Where:
DU1 = Sample result
DU2 = Sample duplicate result

12.5. For automated determinations, the final concentration determined in solid samples when reported on a dry weight basis is calculated as follows:

mg/kg, dry weight = (C x V x D)/(W x S)

Where:

C = Concentration (ug/L) from instrument readout
V = Volume of digestate (L)
D = Instrument dilution factor
W = Weight in g of wet sample digested
S = Percent solids/100

Note: A Percent Solids determination must be performed on a separate aliquot when dry weight concentrations are to be reported. If the results are to be reported on a wet weight basis, the "S" factor should be omitted from the above equation.

12.6. For manual (total) determinations, the final concentration determined in solid samples when reported on a dry weight basis is calculated as follows:

$$mg/kg,\ dry\ weight = (C)/(W \times S)$$

Where:

C = Concentration (ug) from instrument readout
W = Weight in g of wet sample digested
S = Percent solids/100

Note: A Percent Solids determination must be performed on a separate aliquot when dry weight concentrations are to be reported. If the results are to be reported on a wet weight basis, the "S" factor should be omitted from the above equation.

12.7. The LCS percent recovery is calculated according to the following equation:

$$\%R = 100\left(\frac{Found(LCS)}{True(LCS)}\right)$$

12.8. Sample results should be reported with up to three significant figures in accordance with the Quanterra significant figure policy.

13. METHOD PERFORMANCE

13.1. Each laboratory must have initial demonstration of performance data on file for each analyte of interest as described in Section 9.0.

13.2. Method performance is determined by the analysis of method blank, laboratory control sample, matrix spike and matrix spike duplicate samples. The matrix spike recovery should fall within +/- 25 % and the matrix spike duplicates should compare within 20% RPD. The method blanks must meet the criteria in Section 9.3. The

laboratory control sample should recover within 20% of the true value until in house limits are established.

13.3. Training Qualification:

The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience.

14. POLLUTION PREVENTION

14.1. This method does not contain any specific modifications that serve to minimize or prevent pollution.

15. WASTE MANAGEMENT

15.1. Waste generated in the procedure must be segregated and disposed according to the facility hazardous waste procedures. The Environmental Health and Safety Director should be contacted if additional information is required.

16. REFERENCES

16.1. Test Methods for Evaluating Solid Waste , Physical/Chemical Methods, SW-846, 3rd Edition, Final Update II, Revision I, September 1994, Method 7471A (Mercury).

16.2. "Methods for the Chemical Analysis of Water and Wastes", EPA-600/4-79-020, U.S.EPA, August 1983, Method 245.5.

16.3. U.S.EPA Statement of Work for Inorganics Analysis, ILMO3.0.

16.4. QA-003, Quanterra QC Program.

16.5. QA-004, Rounding and Significant Figures.

16.6. QA-005, Method Detection Limits.

17. MISCELLANEOUS (TABLES, APPENDICES, ETC. . .)

17.1. Modifications/Interpretations from reference method.

17.1.1. Modifications from both 7471A and 245.5.

17.1.1.1. A potassium persulfate oxidation step has been included to facilitate the breakdown of organic mercurials which are not completely oxidized by potassium permanganate. Use of potassium persulfate in combination with the permanganate improves the recovery of mercury from organo-mercury compounds. The use of persulfate has been incorporated in several recent EPA mercury protocols.

17.1.1.2. The alternate run sequence presented in Section 11.2.11 is consistent with method requirements. An additional QC analysis (CRA) was added to accommodate the CLP protocol requirements.

17.1.2. Modifications from Method 7471A

17.1.2.1. Chapter 1 of SW846 specify the use of reagent water with a purity equivalent to ASTM Type II water. This SOP specifies the use of a Millipore DI system or equivalent to produce reagent water. This SOP requires that reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

17.1.2.2. Chapter 1 of SW-846 states that the method blank should not contain any analyte of interest at or above the MDL. This SOP states that the method blank must not contain any analyte of interest at or above the reporting limit.

17.1.2.3. Method 7471A does not state control criteria within the text of the method. The QC section of 7471A refers the analyst to Section 8.0 of Method 7000A, the generic atomic absorption method, which discusses flame and furnace methods. The ICV criteria stated in Method 7000A is ± 10%. This SOP requires ICV control limits of ± 20% based on the fact that the mercury ICV, unlike the ICV for the flame and furnace analytes, is digested and therefore is equivalent to a LCS. The CLP protocol 245.5 CLP-M recognizes this factor and requires control limits of ± 20%.

17.1.3. Modifications from 245.5

17.1.3.1. Method 245.5 Section 9.3 states concentrations should be reported as follows: Between 0.1 and 1 ug/g, to the nearest 0.01 ug; between 1 and 10 ug/g, to the nearest 0.1ug; above 10 ug/g, to the nearest ug. Quanterra reports all Hg results under this SOP to two significant figures.

17.2. Modifications from previous SOP

None.

17.3. Facility Specific SOPs

Each facility shall attach a list of facility specific SOPs or approved attachments (if applicable) which are required to implement this SOP or which are used in conjunction with this SOP. If no facility specific SOPs or amendments are to be attached, a statement must be attached specifying that there are none. Refer to the Appendices for any facility specific information required to support this SOP.

17.4. Documentation and Record Management

The following documentation comprises a complete CVAA raw data package:

- Raw data (direct instrument printout)
- Run log printout from instrument software where this option is available or manually generated run log. (A bench sheet may be substituted for the run log as long as it contains an accurate representation of the analytical sequence).
- Data review checklist - See Appendix B
- Standards Documentation (source, lot, date).
- Copy of digestion log.
- Non-conformance summary (if applicable).

Figure 1. Solid Sample Preparation for Mercury - Autoclave Procedure

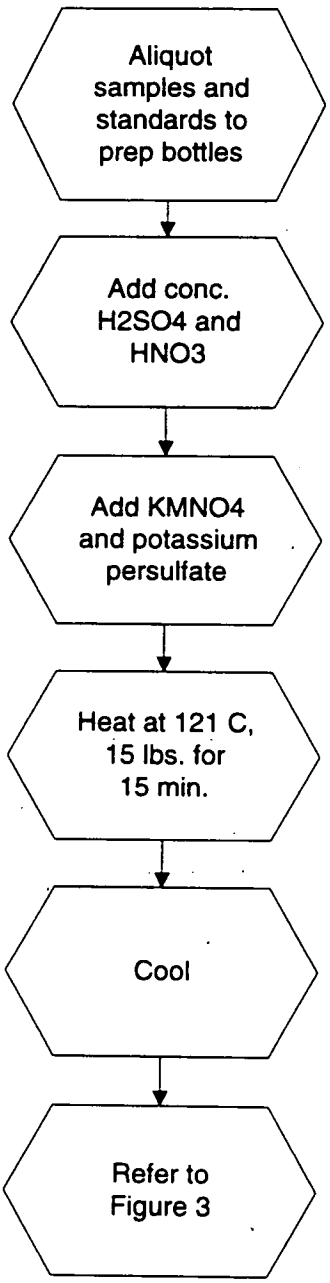


Figure 2. Solid Sample Preparation for Mercury - Water Bath Procedure

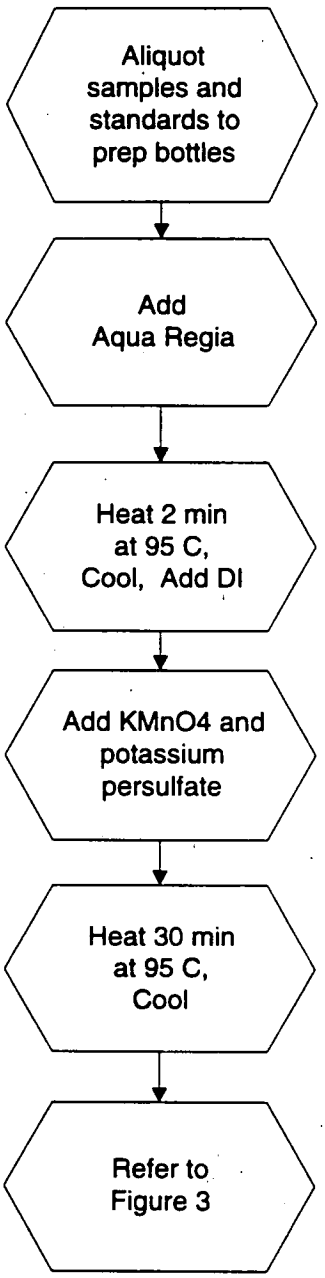
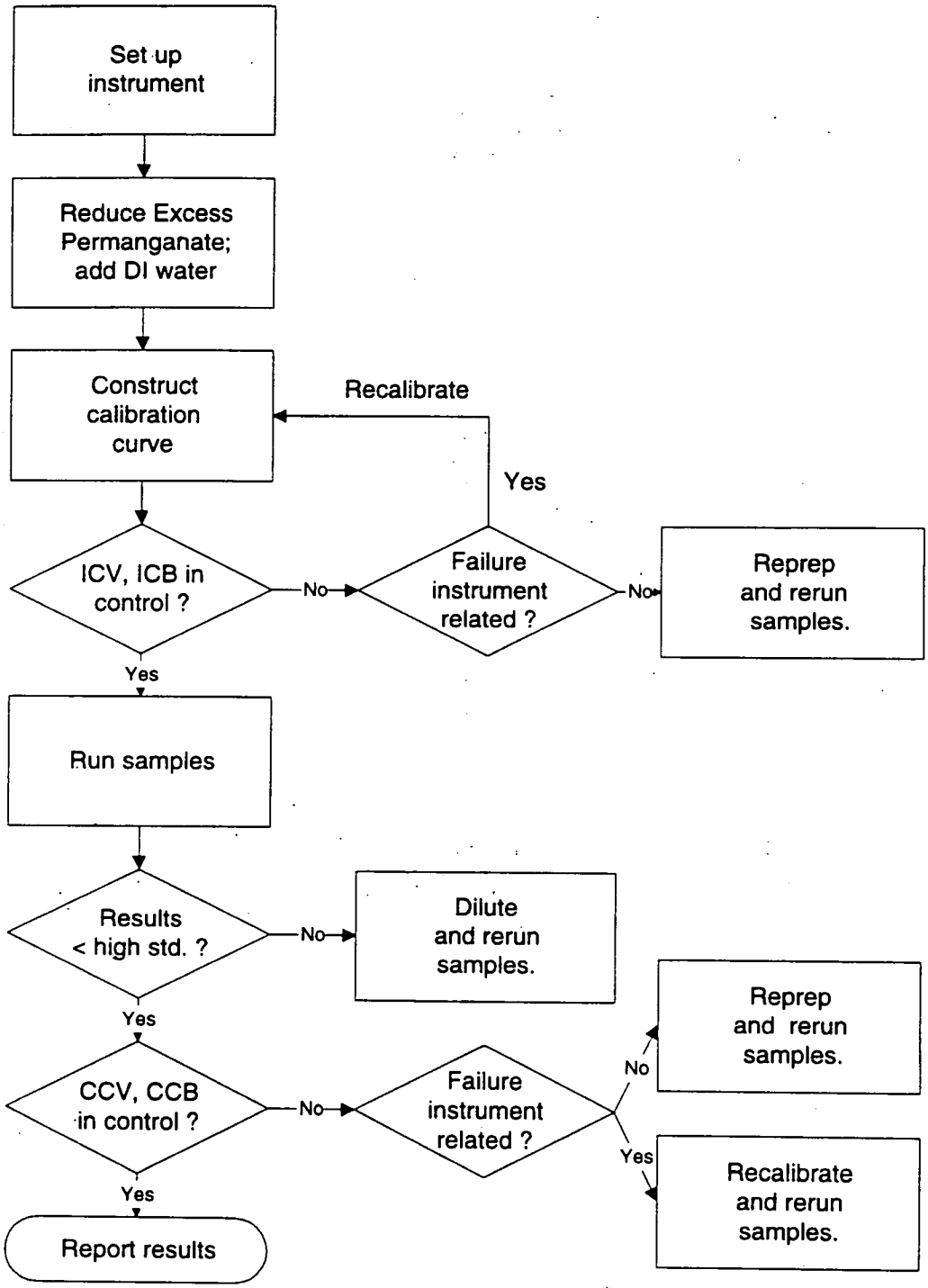


Figure 3. CVAA Mercury Analysis



APPENDIX A

TABLES

TABLE I. MERCURY REPORTING LIMITS, CALIBRATION STANDARD*, QC STANDARD AND SPIKING LEVELS

Soil RL (mg/kg)	0.1
Std 0 (mg/L)	0
Std 1 (mg/L)	0.0002
Std 2 (mg/L)	0.0005
Std 3 (mg/L)	0.001
Std 4 (mg/L)	0.002
Std 5 (mg/L)	0.005
Std 6 (mg/L) **	0.010
ICV (mg/L)	0.001 or 0.0025 ***
Our water LCS for soils currently is being spiked at 1.0 ppb. Our spiked at 5.0 ppb	0.0025 or 0.005 ***
MS (mg/L)	0.001

* **SOP specified calibration levels must be used unless prevented by the instrument configuration or client specific requirements. Deviations from specified calibration levels must be documented in the facility specific instrument operation SOP and must be approved by the facility technical manager and Quality Assurance Manager.**

** **Optional standard which may be used to extend the calibration range as allowed by the instrument configuration. If the instrument configuration prevents the use of 6 standards, the 2 ppb standard may be eliminated in favor of the 10 ppb standard.**

*** **Concentration level dependent on high calibration standard used. CCV must be 50% of the high standard concentration and the ICV must be 20-25% of the high standard concentration.**

TABLE II. Summary Of Quality Control Requirements

QC PARAMETER	FREQUENCY *	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
ICV	Beginning of every analytical run.	80 - 120 % recovery.	Terminate analysis; Correct the problem; Recalibrate or reprep batch (see Section 9.7).
ICB	Beginning of every analytical run, immediately following the ICV.	The result must be within +/- RL from zero.	Terminate analysis; Correct the problem; Recalibrate or reprep batch (see Section 9.7).
CCV	Every 10 samples and at the end of the run.	80 - 120 % recovery.	Terminate analysis; Correct the problem; Recalibrate and rerun all samples not bracketed by acceptable CCV or reprep batch (see Section 9.8).
CCB	Immediately following each CCV.	The result must be within +/- RL from zero.	Terminate analysis; Correct the problem; Recalibrate and rerun all samples not bracketed by acceptable CCB or reprep batch (see Section 9.8).
Method Blank	One per sample preparation batch of up to 20 samples.	The result must be less than or equal to the RL. Sample results greater than 20x the blank concentration are acceptable. Samples for which the contaminant is < RL do not require redigestion (See Section 9.4)	Redigest and reanalyze samples. Note exceptions under criteria section. See Section 9.4 for additional requirements.

*See Sections 11.2.10 and 11.2.11 for exact run sequence to be followed.

TABLE II. Summary of Quality Control Requirements (Continued)

QC PARAMETER	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Laboratory Control Sample (LCS)	One per sample preparation batch of up to 20 samples.	Aqueous LCS must be within 80 - 120% recovery or in-house control limits.	Terminate analysis; Correct the problem; Redigest and reanalyze all samples associated with the LCS (see Section 9.5).
Matrix Spike	One per sample preparation batch of up to 20 samples.	75 - 125 % recovery or in-house control limits. If the MS/MSD is out for an analyte, it must be in control in the LCS.	In the absence of client specific requirements, flag the data; no flag required if the sample level is > 4x the spike added. (see Section 9.6) For TCLP see Section 11.3.12
Matrix Spike Duplicate	See Matrix Spike	75 - 125 % recovery or in-house control limits; RPD ≤ 20%. (See MS)	See Corrective Action for Matrix Spike.

APPENDIX B
QUANTERRA Hg DATA REVIEW CHECKLIST

C4-276

Quanterra Hg Data Review Checklist

Run/Project Information

Run Date: _____ **Analyst:** _____ **Instrument:** _____
Prep Batches Run: _____

Circle Methods used: **7470A / 245.1 :** CORP-MT-0005 Rev 1 **7471 / 245.5** : CORP-MT-0007 Rev 1
CLP - AQ : CORP-MT-0006 Rev 0 **CLP - SOL** : CORP-MT-0008 Rev 0

Review Items

A. Calibration/Instrument Run QC	Yes	No	N/A	2ndLevel
1. Instrument calibrated per manufacturer's instructions and at SOP specified levels ?				
2. ICV/CCV analyzed at appropriate frequency and within control limits?				
3. ICB/CCB analyzed at appropriate frequency and within +/- RL or +/- CRDL (CLP)?				
4. CRA run (CLP only)?				
B. Sample Results				
1. Were samples with concentrations > the high calibration standard diluted and reanalyzed?				
2. All reported results bracketed by in control QC ?				
3. Sample analyses done within holding time?				
C. Preparation/Matrix QC				
1. LCS done per prep batch and within QC limits ?				
2. Method blank done per prep batch and < RL or CRDL (CLP) ?				
3. MS run at required frequency and within limits ?				
4. MSD or DU run at required frequency and RPD within SOP limits?				
D. Other				
1. Are all nonconformances documented appropriately ?				
2. Current IDL/MDL data on file?				
3. Calculations and Transcriptions checked for error ?				
4. All client/ project specific requirements met?				
5. Date of analysis verified as correct ?				

Analyst: _____ **Date:** _____
Comments:

_____

_____

2nd Level Reviewer : _____ **Date:** _____

_____

_____

APPENDIX C

MSA GUIDANCE

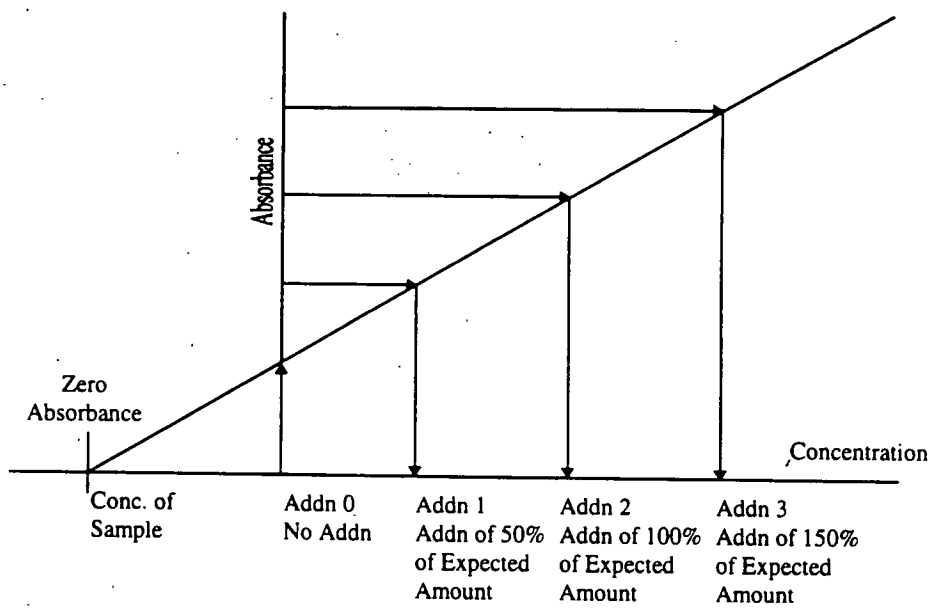
APPENDIX C. MSA GUIDANCE

Method of Standard Addition

Four equal volume aliquots of sample are measured and known amounts of standards are added to three aliquots. The fourth aliquot is the unknown and no standard is added to it. The concentration of standard added to the first aliquot should be 50% of the expected concentration. The concentration of standard added to the second aliquot should be 100% of the expected concentration and the concentration of standard added to the third aliquot should be 150% of the expected concentration. The volume of the unspiked and spiked aliquots should be the same (i.e., the volume of the spike added should be negligible in relation to the volume of sample).

To determine the concentration of analyte in the sample, the absorbance (or response) of each solution is determined and a linear regression performed. On the vertical axis the absorbance (or response) is plotted versus the concentrations of the standards on the horizontal axis using 0 as the concentration of the unspiked aliquot. An example plot is shown in Figure 1. When the resulting line is extrapolated back to zero absorbance, the point of interception of the horizontal axis is the concentration of the unknown. Calculate the correlation coefficient (r) and the x-intercept (where y=0) of the curve. The concentration in the digestate is equal to the negative x-intercept.

Figure 1



- For the method of standard additions to be correctly applied, the following limitations must be taken into consideration.
- The plot of the sample and standards must be linear over the concentration range of concern. For best results, the slope of the curve should be similar to that of a plot of the aqueous standard curve.
- The effect of the interference should not vary as the ratio of the standard added to the sample matrix changes.

APPENDIX D

TROUBLESHOOTING GUIDE

APPENDIX D. TROUBLESHOOTING GUIDE

Problem	Possible Cause
Poor or No Absorbance or Sensitivity Check failed	Incorrect wavelength Dirty windows Window loose Etched or dirty optics Wrong lamp Bad lamp Not enough or no sample introduced Empty sample cup Incorrectly made standards Gas leak EDL power supply set on "Continuous"
Erratic Readings	Source lamp not aligned properly Lamp not prewarmed Injection tip partially clogged Contaminated reagents Contaminated glassware Drying tube saturated Bad lamp Injection tip hitting outside of tube Injection tip coated or not set properly Leak in sample tubing Power fluctuations Air bubbles in tubing
EDL Won't Light	Lamp cable not plugged in Lamp power set at 0 Lamp is dead Power supply fuse is blown Short in cord
Standards reading twice or half normal absorbance or concentration	Incorrect standard used Incorrect dilution performed Dirty cell
Background Correction Light Blinking	Background screen or attenuator faulty

APPENDIX E

CONTAMINATION CONTROL GUIDELINES

APPENDIX E. CONTAMINATION CONTROL GUIDELINES

The following procedures are strongly recommended to prevent contamination:

All work areas used to prepare standards and spikes should be cleaned before and after each use.

All glassware should be washed with detergent and tap water and rinsed with 1:1 nitric acid followed by deionized water.

Proper laboratory housekeeping is essential in the reduction of contamination in the metals laboratory. All work areas must be kept scrupulously clean.

Powdered or Latex Gloves must not be used in the metals laboratory since the powder contains silica and zinc, as well as other metallic analytes. Only vinyl or nitrile gloves should be used in the metals laboratory.

Glassware should be periodically checked for cracks and etches and discarded if found. Etched glassware can cause cross contamination of any metallic analytes.

Autosampler trays should be covered to reduce the possibility of contamination. Trace levels of elements being analyzed in the samples can be easily contaminated by dust particles in the laboratory.

The following are helpful hints in the identification of the source of contaminants:

Reagents or standards can contain contaminants or be contaminated with the improper use of a pipette.

Improper cleaning of glassware can cause contamination.

Separate glassware if an unusually high sample is analyzed and soak with sulfuric acid prior to routine cleaning.

APPENDIX F

PREVENTIVE MAINTENANCE

APPENDIX F. PREVENTIVE MAINTENANCE

A maintenance log is used to record when maintenance is performed on instruments. When an instrument problem occurs indicate the date, time and instrument number, then identify the problem and corrective action in the maintenance log.

The following procedures are required to ensure that that the instrument is fully operational.

Cold Vapor Atomic Absorption (Leeman PS 200) [1]

Daily	Semi-annually	Annually
Check Optic Intensity	Check Hg lamp intensity. If intensity is below 500,000 clean cell and lenses	Change Hg lamp.
Check aperture.		Check liquid/gas separator.
Check argon flow.		
Check tubing.		
Check drain.		
Replace drying tube.		

Cold Vapor Atomic Absorption (PE 5000) [1]

Daily	Monthly
Clean aspirator by flushing with DI water.	Clean cell in aqua regia.
Check tubing and replace if needed.	Clean aspirator in aqua regia.
Clean windows with methanol.	
Change silica gel in drying tube.	
Check argon gas supply.	
Adjust lamp.	

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LABORATORY -SPECIFIC

QUANTERRA STANDARD OPERATING PROCEDURE

TITLE: GRAPHITE FURNACE ATOMIC ABSORPTION SPECTROSCOPY, SW-846 METHOD 7000A AND MCAWW 200 SERIES METHODS

(SUPERSEDES: CORP-MT-0003, REV. 1.0)

Prepared by: Stephen Spain

Reviewed by: _____
Technical Specialist, Robert Dovi

Approved by: _____
Quality Assurance Manager, Gail Cho Celaschi

Approved by: _____
Environmental Health and Safety Coordinator, Cheryl Ann Stith

Approved by: _____
Laboratory Manager, Roger Freize II

Reviewed by: _____
Corporate Technology and/or Corporate Quality Assurance

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C4-287

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1. SCOPE AND APPLICATION

1.1. This procedure describes the analysis of certain metals by Graphite Furnace Atomic Absorption Spectroscopy (GFAA) using SW-846 protocol, Method 7000A and the MCAWW 200 series methods. Table I (Appendix A) lists the routine elements approved for analysis by Method 7000A and the 200 series methods. Table IA (Appendix A) lists non-routine elements that may be analyzed under Method 7000A and the 200 series methods, provided the method performance criteria presented in Section 13.0 are met.

1.2. GFAA analysis provides for the determination of metal concentrations in the parts per billion range. Detection limits, sensitivity and optimum concentration ranges of the metals will vary with the matrices and instrumentation used. The sensitivity and limited linear dynamic range of GFAA often implies the need to dilute a sample prior to analysis. The actual magnitude of the dilution can dramatically influence the quality of the analytical results. Therefore, sample types requiring large dilutions (>50:1) should be analyzed by another client approved test procedure that has a larger linear dynamic range or that is inherently less sensitive than GFAA.

1.3. Method 7000A is applicable to the determination of dissolved, suspended, total recoverable and total elements in ground water, aqueous samples, solids, sludges, wastes, sediments, air sampling media, biological tissue, wipes and TCLP, EP and other leachates/extracts. All matrices require digestion prior to analysis, with the exception of analyses for dissolved metals in filtered and acidified aqueous samples. Although digestion is not specifically required by the method, some clients and regulators do require digestion of dissolved samples and this must be clarified before project initiation.

1.4. Series methods are applicable to the determination of dissolved, suspended, total recoverable and total elements in ground water, aqueous samples, solids, sludges, wastes, and sediments. All matrices require digestion prior to analysis, with the exception of analyses for dissolved metals in filtered and acidified aqueous samples if the criteria in Section 11.2 are met. Although digestion is not specifically required by the method, some clients and regulators do require digestion of dissolved samples and this must be clarified before project initiation.

1.5. This method is not applicable to drinking water samples due to the wide array of state specific requirements which must be accommodated. Refer to facility specific SOPs for guidance on performing drinking water analyses.

2. SUMMARY OF METHOD

2.1. This method describes a technique for the determination of elements in solution. The basis of the method is the measurement of atomic absorption by an optical spectroscopic technique. A representative aliquot of a sample is placed in a graphite tube in the furnace, evaporated to dryness, charred and atomized, causing the atoms to be vaporized and dissociated within the graphite tube. A light beam from a hollow cathode lamp (HCL) or electrodeless discharge lamp (EDL) is directed through the graphite tube. The intensity of the transmitted radiation decreases in proportion to the amount of the ground state atoms in the vapor contained within the graphite tube. Because the wavelength of the light beam is characteristic of the metal being determined, the light energy absorbed by the sample in the tube is a measure of the concentration of that metal in the sample. Concentration of the analyte in the sample is determined by comparison of the sample absorbance to the calibration curve (absorbance vs. concentration).

2.2. Consult the appropriate SOP's for details on sample preparation methods.

3. DEFINITIONS

3.1. Dissolved Metals: Those elements which pass through a 0.45 um membrane. (Sample is acidified after filtration).

3.2. Suspended Metals: Those elements which are retained by a 0.45 um membrane.

3.3. Total Metals: The concentration determined on an unfiltered sample following vigorous digestion.

3.4. Total Recoverable Metals: The concentration determined on an unfiltered sample following treatment with hot, dilute mineral acid.

4. INTERFERENCES

Chemical and physical interferences are prevalent when analyzing samples using these methods.

4.1. The problem of oxide formation is greatly reduced with furnace procedures because atomization occurs in an inert atmosphere. The technique, however, is still subject to chemical interferences. The composition of the sample matrix can have a major effect on the analysis and must be taken into consideration in the analysis of each different matrix encountered. The effects of the sample matrix are evaluated by following the spike recovery check procedure described in Section 9.7.

4.2. Matrix modifiers are added to the samples prior to the determination of most analytes. The modifier prevents premature loss of the analytes as volatile salts and/or helps prevent signal suppression during atomization due to the formation of high temperature stable complexes.

4.3. Background interferences must be compensated for by the use of background correction. Background correction is especially important below 350 nm. Certain samples, when atomized, may absorb or scatter light from the lamp. This can be caused by the presence of gaseous molecular species, salt particles, or smoke in the sample beam. Zeeman background correction must be used for all analysis. Zeeman background correction is effective in overcoming composition or structured background interferences. It is particularly useful when analyzing for As in the presence of Al and when analyzing for Se in the presence of Fe.

4.4. Interference from a smoke-producing sample matrix can sometimes be reduced by extending the charring time at a higher temperature or utilizing an ashing cycle in the presence of air, or by diluting the sample. Care must be taken, however, to prevent the loss of the analyte.

4.5. Samples containing large amounts of organic materials should be oxidized by acid digestion before being placed in the furnace. In this way, broad band absorption will be minimized.

4.6. Carbide formation, resulting from the chemical environment of the furnace, has been observed in certain elements that form carbides at higher temperatures (e.g., Mo, V). When carbides form, the metal is released very slowly from the resulting metal carbide during the atomization cycle. For example, molybdenum may require 30 sec or more atomization time for the signal to return to the baseline level. Carbide formation is greatly reduced and sensitivity increased with the use of pyrolytically coated graphite and must be used for all analysis.

4.7. Based on anion interference studies in the graphite furnace, it is generally accepted that nitrate is the preferred anion to combine with metal cations. Therefore, nitric acid is preferable for any digestion or solubilization step. If another acid in addition to nitric acid is required, a minimum amount should be used. This applies particularly to hydrochloric acid and, to a lesser extent, to sulfuric and phosphoric acids.

4.8. Memory effects occur when the analyte is not totally volatilized during atomization. The analyte which remains in the furnace can produce false positive signals on subsequent samples. This condition depends on several factors: volatility of the element and its chemical form, whether pyrolytic graphite is used, the rate of atomization and furnace design. The analyst should establish the analyte

concentration which can be injected into the furnace and adequately removed in one complete set of furnace cycles. This concentration represents the maximum concentration of analyte within a sample which will not cause a memory effect on the subsequent samples. If during a sample analysis run this concentration is exceeded, the tube should be cleaned by performing blank tube burns , as needed, to assure the memory interference has been eliminated before continuing sample analysis.

4.9. Specific element interferences

- High lead concentrations cause spectral interferences for **antimony** on the 217.6-nm line. The interference is eliminated by the use of Zeeman background correction or the use of the secondary wavelength. Antimony also suffers from an interference produced by K_2SO_4.

- **Arsenic** and **selenium** analyses are subject to many interferences. Elemental arsenic and selenium and their compounds are volatile and are subject to losses during sample preparation. Verify that the analyte is not volatilized using spike samples. A matrix modifier must be added to minimize volatilization during drying and ashing. Caution must be employed in selecting temperatures and times for the dry and char steps. In addition to the normal interferences, arsenic and selenium can suffer from severe nonspecific absorption and light scattering caused by matrix components during atomization. Arsenic analysis is particularly susceptible to these problems because of its low analytical wavelength (193.7 nm). Zeeman background correction is used to avoid erroneous high results for arsenic from aluminum interferences and low bias for selenium from iron interferences.

- **Cadmium** can be affected by severe nonspecific absorption and light scattering caused by matrix during atomization. To minimize this, simultaneous background correction is required. A matrix modifier solution can reduce the effects of premature volatilization from excess chlorides.

- Due to interferences from nonspecific absorption and scattering, background correction should be used when analyzing for **chromium**. Low concentrations of calcium and/or phosphate may also cause interferences. Chromium is very susceptible to carbide formation and memory effects. . Nitrogen should not be used as the purge gas because of a possible CN band interference.

- For **lead**, matrix modification should be used to suppress sulfate interferences.

- The use of hydrochloric acid should be avoided for **silver** analysis since silver chloride is insoluble. Storage of silver standards in brown bottles may prevent silver nitrates from plating on the container walls.

- Hydrochloric acid or excessive chlorides will cause volatilization of **thallium** at low temperatures. The use of spike samples helps to verify that losses are not occurring.

5. SAFETY

5.1. Procedures shall be carried out in a manner that protects the health and safety of all Quanterra associates.

5.2. Eye protection that satisfies ANSI Z87.1 (as per the Chemical Hygiene Plan), laboratory coat, and chemically resistant gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.Neoprene, natural rubber, nitrile and TRIonic® Cleanroom gloves provide varying degrees of protection against those chemicals listed. Refer to permeation/degradation charts for the actual data.

5.3. The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the Material Safety Data Sheets (MSDS) maintained in the laboratory. The following specific hazards are known:

5.3.1. The following materials are known to be **corrosive:**

hydrochloric acid, nitric acid, sulfuric acid, phosphoric and hydrofluoric acid.

5.3.2. The following materials are known to be **oxidizing agents**:

nitric acid and hydrogen peroxide.

5.4. Exposure to chemicals must be maintained **as low as reasonably achievable.** Therefore, unless they are known to be non-hazardous, all samples should be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.5. The preparation of standards,reagents, and the processing of samples will be conducted in a fume hood with the sash closed as far as the operation will permit.

5.6. The use of hydrofluoric acid requires special safety precautions. Consult the facility EH&S manager and laboratory supervisor for guidance.

NOTE: Contact may not be painful at first. The fluoride ion rapidly penetrates the skin and may cause delayed effects: lasting ulcerations, bone degeneration, pulmonary edema, muscle paralysis, and cardiac arrest. Any suspected exposure to HF liquid/fumes must be immediately evaluated by appropriate medical staff (i.e. U.C. Davis Medical Group Emergency Room.) Review Appendix H for a detailed first aid plan before work with HF begins.

5.6.1. The CA Emergency Medical Services Authority recommends a magnesium sulfate solution (Epsom salt) or lime water (CaOH) as effective irrigating solutions. Magnesium-containing antacids (Maalox® or Mylanta®) can be applied topically.

5.6.2. Some metals react with HF to release flammable hydrogen gas.

5.6.3. Glass reacts with HF to produce toxic silicon tetrafluoride.

5.7. All work must be stopped in the event of a known or potential compromise to the health and safety of a Quanterra associate. The situation must be reported **immediately** to a laboratory supervisor.

5.8. The Zeeman background correction technique utilizes a strong magnet. People with electronic cardiac pacemakers or similar devices that are affected by strong magnetic fields should not be near the instruments during operation.

5.9. Do not look directly into the beam of a HCL or EDL. The UV light that these lamps radiate is harmful to the eyes.

5.10. Do not look directly at the graphite tube when it is heated to incandescence to avoid possible injury to the eyes from the intense radiation.

5.11. The furnace heats to approximately 3000°C during atomization. Therefore, to avoid burns, be cautious not to touch the furnace housing until the furnace has cooled sufficiently.

5.12. Cylinders of compressed gas must be handled with caution, in accordance with local regulations. It is recommended that, wherever possible, cylinders be located outside the laboratory and the gas led to the instrument through approved lines.

5.13. The furnace must be properly vented with an exhaust hood directly over the furnace chamber to remove potentially harmful fumes generated when samples are heated.

6. EQUIPMENT AND SUPPLIES

6.1. Atomic Absorption Spectrophotometer equipped with graphite furnace, autosampler and background correction.

6.2. Hollow cathode (HCL) or electrodeless discharge lamp (EDL) with EDL power supply.

6.3. Argon gas supply, welding grade or equivalent.

6.4. Pyrolytically coated graphite tubes and platforms.

6.5. Coolflow or appropriate water cooling device.

6.6. Calibrated automatic pipettes or Class A glass volumetric pipettes.

6.7. Class A volumetric flasks.

6.8. Autosampler cups.

6.9. Disposable cups or tubes.

7. REAGENTS AND STANDARDS

7.1. Stock standards are purchased as custom Quanterra multi-element mixes or as single element solutions. All standards must be stored in FEP fluorocarbon or previously unused polyethylene or polypropylene bottles. Stock standard solutions must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the stock solutions may be used for up to one year and must be replaced sooner if verification from an independent source indicates a problem.

7.2. Working calibration and calibration verification solutions must be made daily. Standards must be prepared in a matrix of 2% HNO_3 per 100 mL for all analytes except antimony. Antimony standards must be made in a matrix of 2% HNO_3 and 5% HCl.

7.3. Refer to Tables III, IV, and V (Appendix A) for details regarding the working standard concentrations for calibration, calibration verification and spiking solutions.

7.4. Concentrated nitric acid (HNO_3), trace metal grade or better.

7.5. Concentrated hydrochloric acid (HCl), trace metal grade or better.

7.6. Hydrogen peroxide (H_2O_2), 30%.

7.7. Reagent water must be produced by a Millipore DI system or equivalent. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

7.8. Matrix modifiers as recommended by instrument manufacturers. The use of a palladium modifier is strongly recommended for the determination of all analytes. This will correct for general chemical interferences as well as allow for higher char and atomization temperatures without allowing the premature liberation of analyte.

8. SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1. Sample holding times for metals are six months from time of collection to the time of analysis.

8.2. Aqueous samples are preserved with nitric acid to a pH of <2 and may be stored in either plastic or glass. Refrigeration is not required. Preservation must be verified prior to analysis.

8.3. Soil samples do not require preservation but must be stored at 4°C ± 2° until the time of analysis.

9. QUALITY CONTROL

Table VI (Appendix A) provides a summary of quality control requirements including type, frequency, acceptance criteria and corrective action.

9.1. Initial Demonstration of Capability

Prior to the analysis of any analyte using 7000A or the 200 series methods, the following requirements must be met.

9.1.1. Instrument Detection Limit (IDL) -The IDL for each analyte must be determined for each analyte wavelength used on each instrument. The IDL must be determined annually. If the instrument is adjusted in any way that may affect the IDL, the IDL for that instrument must be redetermined. For example, the IDL must be redetermined after the instrument is moved or the internal optics cleaned. The IDL shall be determined by multiplying by 3, the standard deviation obtained from the analysis of a standard solution (each analyte in reagent water) at a concentration 3 - 5 times the previously determined IDL, with seven consecutive measurements. Each measurement

must be performed as though it were a separate analytical sample (i.e., each measurement must be followed by a rinse and/or any other procedure performed between the analysis of separate samples). Analytical spikes are not required for IDL determinations by 7000A and 200 series methods. IDLs determined by the CLP procedure can be used for this method. The result of the IDL determination must be below the Quanterra reporting limit.

9.1.2. Method Detection Limit (MDL) - An MDL must be determined for each analyte/matrix prior to the analysis of any samples. The MDL is determined using seven replicates of reagent water, spiked with all the analytes of interest, that have been carried through the entire analytical procedure. MDLs must be redetermined on an annual basis in accordance with 40 CFR Part 136 Appendix B requirements as detailed in Quanterra QA Policy QA-005. The spike level must be between the calculated MDL and 10X the MDL to be valid. The result of the MDL determination must be below the Quanterra reporting limit.

9.2. Method Blank (MB) - One method blank must be processed with each preparation batch. The method blank consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. The method blank should not contain any analyte of interest at or above the reporting limit (exception: common laboratory contaminants, see below) or at or above 5% of the measured concentration of that analyte in associated samples, whichever is higher (sample result must be a minimum of 20 times higher than the blank contamination level).

- If the analyte is a common laboratory contaminant (copper, iron, lead or zinc) the data may be reported with a qualifier if the concentration of the analyte in the method blank is less than two times the RL (see Appendix G) **Such action must be taken in consultation with the client and must be addressed in the project narrative.**

- Repreparation and reanalysis of all samples associated with an unacceptable method blank is required when reportable concentrations are determined in the samples (see exception noted above).

- If there is no analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. **Such action must be taken in consultation with the client and must be addressed in the project narrative.**

- If the above criteria are not met and reanalysis is not possible, then the sample data must be qualified. **This anomaly must be addressed in the project narrative and the client must be notified.**

- For dissolved metals samples which have not been digested, a continuing calibration blank (CCB) result is reported as the method blank. The CCB run immediately prior to the start of the dissolved sample analyses must be used for this purpose. No more than 20 samples can be associated with one CCB.

9.3. Laboratory Control Sample (LCS) - One aqueous LCS must be processed with each preparation batch. The LCS must contain all analytes of interest and must be carried through the entire analytical procedure. The LCS is used to monitor the accuracy of the analytical process. Aqueous LCS spike levels are provided in Table III (Appendix A). On-going monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines.

- If any analyte is outside established control limits the system is out of control and corrective action must occur. Until in-house control limits are established, a control limit of 80 - 120% recovery must be applied.

- In the event that an MS/MSD analysis is not possible, a Laboratory Control Sample Duplicate (LCSD) must be analyzed. The RPD of the LCS and LCSD must be compared to the matrix spike RPD limits.

- In the instance where the LCS recovery is greater than 120% and the sample results are < RL, the data may be reported with qualifiers. **Such action must be taken in consultation with the client and must be addressed in the case narrative.**

- Corrective action will be repreparation and reanalysis of the batch unless the client agrees that other corrective action is acceptable.

- For dissolved metals samples which have not been digested, a continuing calibration verification (CCV) result is reported as the LCS. The CCV run immediately prior to the start of the dissolved sample analyses must be used for this purpose. No more than 20 samples can be associated with one CCV.

9.4. Matrix Spike/Matrix Spike Duplicate (MS/MSD) - One MS/MSD pair must be processed for each preparation batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Some client specific data quality objectives (DQO's) may require the use of sample duplicates in place of or in

addition to MS/MSD's. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, these results may have immediate bearing only on the specific sample spiked. Samples identified as field blanks cannot be used for MS/MSD analysis. Spiking levels are provided in Table III (Appendix A).

- If any analyte recovery or RPD falls outside the acceptance range, the recovery of that analyte must be in control for the LCS and the MS/MSD must be reanalyzed once to confirm the out of control event. Until in-house control limits are established, a control limit of 80 - 120 % recovery and 20% RPD must be applied to the MS/MSD. If the LCS recovery is within limits, then the laboratory operation is in control and the results may be accepted. If the recovery of the LCS is outside limits, corrective action must be taken. Corrective action will include repreparation and reanalysis of the batch. MS/MSD results which fall outside the control limits must be addressed in the narrative.

- If the native analyte concentration in the MS/MSD exceeds 4 times the spike level for that analyte, the recovery data are reported as NC (i.e., not calculated). If the reporting software does not have the ability to report NC then the actual recovery must be reported and narrated as follows: "Results outside of limits do not necessarily reflect poor method performance in the matrix due to high analyte concentrations in the sample relative to the spike level."

- If an MS/MSD is not possible due to limited sample volume, then a laboratory control sample duplicate (LCSD) should be analyzed. The RPD of the LCS and LCSD must be compared to the matrix spike RPD limits.

- For dissolved metals samples which have not been digested, a MS/MSD must be performed per batch of up to 20 samples by spiking two aliquots of the sample at the levels specified in Table III (Appendix A).

9.5. Initial Calibration Verification (ICV/ICB) - Calibration accuracy is verified by analyzing a second source standard (ICV). The ICV result must fall within 10% of the true value for that solution. An ICB is analyzed immediately following the ICV to monitor low level accuracy and system cleanliness. The ICB result must fall within +/- the reporting limit (RL) from zero. If either the ICV or ICB fail to meet criteria, the analysis should be terminated, the problem corrected and the instrument recalibrated. (See Section 11.14 for required run sequence).

9.6. Continuing Calibration Verification (CCV/CCB) - Calibration accuracy is monitored throughout the analytical run through the analysis of a known standard after every 10

samples. The CCV must be a mid-range standard at a concentration other than that of the ICV. For analyses that require 200 series methods the CCV result must fall within 10% of the true value. For Method 7000A, the CCV result must fall within 20% of the true value for that solution. A CCB is analyzed immediately following each CCV. (See Section 11.14 and 11.15 for required run sequence.) The CCB result must fall within +/- RL from zero. Each CCV and CCB analyzed must reflect the conditions of analysis of all associated samples. Sample results may only be reported when bracketed by valid ICV/CCV and ICB/CCB pairs. If a mid-run CCV or CCB fails, the analysis must be terminated, the problem corrected, the instrument recalibrated, the calibration verified and the affected samples reanalyzed.

9.7. Spike Recovery Check (AS) - In order to verify the presence or absence of matrix interferences, a post digest analytical spike is analyzed for each sample, including the method blank. A post digest spike is not required on the LCS or matrix spike sample. The analytical spike of a sample must be run immediately after that sample. See Table III (Appendix A) for spiking levels. The percent recovery of the spike will then determine how the sample will be quantitated and reported as follows:

- If the spike recovery is less than 40%, the sample must be diluted and rerun with another spike (recommended dilution 5-10X) and flagged with the appropriate qualifier (see Appendix G). If the spike recovery still falls less than 40% the client should be contacted and an alternate method of analysis recommended (i.e., Trace or ICP).

- If the spike recovery is greater than or equal to 40% and less than 85%, report the data with the following flag: "Post digestion spike recovery fell between 40-85% due to matrix interference."

- If the spike recovery is greater than or equal to 85% and less than or equal to 115%, report the data unflagged.

- If the spike recovery is greater than 115% and less than 150% and the sample result is less than the Quanterra reporting limit, then report the data unflagged.

- If the spike recovery is greater than 115% and less than 150% and the sample result is greater than the Quanterra reporting limit, then report the data with the following flag "1": "Post digestion spike recovery fell between 115-150% due to matrix interference."

- If the spike recovery is greater than 150% and the sample result is less than the Quanterra reporting limit, then report the data unflagged.

- If the spike recovery is greater than 150% and the sample result is greater than the Quanterra reporting limit then dilute the sample and rerun with another spike. Until the spike recovery falls less than 150%, this dilution step must be repeated.

- If the method blank (MB) analytical spike recovery is less than 85% or greater than 115%, the spiking solution must be verified by respiking and rerunning the MB once. If the MB analytical spike is still out of control, correct the problem and reanalyze all analytical samples associated with that blank.

9.8. Method of Standard Addition (MSA) -This technique involves adding known amounts of standard to one or more aliquots of the processed sample solution. This technique compensates for a sample interferent that may enhance or depress the analyte signal, thus producing a different slope from that of the calibration standards. It will not correct for additive interferences which cause a baseline shift. By definition, the recovery check procedure defined in Section 9.7 is a single point MSA. Sample results are not biased corrected based on the single point MSA result unless this is specifically requested by the client. Refer to Section 11.16 for additional information on when full 4 point MSA is required as well as Appendix C for specific MSA requirements.

9.9. Quality Assurance Summaries - Certain clients may require specific project or program QC which may supersede the SOP requirements. Quality Assurance Summaries (QAS) should be developed to address these requirements.

10. CALIBRATION AND STANDARDIZATION

10.1. Set up the instrument with the operating parameters recommended by the manufacturer. Allow the instrument to become thermally stable before beginning calibration (approximately 30 minutes of warm-up is required). Refer to the facility specific instrument SOP and GFAA instrument manual for detailed setup and operation protocols.

10.2. Calibrate the instrument according to instrument manufacturer's instructions, using a minimum of three standards and a blank. See Table V (Appendix A) for calibration standard details. One standard must be at the Quanterra reporting limit as defined in Tables I and IA (Appendix A). Calibration standards must be prepared fresh daily.

10.3. Calibration must be performed daily (every 24 hours) and each time the instrument is set up for an element. The instrument calibration date and time must be included in the raw data.

10.4. Refer to Section 9.0 for calibration verification procedures, acceptance criteria and corrective actions.

11. PROCEDURE

11.1. Because of differences between various makes and models of GFAA instrumentation, no detailed operating instructions can be provided. Refer to the facility specific instrument operating SOP and the GFAA instrument manual for detailed setup and operation protocols.

- Table II (Appendix A) provides recommended GFAA analytical guidelines for wavelength, char and atomization temperatures, and tube type.

- All labs are required to detail the furnace conditions/programs utilized for each analyte/instrument within the facility specific instrument operation SOP.

11.2. For 200 series analyses, dissolved samples must be digested unless it can be documented that the sample meets all of the following criteria:

A. COD is < 20 ppm.
B. Visibly transparent with a turbidity measurement of 1 NTU or less.
C. Colorless with no perceptible odor.
D. Is of one liquid phase and free of particulate or suspended matter following acidification.

11.3. The use of an autosampler for all runs is required. The use of an autosampler for the addition of matrix modifier and analytical post digestion spikes is strongly recommended if the instrument in use is capable of the automated additions.

11.4. To reduce the number of reruns due to dilutions it is recommended that whenever possible the samples be run by ICP first to "screen" the samples prior to GFAA analysis. The information obtained from the ICP run will enable the analyst to either eliminate the GFAA analysis completely (i.e., if sufficiently high report ICP result with client approval) or dilute the samples appropriately. Screening also may prevent potential contamination of the analytical system as well as other samples during the instrument run.

11.5. To facilitate the early identification of QC failures and samples requiring rerun it is strongly recommended that sample data be reviewed periodically throughout the run.

11.6. To facilitate the data review and reporting processes it is strongly recommended that all necessary dilutions and post digestion spikes be performed before closing out the instrument run.

11.7. Daily, prior to the analysis of samples, a sensitivity check must be performed and recorded. The sensitivity of the instrument may be checked by calculating the

characteristic mass. If the daily results do not compare within +/- 30% of the reference sensitivity the instrument may need adjustment. Refer to Appendix D for assistance if the criterion is not met.

11.8. At a minimum, single burn analyses will be performed. When double burns are performed, the CV or RSD must be less than or equal to 20% if the sample results are greater than the reporting limit.

11.9. All measurements must fall within the defined calibration range to be valid. Dilute and reanalyze all samples for analytes that exceed the highest calibration standard. Acid strength must be maintained in the dilution of samples through the use of acidified dilution water (2% HNO_3).

11.10. If the sample results are negative and the absolute value of the negative result is greater than the reporting limit, the sample must be diluted and reanalyzed.

11.11. Samples requiring large dilutions due to the presence of high concentrations of analyte may be reported using alternate techniques (i.e., ICP, flame) with the approval of the client. Whenever possible, samples should be processed by ICP, prior to GFAA, to facilitate in determining appropriate dilution factors or eliminate the GFAA analysis altogether.

11.12. Baseline correction or cleanout burns are acceptable as long as it is performed after every sample or after the CCV and CCB; resloping is acceptable as long as it is immediately preceded and followed by a compliant CCV and CCB. Cleanout burns must be counted as injections when determining instrument QC frequency.

11.13. Graphite tubes may be changed during an analytical run, providing the last samples analyzed on the tube are a compliant CCV and CCB, and the first samples analyzed on the new tube are a compliant CCV and CCB.

11.14. The following analytical sequence must be used with 7000A and 200 series methods:

Instrument Calibration (Blank and three standards)
ICV
ICB
Maximum 10 samples
CCV
CCB
Maximum 10 samples
CCV
CCB

Repeat sequence of 10 samples between CCV/CCB pairs as required to complete run
CCV
CCB

Refer to Quality Control Section 9.0 and Table VI (Appendix A) for quality control criteria to apply to Methods 7000A and the 200 series methods.

Note: Samples include the method blank, LCS, MS, MSD, duplicate, field samples and sample dilutions. Spike recovery check (AS) injections are not considered as independent samples for the purpose of determining instrument QC frequency.

11.15. The following run sequence is consistent with 7000A, CLP and 200 series, and may be used as an alternate to the sequence in 11.14. This run sequence is recommended if multiple method requirements must be accommodated in one analytical run:

Instrument Calibration (Blank and three standards)
ICV
ICB
CRA**
CCV
CCB
5* samples with AS (maximum 20 burns)
CCV
CCB
5* samples with AS (maximum 20 burns)
CCV
CCB
Repeat sequence of 5* samples between CCV/CCB pairs as required to complete run.
CCV
CCB

Refer to the appropriate CLP SOP (CORP-MT-0004) for quality control requirements for QC samples.

* Note- Only 5 samples can be analyzed between CCV/CCB because double burn analyses are performed as required by CLP protocols.

**Note- Refer to the CLP SOP for information on the CRA.

11.16. For TCLP samples, full four point MSA will be required if all of the following conditions are met:

1) recovery of the analyte in the matrix spike is not at least 50%,

2) the concentration of the analyte does not exceed the regulatory level, and,

3) the concentration of the analyte is within 20% of the regulatory level.

The reporting and regulatory limits for TCLP analyses as well as matrix spike levels are detailed in Table IV (Appendix A). Appendix C provides guidance on performing MSA analyses.

11.17. Guidelines are provided in the appendices on procedures to minimize contamination of samples and standards, preventive maintenance and troubleshooting.

11.18. One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and is approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

11.19. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

12. DATA ANALYSIS AND CALCULATIONS

12.1. ICV percent recoveries are calculated according to the equation:

$$\%R = 100\left(\frac{Found(ICV)}{True(ICV)}\right)$$

12.2. CCV percent recoveries are calculated according to the equation:

$$\%R = 100\left(\frac{Found(CCV)}{True(CCV)}\right)$$

12.3. Analytical spike and matrix spike recoveries are calculated according to the following equation:

$$\% R = 100\left(\frac{SSR - SR}{SA}\right)$$

Where:

SSR = Spike Sample Result
SR = Sample Result
SA = Spike Added

NOTE: When the sample concentration is less than the IDL, use SR = 0 for purposes of calculating analytical spike recovery.

12.4. The relative percent difference (RPD) of matrix spike/matrix spike duplicates or sample duplicates are calculated according to the following equations:

$$RPD = 100\left[\frac{|MSD - MS|}{\left(\frac{MSD + MS}{2}\right)}\right]$$

Where:

MS = determined spiked sample concentration
MSD = determined matrix spike duplicate concentration

$$RPD = 100\left[\frac{|DU1 - DU2|}{\left(\frac{DU1 + DU2}{2}\right)}\right]$$

Where:

DU1 = Sample result
DU2 = Sample duplicate result

12.5. The final concentration for a digested aqueous sample is calculated as follows:

$$mg/L = \frac{C \times V1 \times D}{V2}$$

Where:

C = Concentration (mg/L) from instrument readout

D = Instrument dilution factor
V1 = Final volume in liters after sample preparation
V2 = Initial volume of sample digested in liters

12.6. The final concentration determined in digested solid samples when reported on a dry weight basis is calculated as follows:

$$mg / Kg, dry\ weight = \frac{C \times V \times D}{W \times S}$$

Where:
C = Concentration (mg/L) from instrument readout
D = Instrument dilution factor
V = Final volume in liters after sample preparation
W = Weight in Kg of wet sample digested
S = Percent solids/100

Note: A Percent Solids determination must be performed on a separate aliquot when dry weight concentrations are to be reported. If the results are to be reported on a wet weight basis, the "S" factor should be omitted from the above equation.

12.7. The LCS percent recovery is calculated according to the following equation:

$$\%R = 100\left(\frac{Found(LCS)}{True(LCS)}\right)$$

12.8. Appropriate factors must be applied to sample values if dilutions are performed.

12.9. Sample results should be reported with up to three significant figures in accordance with the Quanterra significant figure policy.

13. METHOD PERFORMANCE

13.1. Each laboratory must have initial demonstration of performance data on file for each analyte of interest as described in Section 9.0.

13.2. Refer to Tables I & IA in Appendix A for the list of routine as well as non-routine analytes that may be analyzed using this SOP.

13.3. Method performance is determined by the analysis of matrix spike and matrix spike duplicate samples as well as method blanks and laboratory control samples. The matrix spike recovery should fall within +/- 20 % and the matrix spike duplicates

should compare within 20% RPD. Method blanks must meet the criteria specified in Section 9.2. The laboratory control samples should recover within 20% of the true value until in house control limits are established.

13.4. Training Qualification:

The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience.

14. POLLUTION PREVENTION

14.1. Standards and/or solutions are not approved for disposal to the sinks.

15. WASTE MANAGEMENT

15.1. Waste generated in the procedure must be segregated and disposed according to the facility hazardous waste procedures. The Environmental Health and Safety Director should be contacted if additional information is required.

15.2. Standards should be purchased and prepared in volumes consistent with laboratory use to minimize the volume of expired standards to be disposed.

15.3. All waste must be disposed of according to the facility hazardous waste management procedures, Attachment C of the Quanterra Inc., Chemical Hygiene Plan, Section WS002, Table 1.

15.4. Samples and other solutions containing high concentrations of toxic materials must be disposed of according to the facility hazardous waste management procedures, Attachment C of the Quanterra, Inc., Chemical Hygiene Plan, Section WS003, Disposal of Samples After Analysis.

16. REFERENCES

16.1. Test Methods for Evaluating Solid Waste , Physical/Chemical Methods, SW-846, 3rd Edition, Final Update I, July 1992. Methods 7000A, 7060A, 7740, 7421, 7841, 7041, 7131A, 7191, 7211 and 7761.

16.2. Methods for the Chemical Analysis of Water and Waste, 1983.

16.3. CFR Part 136, Table IB, 7-1-92.

16.4. CORP-MT-0004, Graphite Furnace Atomic Absorption Spectroscopy Method, 2XX.X - CLP-M, SOW ILMO3.0.

16.5. QA-003, Quanterra QC Program.

16.6. QA-004, Rounding and Significant Figures.

16.7. QA-005, Method Detection Limits.

16.8. LS-CAL-2002, STANDARDS AND QC SOLUTIONS PREPARATION-METALS.

16.9. SAC-QA-0004, MAINTENANCE AND CALIBRATION CHECK OF FIXED AND ADJUSTABLE VOLUME AUTOPIPETTORS.

17. MISCELLANEOUS (TABLES, APPENDICES, ETC. . .)

17.1. Modifications/Interpretations from reference method.

17.1.1. Modifications from both 7000A and 200 series methods.

17.1.1.1. The 200 series methods and Chapter 1 of SW846 specify the use of reagent water with a purity equivalent to ASTM Type II water. This SOP specifies the use of a Millipore DI system or equivalent to produce reagent water. This SOP requires that reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

17.1.1.2. The alternate run sequence presented in Section 11.15 is consistent with method requirements. Additional QC analyses were added to accommodate the CLP protocol requirements.

17.1.2. Modifications from Method 7000A.

17.1.2.1. Chapter 1 of SW-846 states that the method blank should not contain any analyte of interest at or above the MDL. This SOP states that the method blank must not contain any analyte of interest at or above the reporting limit. Common lab contaminants are allowed up to two times the reporting limit in the blank following consultation with the client.

17.1.2.2. The SW846 methods contain contradictory statements regarding the frequency of CCV analysis. Section 7.3.6 of Method 7000A

states a check standard should be run after approximately every 10 sample injections. Section 8.3 (Quality Control) states a check sample must be run every 10 samples. Since Quanterra defines an analytical sample as a sample with its corresponding interference test and the SW846 document contains discrepancies in the frequency requirements for CCV analysis, this SOP requires that a CCV be analyzed every 10 analytical samples or 20 burns, whichever is more frequent.

17.1.2.3. Section 1.2 of Method 7000A states "To ensure valid data with furnace techniques, the analyst must examine each matrix for interference effects and, if detected, treat them accordingly, using either successive dilution, matrix modification, or method of standard addition." Quanterra utilizes matrix modifiers for all GFAA analyses and has also elected to run all samples using single point MSA rather than spike one "representative" sample per matrix type. The serial dilution test is presented in Method 7000A as a means to determine if interferences are present. Since the Quanterra SOP proceeds directly to single point MSA on all samples the use of the serial dilution test is not incorporated in this SOP. Sample results are not biased corrected based on the single point MSA result unless this is specifically requested by the client.

17.1.2.4. The SW846 methods contain contradictory statements regarding the acid matrix for calibration standards. Section 5.7 of Method 7000A states that the calibration standards should be prepared using the same type of acid or combination of acids and at the same concentration as will result in the samples following processing. Section 5.0 in each of the 7000 series methods states differing specific concentrations of acids in each method (i.e., Method 7421 states a 0.5% HNO_3 matrix). Since the addition of nitric acid to samples acts as a modifier (i.e., improves the recovery of the analyte) and the digestion procedures used incorporate higher nitric acid concentrations than specified in the individual methods, this SOP requires a 2% HNO_3 matrix for all standards.

17.1.3. Modifications from 200 series methods.

17.1.3.1. Method 200.0 section 9.3.7 states that a check standard should be run after approximately every 10 sample injections. Method 200.0 section 10.2.2 states that the curve must be verified every 20 samples. Since Quanterra defines an analytical sample as a sample

with its corresponding interference test and Method 200.0 contains discrepancies in the frequency requirements for CCV analysis, this SOP requires that a CCV be analyzed every 10 analytical samples or 20 burns whichever is more frequent.

17.1.3.2. The 200 series methods contain contradictory statements regarding the number of calibration standards to be used for calibration. Section 8.3 of the introductory method states to "Prepare a blank and at least four calibration standards using the same type of acid or combination of acids and at the same concentration as will result in the samples following processing. Section 10.2.1 of the same document states that a calibration curve is composed of a minimum of a reagent blank and three standards. This SOP requires the use of a blank and three standards.

17.1.3.3. Each analyte specific 200 series method specifies matrix modifiers. EPA recognizes that there have been many improvements in matrix modifier technology since these methods were originally written. In order to minimize interferences due to sample matrix, Quanterra has adopted the use of a palladium matrix modifier and/or currently recommended manufacturer modifiers.

17.2. Modifications from previous SOP

Facility specific flags were identified for method blanks, and MSA reporting.

17.3. Facility Specific SOPs

Each facility shall attach a list of facility specific SOPs or approved attachments (if applicable) which are required to implement this SOP or which are used in conjunction with this SOP. If no facility specific SOPs or amendments are to be attached, a statement must be attached specifying that there are none. Refer to the Appendices for any facility specific information required to support this SOP.

17.4. Documentation and Record Management

The following documentation comprises a complete GFAA raw data package:

- Raw data (direct instrument printout signed by analyst)
- Relevant sample preparation benchsheets.

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- Run log printout from instrument software where this option is available or manually generated run log. (A bench sheet may be substituted for the run log as long as it contains an accurate representation of the analytical sequence and sufficient date and time information to verify that the QC time criteria were met).

  Note: The Ward CVT benchsheet option is recommended as an efficient way to generate a benchsheet and run log.

- Data review checklist - See Appendix B

- Standards Documentation (including prep date, source and lot number).

17.5. Non-conformance summary (if applicable).

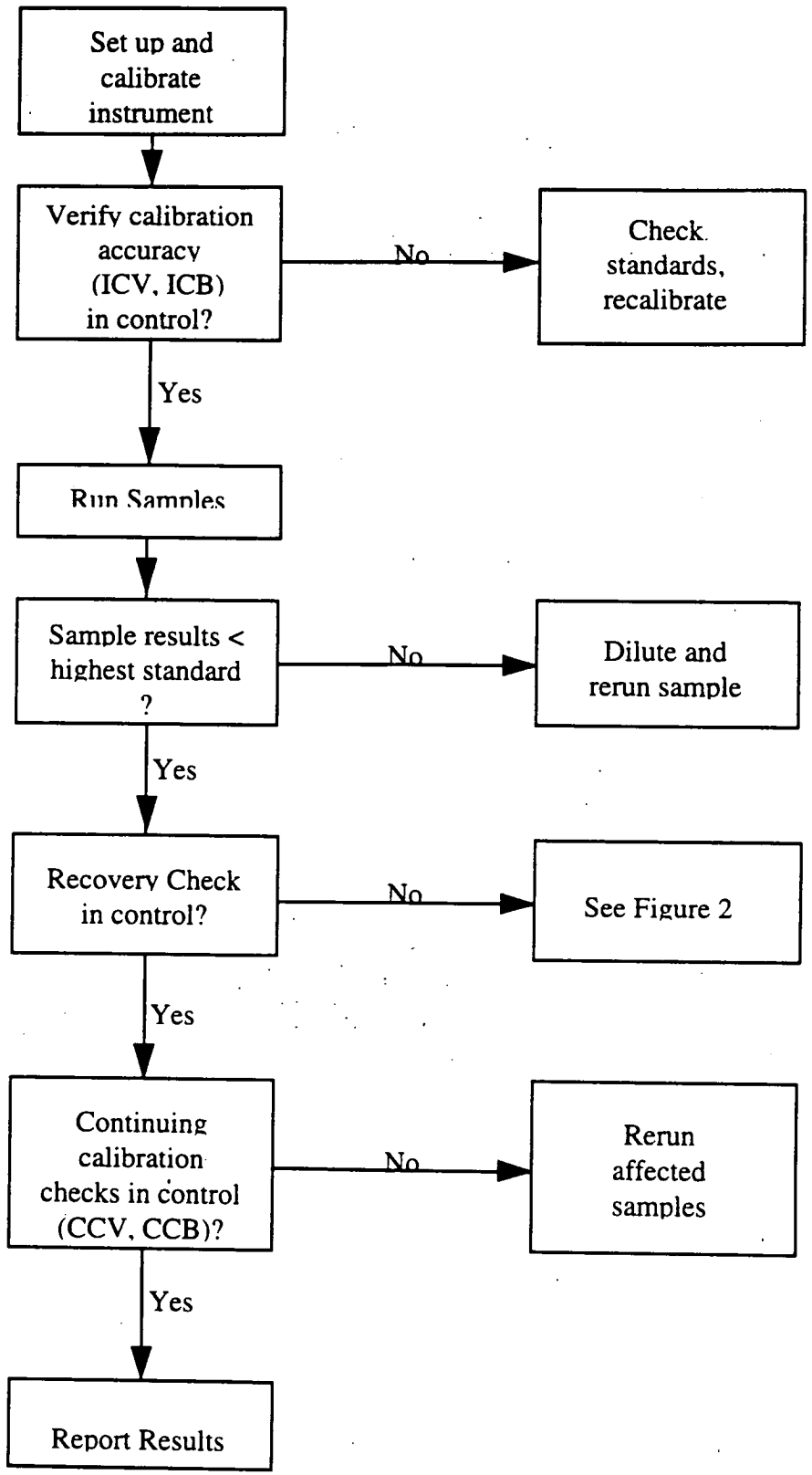

Figure 1. GFAA Analysis Flow Diagram

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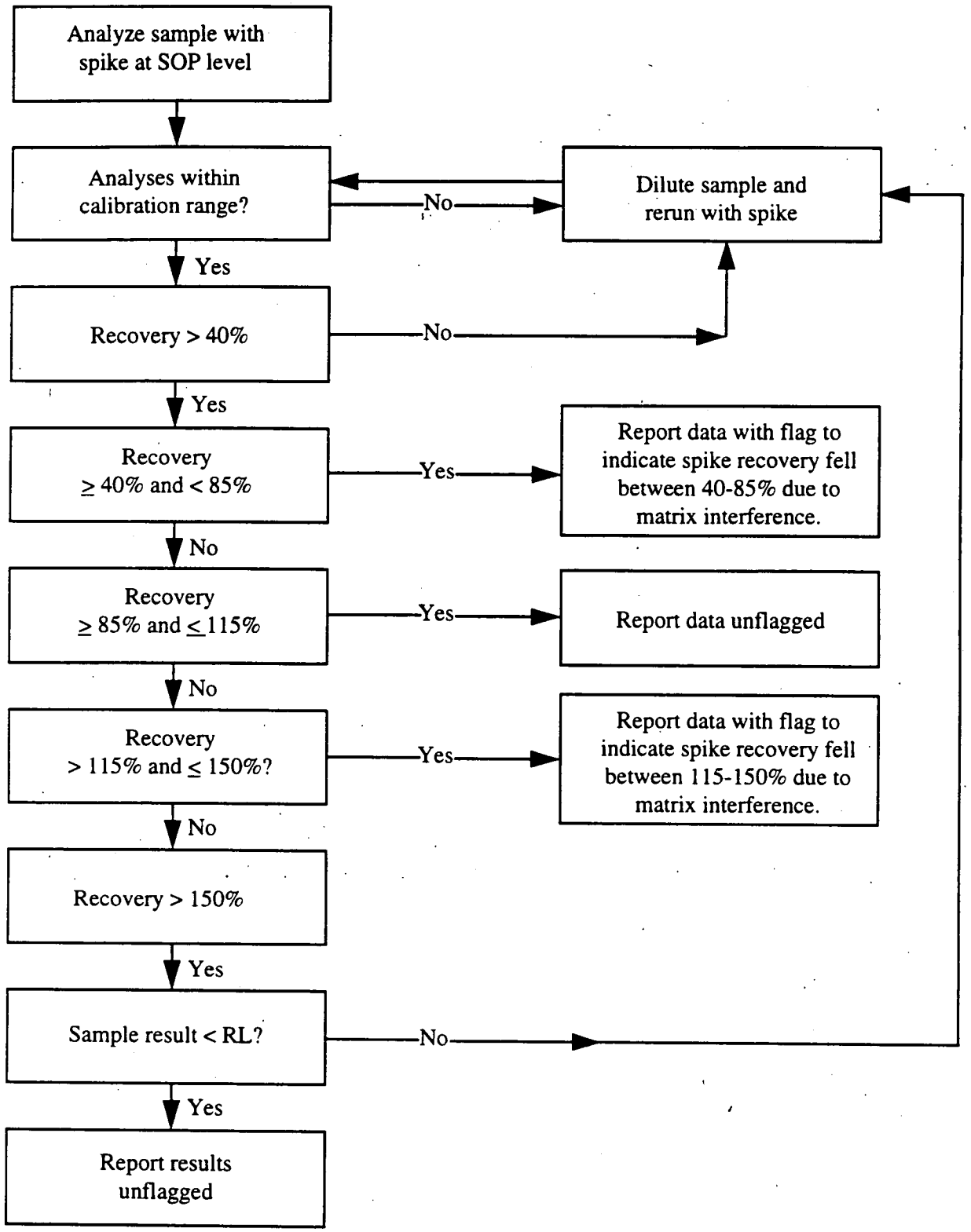

Figure 2. Recovery Check Protocol

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APPENDIX A

TABLES

TABLE I. 7000 and 200 Series Routine Analyte List

ELEMENT	Symbol	CAS #	SW-846 Method	MCAWW Method	Aqueous RL (ug/L)	Solid RL (mg/Kg)
Arsenic	As	7440-38-2	7060A	206.2	10	1.0
Selenium	Se	7782-49-2	7740	270.2	5	0.5
Lead	Pb	7439-92-1	7421	239.2	3	0.3
Thallium	Tl	7440-28-0	7841	279.2	10	1.0
Antimony	Sb	7440-36-0	7041	204.2	10	1.0
Cadmium	Cd	7440-43-9	7131A	213.2	1	0.1
Chromium	Cr	7440-47-3	7191	218.2	5	0.5
Copper	Cu	7440-50-8	7211	220.2	5	0.5
Silver	Ag	7440-22-4	7761	272.2	1	0.1
Nickel	Ni	7440-02-0	NA	249.2	5	0.5

TABLE IA. Non-routine GFAA Analytes *

ELEMENT	Symbol	CAS #	SW846 Method	MCAWW Method
Barium	Ba	7440-39-3	7081	208.2
Beryllium	Be	7440-41-7	7091	210.2
Cobalt	Co	7440-48-4	7201	219.2
Iron	Fe	7439-89-6	7381	236.2
Manganese	Mn	7439-96-5	7461	243.2
Molybdenum	Mo	7439-98-7	7481	246.2
Vanadium	V	7440-62-2	7911	286.2
Zinc **	Zn	7440-66-6	7951	289.2

Note: Elements are not available at all Quanterra Laboratories.

* Additional elements may be possible by this method. ICP is recommended for all elements listed in Table IA.

**Not recommended by this procedure due to background contamination levels.

NA - Not applicable.

TABLE II. Recommended GFAA Analytical Guidelines

Element	λ (nm)	Char Temp°C	Atom Temp°C	Tube
Arsenic	193.7	1200-1400	2000-2800	Py/Pl
Selenium	196.0	900-1400	1900-2700	Py/Pl
Lead	283.3	700-1100	1500-2300	Py/Pl
Thallium	276.8	400-1100	1400-2600	Py/Pl
Antimony	217.6	400-1400	1900-2800	Py/Pl
Cadmium	228.8	500-1200	1400-2300	Py/Pl
Chromium	357.9	1100-1650	2300-2800	Py/Pl
Copper	324.8	800-1300	1900-2500	Py/Pl
Silver	328.1	500-1000	1600-2600	Py/Pl
Nickel	232.0	900-1300	2100-2500	Py/Pl

*Py/Pl refers to a pyrolytically coated platform tube.

TABLE III . Matrix Spike, Analytical Spike and Laboratory Control Sample Levels (ug/L)

ELEMENT	LCS/ Matrix Spike	Analytical Spike
Arsenic	40	20
Selenium	40	20*
Lead	40	20
Thallium	40	20
Antimony	40	20
Cadmium	4	4
Chromium	10	10
Copper	20	10
Silver	5	5
Nickel	20	20

*If running in combined protocol mode (Section 11.15), a spike level of 10ppb is used to accommodate CLP criteria.

TABLE IV. TCLP Reporting Limits, Regulatory Limits and Matrix Spike Levels

ELEMENT	RL (ug/L)	Regulatory Limit (ug/L)	Spike Level (ug/L)
Arsenic	500	5000	5000
Barium	10000	100000	50000
Cadmium	100	1000	1000
Chromium	500	5000	5000
Lead	500	5000	5000
Selenium	250	1000	1000
Silver	500	5000	1000

TABLE V. Calibration and Calibration Verification Standards

ELEMENT	Std 1	Std 2	Std 3	ICV	CCV
Arsenic	10	40	80	30	40
Selenium	5	40	80	30	40
Lead	3	40	80	30	40
Thallium	10	30	60	20	30
Antimony	10	25	50	20	25
Cadmium	1	2.5	5	2	2.5
Chromium	5	15	25	10	15
Copper	5	20	40	10	20
Silver	1	5	10	4	5
Nickel	5	50	100	40	50

***SOP specified calibration levels must be used unless prevented by the instrument configuration or client specific requirements. Deviations from specified calibration levels must be documented in the facility specific instrument operation SOP and must be approved by the facility technical manager and Quality Assurance Manager.**

TABLE VI. Summary Of Quality Control Requirements

QC PARAMETER	FREQUENCY *	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
ICV	Beginning of every analytical run.	90 - 110 % recovery	Terminate analysis; Correct the problem; Recalibrate.
ICB	Beginning of every analytical run, immediately following the ICV.	The result must be within +/- RL from zero.	Terminate analysis; Correct the problem; Recalibrate.
CCV	Every 10 samples and at the end of the run.	Method 7000A: 80 - 120 % recovery. 200 series: 90 -110 % recovery.	Terminate analysis; Correct the problem; Recalibrate and rerun all samples not bracketed by acceptable CCV.
CCB	Immediately following each CCV.	The result must be within +/- RL from zero.	Terminate analysis; Correct the problem; Recalibrate and rerun all samples not bracketed by acceptable CCB.
Method Blank	One per sample preparation batch of up to 20 samples.	The result must be less than or equal to the RL. Common lab contaminants may be accepted up to 2x the RL after consultation with the client (See Section 9.2) Sample results greater than 20x the blank concentration are acceptable. Samples for which the contaminant is < RL do not require redigestion (See Section 9.2).	Redigest and reanalyze samples. Note exceptions under criteria section. See Section 9.2 for additional requirements.

*See sections 11.14 and 11.15 for exact run sequence to be followed.

TABLE VI. Summary of Quality Control Requirements (Continued)

QC PARAMETER	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Laboratory Control Sample (LCS)	One per sample preparation batch of up to 20 samples.	Aqueous LCS must be within 80 - 120% recovery or in-house control limits. Samples for which the contaminant is < RL and the LCS results are > 120% may not require redigestion or reanalysis (see Section 9.3)	Terminate analysis; Correct the problem; Redigest and reanalyze all samples associated with the LCS.
Matrix Spike	One per sample preparation batch of up to 20 samples.	80 - 120 % recovery or in-house control limits. If the MS/MSD is out for an analyte, it must be in control in the LCS.	In the absence of client specific requirements, flag the data; no flag required if the sample level is > 4x the spike added. For TCLP see Section 11.16
Matrix Spike Duplicate	See Matrix Spike	80 - 120 % recovery or in-house control limits; RPD ≤ 20%. (See MS)	See Corrective Action for Matrix Spike.
Analytical Spike	One per each sample analyzed including method blank.	85 - 115 % recovery.	See Section 9.7 (Spike Recovery Check).

APPENDIX B
QUANTERRA GFAA DATA REVIEW CHECKLIST

Quanterra GFAA Data Review Checklist

Run/Project Information

Run Date: _____ **Analyst:** _____ **Instrument:** _____
Prep Batches Run: _____

Circle Methods used: **7000A / 2XX.X : CORP-MT-0003 Rev 1**
CLP : CORP-MT-0004 Rev 1

Review Items

A. Calibration/Instrument Run QC	Yes	No	N/A	2nd Level
1. Instrument calibrated per manufacturer's instructions and at SOP specified levels ?				
2. ICV/CCV analyzed at appropriate frequency and within control limits?				
3. ICB/CCB analyzed at appropriate frequency and within +/- RL or +/- CRDL (CLP)?				
4. CRA run (CLP only)?				
B. Sample Results				
1. Were samples with concentrations > the high calibration standard diluted and reanalyzed?				
2. RSD of replicate burns < 20 % when sample results > RL ?				
3. All reported results bracketed by in control QC ?				
4. Sample analyses done within holding time?				
C. Preparation/Matrix QC				
1. LCS done per prep batch and within QC limits ?				
2. Method blank done per prep batch and < RL or CRDL (CLP) ?				
3. MS run at required frequency and within limits ?				
4. MSD or DU run at required frequency and RPD within SOP limits?				
5. Analytical spike within QC limits or appropriate action taken ?				
D. Other				
1. Are all nonconformances documented appropriately ?				
2. Current IDL/MDL data on file?				
3. Calculations and Transcriptions checked for error ?				
4. All client/ project specific requirements met?				
5. Date/time of analysis verified as correct ?				

Analyst: _____ **Date:** _____
Comments:
_____
_____

2nd Level Reviewer : _____ **Date:** _____
_____
_____

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APPENDIX C

MSA GUIDANCE

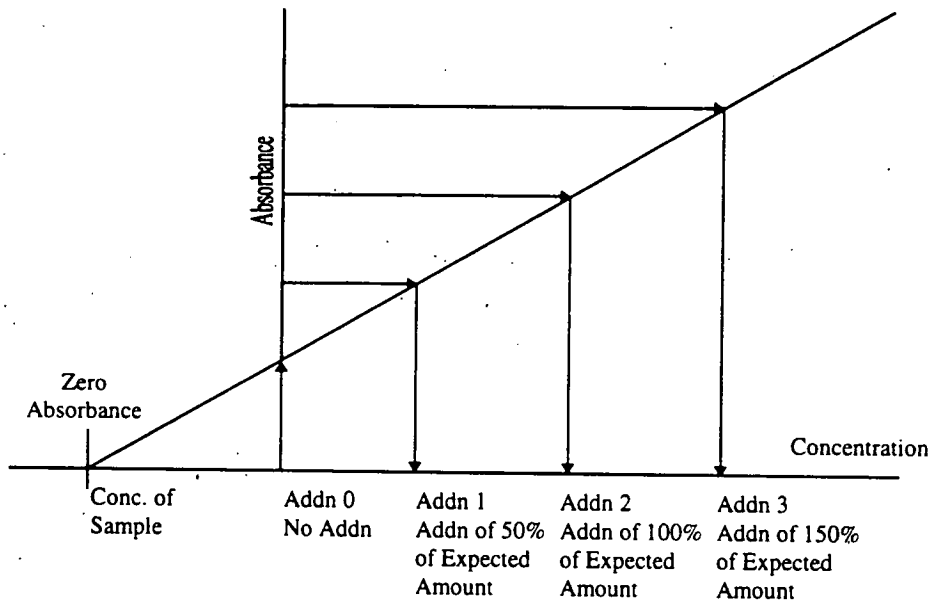
APPENDIX C. MSA GUIDANCE

Method of Standard Addition

Four equal volume aliquots of sample are measured and known amounts of standards are added to three aliquots. The fourth aliquot is the unknown and no standard is added to it. The concentration of standard added to the first aliquot should be 50% of the expected concentration. The concentration of standard added to the second aliquot should be 100% of the expected concentration and the concentration of standard added to the third aliquot should be 150% of the expected concentration. The volume of the unspiked and spiked standard should be the same.

In order to determine the concentration of analyte in the sample, the analytical value of each solution is determined and a plot or linear regression performed. On the vertical axis the analytical value is plotted versus the concentrations of the standards on the horizontal axis. An example plot is shown in Figure 1. When the resulting line is extrapolated back to zero absorbance, the point of interception of the horizontal axis is the concentration of the unknown.

Figure 1



- For the method of standard additions to be correctly applied, the following limitations must be taken into consideration.
- The plot of the sample and standards must be linear over the concentration range of concern. For best results, the slope of the curve should be similar to that of a plot of the aqueous standard curve.
- The effect of the interference should not vary as the ratio of the standard added to the sample matrix changes.

APPENDIX D

TROUBLESHOOTING GUIDE

APPENDIX D. TROUBLESHOOTING GUIDE

Problem	Possible Cause
Poor or No Absorbance or Sensitivity Check failed (See Section 11.4)	Read time too short Incorrect wavelength Low atomization temperature Char temperature too high Dry temperature too high No read step in program Dirty windows Window loose Etched or dirty optics Wrong lamp Bad lamp Bad graphite tube Bad contact rings Not enough or no sample injected Empty sample cup No modifier in sample Incorrectly made standards Poor injection into tube or on platform Flush solution empty Gas flow during read step Gas leak EDL power supply set on "Continuous"
Tube Impedance Error	Bad tube Bad contact rings Sample matrix Door not tight Argon supply out Cooling system malfunction

Problem	Possible Cause
Erratic Readings or High % RSD	Source lamp not aligned properly Lamp not prewarmed Injection tip partially clogged Rinse bottle empty Water cooling not on Bad lamp Injection tip hitting outside of tube Injection tip coated or not set properly Salt buildup on tip Leak in sample capillary Ruby valve clogged Wrong or no modifier Incomplete dry step Dry temperature too high Bad tube Bad contact rings Unstable power supply Air bubbles in syringe or cylinders(Varian)
EDL Won't Light	Lamp cable not plugged in Lamp power set at 0 Lamp is dead Power supply fuse is blown Short in cord Lamp driver faulty (EDL2)
Sample Not Drying or Crackling	Incorrect dry temperature Tube is placed incorrectly Tube is bad Platform not seated properly Furnace door not tight Sample matrix Bad contact rings
Standards reading twice normal absorbance or concentration	Incorrect standard used Incorrect dilution perform
Background Correction Light Blinking	Background screen or attenuator faulty D2 lamp and element lamp energy don't match Analyte lamp energy too low (bad lamp) Background screen or attenuator faulty
Preparation blank spike recovery outside recovery limits	Incorrect concentration of nitric acid HCl added to sample in prep lab Injection tip worn or clogged Sample pump worn Contaminated cup or spike solution

APPENDIX E

CONTAMINATION CONTROL GUIDELINES

APPENDIX E. CONTAMINATION CONTROL GUIDELINES

The following procedures are strongly recommended to prevent contamination:

All work areas used to prepare standards and spikes should be cleaned before and after each use.

All glassware should be washed with detergent and tap water and rinsed with 1:1 nitric acid followed by deionized water.

Proper laboratory housekeeping is essential in the reduction of contamination in the metals laboratory. All work areas must be kept scrupulously clean.

Powdered or Latex Gloves must not be used in the metals laboratory since the powder contains silica and zinc, as well as other metallic analytes. Only vinyl or nitrile gloves should be used in the metals laboratory.

Glassware should be periodically checked for cracks and etches and discarded if found. Etched glassware can cause cross contamination of any metallic analytes.

Autosampler trays should be covered to reduce the possibility of contamination. Trace levels of elements being analyzed in the samples can be easily contaminated by dust particles in the laboratory.

The following are helpful hints in the identification of the source of contaminants:

Yellow pipet tips and volumetric caps can sometimes contain cadmium.

Some sample cups have been found to contain lead.

The markings on glass beakers have been found to contain lead. If acid baths are in use for glassware cleaning, they should be periodically checked for contaminants since contaminant concentrations will increase over time.

The buildup of matrix modifiers in the furnace housing can cause cross contamination. For example: If nickel nitrate is used as a matrix modifier, the furnace housing should be thoroughly cleaned prior to the analysis of nickel.

New glassware, especially beakers, can be a source of silica and boron.

Reagents or standards can contain contaminants or be contaminated with the improper use of a pipette.

Improper cleaning of glassware can cause contamination.

Pyrolytic tubes can contain contaminants. If suspected, the tube should be cleaned by operating the furnace at full power as required to eliminate the contamination.

Latex gloves contain over 500 ppb of zinc.

APPENDIX F

PREVENTIVE MAINTENANCE

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APPENDIX F. PREVENTIVE MAINTENANCE

A maintenance log is used to record when maintenance is performed on instruments. When an instrument problem occurs indicate the date, time and instrument number, then identify the problem and corrective action in the maintenance log.

The following procedures are required to ensure that that the instrument is fully operational.

Daily
- Clean contact rings and windows
- Check tube and platform, replace if corroded, flaking or sensitivity check fails
- Check gas lines and supply level
- Check rinse solution and fill if needed
- Check drain lines and waste containers, empty if needed
- Clean outside of capillary tip
- Adjust autosampler arm as needed
- Debubble syringe (Varian instruments only)
- Clean fume extraction tip, replace fume extraction filter and water trap (PE4100ZL)

Weekly
- Trim capillary tip as needed
- Check water level in coolflow
- Wipe exterior of instrument

Monthly
- Clean filters and radiators

Quarterly
- Change contact rings (if needed)
- Clean instrument water system

Annually
- Notify manufacturer service engineer to clean optics

To obtain the maximum graphite tube life the following guidelines should be followed.

1. Contact rings should be cleaned when replacing the graphite tube.
2. After insertion, every new tube should be thermally conditioned. It is recommended that for the first heating, a ramp time between 60 and 100 seconds and a temperature range from ambient temperature to 2650°C be applied. After a few seconds at 2650°C and a cool down of 20 seconds, the process should be repeated three times with a ramp time 10 seconds.
3. It is very important to long tube life to always use a ramp time for thermal pretreatment. No higher temperature increase than 50°C per second is recommended.

4. For optimal performance, no higher atomization temperatures should be used than 2650°C for pyrolytically coated tubes and 2700°C for uncoated tubes. For refractory elements, application of higher atomization temperature can initially increase the signal, but will drastically reduce the tube lifetime.

5. When using peak area, atomization time has to be sufficiently long for the signal to come back to the baseline. High atomization and heatout temperatures should be applied as long as required, and not a second longer.

6. To avoid a negative effect of acids on tube lifetime, always keep a check on acid concentrations, use a slow ramp for thermal pretreatment and use pyrolytically coated tubes as specified in Table II.

7. If the temperature of the GFAA cooling water is too low and the flowrate is too high, condensation from atmospheric humidity can occur around the contact rings. A cooling water flowrate of 2 L/min +/-0.5 L/min is specified by Perkin Elmer. The use of a coolflow is strongly recommended since it always provides the same water flow and temperature.

8. Nitrogen gas results in a lower peak sensitivity for several elements. It can also cause the generation of cyanogen above 2300°C, making it potentially dangerous in a poorly ventilated room. Perkin Elmer recommends the use of argon.

APPENDIX G

Footnotes and Qualifiers

The following are footnotes that are to be applied to data by the reviewer during data reduction and LIMS entry. Use these qualifiers in the absence of any other client-specific requirements.

Qualifier/Flag	Definition	Conditions for use and how applied
B	This analyte is also detected in the blank at a reportable level.	The flag is applied to each applicable analyte in each sample when: • analytes are detected above the reporting limit in both the blank and the samples or • laboratory contaminants are detected in the blank at a concentration less than 2X the reporting limit. See section 9.2 for specific acceptance procedures.
p	Reporting limit was raised when the sample was diluted to verify a suspected matrix interference.	Apply to the sample if: • the AS recovery is <40%, • the sample is diluted, and reanalyzed with another analytical spike. See section 9.7 of this SOP.
l	The post-digestion spike recovery fell between 115%-150% due to matrix interference.	Apply to the sample if: • the AS recovery is >115% and • the AS recovery is <150% and • the sample result is greater than the reporting limit.

APPENDIX H

FIRST AID PLAN/HYDROFLUORIC ACID

SANTA ANA - SPECIFIC

QUANTERRA STANDARD OPERATING PROCEDURE

TITLE: TOXICITY CHARACTERISTIC LEACHING PROCEDURE

AND SYNTHETIC PRECIPITATION LEACHING PROCEDURE

(SUPERSEDES: NONE)

Prepared by: _____

Reviewed by: _____
Technical Specialist

Approved by: _____
Quality Assurance Manager

Approved by: _____
Environmental Health and Safety Coordinator

Approved by: _____
Laboratory Manager

Approved by: _____
Corporate Technology and/or Corporate QA

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1. SCOPE AND APPLICATION

1.1. This SOP describes the application of the Toxicity Characteristic Leaching Procedure (TCLP), SW846 Method 1311. The Toxicity Characteristic (TC) of a waste material is established by determining the levels of 8 metals and 31 organic chemicals in the aqueous leachate of a waste. The TC is one of four criteria in 40 CFR Part 261 to determine whether a solid waste is classified as a hazardous waste. The other three are corrosivity, reactivity and ignitability. The TC Rule utilizes the TCLP method to generate the leachate under controlled conditions which were designed to simulate leaching through a landfill. EPA's "worst case" waste disposal model assumes mismanaged wastes will be exposed to leaching by the acidic fluids generated in municipal landfills. The EPA's model also assumes the acid/base characteristics of the waste will be dominated by the landfill fluids. The TCLP procedure directs the testing laboratory to use a more acidic leaching fluid if the sample is an alkaline waste, again in keeping with the model's assumption that the acid fluids will dominate leaching chemistry over time.

1.2. The specific list of TC analytes and regulatory limits may be found in Appendix A.

Note: The list in Appendix A does not include the December 1994 EPA rule for Universal Treatment Standards for Land Disposal Restrictions. Those requirements include 216 specific metallic and organic compounds and, in some cases, lower detection limit requirements (see 40 CFR 268.40). TCLP leachates are part of the new Universal Treatment Standards, but the conventional analytical methods will not necessarily meet the new regulatory limits. Consult with the client and with Quanterra Technical Specialists before establishing the instrumental methods for these regulations.

1.3. This SOP also describes the application of the Synthetic Precipitation Leaching Procedure (SPLP) which was designed to simulate the leaching that would occur if a waste was disposed in a landfill and exposed only to percolating rain water. The procedure is based on SW846 Method 1312. The list of analytes for SPLP may extend beyond the toxicity characteristic compounds shown in Appendix A. With the exception of the use of a modified extraction fluid, the SPLP and TCLP protocols are essentially equivalent. Where slight differences may exist between the SPLP and TCLP they are distinguished within this SOP.

1.4. The procedure is applicable to liquid, solid, and multiphase wastes.

1.5. The results obtained are highly dependent on the pH of the extracting solution, the length of time that the sample is exposed to the extracting solution, the temperature

during extraction, and the particle size/surface area of the sample. These parameters must be carefully controlled.

1.6. The reporting limits are based on the individual samples as well as the individual analysis techniques. However, the sample is determined to be hazardous if it contains any analyte at levels greater than or equal to the regulatory limits.

1.7. If a total analysis of the waste demonstrates that individual analytes are not present in the waste, or that they are present but at such low concentrations that the appropriate regulatory levels could not possibly be exceeded, the procedure need not be run. If the total analysis results indicate that TCLP is not required, the decision to cease TCLP analysis should be remanded to the client.

1.8. If an analysis of any one of the liquid fractions of the procedure leachate indicates that a regulated compound is present at such a high concentration that, even after accounting for dilution from the other fractions of the leachate, the concentration would be equal to or above the regulatory level for that compound, then the waste is hazardous and it may not be necessary to analyze the remaining fractions of the leachate. However, the remaining analyses should not be terminated without the approval of the client.

1.9. Volatile organic analysis of the leachate obtained using a bottle extraction, normally used for extractable organics and metals, can be used to demonstrate that a waste is hazardous, but only the ZHE option can be used to demonstrate that the concentration of volatile organic compounds is below regulatory limits.

2. SUMMARY OF METHOD

2.1. For liquid wastes that contain less than 0.5% dry solid material, the waste, after filtration through 0.6 to 0.8 µm glass fiber filter, is defined as the TCLP leachate.

2.2. For wastes containing greater than or equal to 0.5% solids, the liquid, if any, is separated from the solids and stored for later analysis. The particle size of the remaining solid phase is reduced, if necessary. The solid phase is extracted with an amount of extraction fluid equal to 20 times the weight of the solid phase. For TCLP, the extraction fluid employed for extraction of non-volatile analytes is a function of the alkalinity of the solid phase of the waste. For SPLP, the extraction fluid employed is a function of the region of the country where the sample site is located if the sample is a soil. If the sample is a waste or wastewater the extraction fluid employed is a pH 4.2 solution. Two leachates may be generated: a) one for analysis of non-volatile constituents (semi-volatile organics, pesticides, herbicides and metals and/or b) one from a Zero Headspace Extractor (ZHE) for analysis of volatile organic constituents.

Following extraction, the liquid leachate is separated from the solid phase by filtration through a 0.6 to 0.8 µm fiber filter.

2.3. If compatible (i.e., multiple phases will not form on combination), the initial liquid phase of the waste is added to the liquid leachate and these are prepared and analyzed together. If incompatible, the liquids are analyzed separately and the results are mathematically combined to yield a volume-weighted average concentration.

3. DEFINITIONS

3.1. "Leachate" is used to refer to the TCLP solution generated from this procedure.

3.2. "Percent Wet Solids" is that fraction of a waste sample (as a percentage of the total sample) from which no liquid may be forced out by an applied pressure.

4. INTERFERENCES

4.1. Oily wastes may present unusual filtration and drying problems. As recommended by EPA (see Figure 3), oily wastes will be assumed to be 100% liquid and analysis for total concentrations of contaminants will be performed. This applies specifically to samples containing viscous non-aqueous liquids that would be difficult to filter.

4.2. Wastes containing free organic liquids (i.e., those with separable non-aqueous liquid phases) will be assumed to be 100% liquid and totals analysis will be performed to determine if the oil exceeds TCLP limits.

4.3. Solvents, reagents, glassware and other sample processing hardware may yield artifacts and/or interferences to sample analysis. All these materials must be demonstrated to be free from interferences under the conditions of the analysis by analyzing method blanks as described in the Section 9.0 and the individual determinative SOPs.

4.4. Glassware and equipment contamination may result in analyte degradation. Soap residue on glassware and equipment may contribute to this. All glassware and equipment should be rinsed very carefully to avoid this problem.

4.5. Phthalates may be eliminated by proper glassware cleanup and by avoiding plastics. Only glass, Teflon or Type 316 stainless steel tumblers may be used for leachates to be analyzed for organics. Plastic tumblers may be used for leachates to be analyzed for the metals.

4.6. Overexposure of the sample to the environment will result in the loss of volatile components.

4.7. Potential interferences that may be encountered during analysis are discussed in the individual analytical methods.

5. SAFETY

5.1. Procedures shall be carried out in a manner that protects the health and safety of all Quanterra associates.

5.2. Eye protection that satisfies ANSI Z87.1 (as per the Chemical Hygiene Plan), laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.3. The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the Material Safety Data Sheets (MSDS) maintained in the laboratory. The following specific hazards are known:

5.3.1. Chemicals that have been classified as **carcinogens**, or **potential carcinogens**, under OSHA include:

Methylene chloride

5.3.2. Chemicals known to be **flammable** are:

Methanol

5.3.3. The following materials are known to be **corrosive**:

Hydrochloric acid, nitric acid, sulfuric acid, acetic acid, sodium hydroxide

5.3.4. The following materials are known to be **oxidizing agents**:

Nitric Acid.

5.4. Gas pressurized equipment is employed in this procedure. Be sure all valves and gauges are operating properly and that none of the equipment, especially tubing, is

over-pressurized. CAUTION: Do not open equipment that has been pressurized until it has returned to ambient pressure.

5.5. A rotary agitation apparatus is used in this procedure. Certain samples may break the glass jars used in the procedure. For these samples, extra caution, including plastic or polyethylene overwraps of the glass jar, may be necessary.

5.6. Secure tumbler and extraction apparatus before starting rotary agitation apparatus.

5.7. During sample rotation, pressure may build up inside the bottle. Periodic venting of the bottle will relieve pressure.

5.8. Exposure to chemicals must be maintained as low as reasonably achievable, therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.9. The preparation of standards and reagents and glassware cleaning procedures that involve solvents such as methylene chloride will be conducted in a fume hood with the sash closed as far as the operation will permit.

5.10. All work must be stopped in the event of a known or potential compromise to the health and safety of a Quanterra associate. The situation must be reported **immediately** to a laboratory supervisor.

5.11. Due to the potential for ignition and/or flammability, do not attempt to dry non-aqueous liquid samples in an oven.

6. EQUIPMENT AND SUPPLIES

6.1. Extraction vessels

6.1.1. For volatile analytes - zero-headspace extraction (ZHE) vessel, gas-pressure actuated, Millipore YT3009OHW or equivalent (see Figure 2).

6.1.2. For metals - either borosilicate glass jars (1/2 - 1 gallons, with Teflon lid inserts) or 1 L HDPE (Nalgene or equivalent) bottles may be used.

6.1.3. For non-volatile organics - only borosilicate glass may be used.

6.2. Vacuum filtration apparatus, capable of 0 - 50 psi and stainless steel pressure filtration apparatus (142 mm diameter), capable of 0 - 50 psi.

6.3. Borosilicate glass fiber filters, 0.6 - 0.8 µm (Whatman GF/F 14.2 cm, 0.7 µm or equivalent). When analyzing for metals, wash the filters with 1 N nitric acid and de-ionized water prior to use. Glass fiber filters are fragile and should be handled with care.

6.4. Rotary agitation apparatus, multiple-vessel, Associated Design and Manufacturing Company 3740-6 or equivalent (see Figure 1). The apparatus must be capable of rotating the extraction vessel in an end-over-end fashion at 30 ± 2 rpm.

6.5. ZHE Extract Collection Devices are used to collect the initial liquid phase and the final extract of the waste from the ZHE device, either of the following may be used:

6.5.1. Gas-tight syringes, 100 mL capacity, Hamilton 0158330 or equivalent, or

6.5.2. Tedlar bags

6.6. Top loading balance, capable of 0 - 4000 ± 0.01g (all measurements are to be within ± 0.1 grams).

6.7. pH meter and probe capable of reading to the nearest 0.01 unit, and with automatic temperature compensation.

6.8. pH probes.

6.9. Magnetic stirrer/hotplate and stirring bars.

6.10. VOA vials, 20 mL, with caps and septa.

6.11. Glass jars, 1/2 - 1 gallon, with Teflon lid-inserts.

6.12. Nalgene plastic bottles, 1 liter.

6.13. Miscellaneous laboratory glassware and equipment.

7. REAGENTS AND STANDARDS

7.1. Reagent water for non-volatile constituents must be produced by a Millipore DI system or equivalent. For volatile constituents, water must be passed through an activated carbon filter bed (Milli-Q or tap water passed through activated carbon). Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

7.2. Hydrochloric acid, 1 N: Carefully add 83 mL concentrated reagent grade HCl to 800 mL reagent water, cool and dilute to 1 liter with reagent water. Cap and shake to mix well.

7.3. Nitric acid, 1 N: Carefully add 64 mL concentrated reagent grade HNO_3 to 800 mL of reagent water, cool and dilute to 1 liter with reagent water. Cap and shake to mix well.

7.4. Sodium hydroxide, 1 N: Carefully add 40 g reagent grade NaOH pellets to 800 mL reagent water, stir until the pellets are completely dissolved, cool and dilute to 1 liter with reagent water.

CAUTION: Heat is generated during this process.

7.5. Acetic acid, glacial: concentrated, reagent grade liquid (HOAc).

7.6. pH calibration solutions: buffered to a pH of 4, 7, and 10. Commercially available. Fresh buffer solution must be used each day of analysis.

7.7. TCLP Leaching Fluids

7.7.1. General Comments

7.7.1.1. The pH of both solutions listed below should be monitored daily *and/or prior to use* and the pH probes are to be calibrated prior to use.

7.7.1.2. The leaching fluids MUST be prepared correctly. If the desired pH range is not achieved and maintained, the TCLP may yield erroneous results due to improper leaching. If the pH is not within the specifications, the fluid must be discarded and fresh extraction fluid prepared.

7.7.1.3. Additional volumes of extraction fluids listed above may be prepared by multiplying the amounts of acetic acid and NaOH by the number of liters of extraction fluid required.

7.7.2. TCLP Fluid #1: Carefully add 5.7 mL glacial acetic acid and 64.3 mL of 1 N NaOH to 500 mL reagent water in a 1 liter volumetric flask. Dilute to a final volume of 1 L with reagent water, cap and shake to mix well. When correctly prepared, the pH of this solution is 4.93 ± 0.05.

7.7.3. TCLP Fluid #2: Carefully add 5.7 mL glacial acetic acid to 500 mL reagent water in a 1 liter volumetric flask. Dilute to a final volume of 1 L with reagent water, cap and shake to mix well. When correctly prepared, the pH of this solution is 2.88 ± 0.05.

7.8. Nitric acid, 50% solution: Slowly and carefully add 500 mL concentrated HNO_3 to 500 mL reagent water. Cap and shake to mix well.

7.9. Sulfuric acid / nitric acid (60/40 weight percent mixture) H_2SO_4/HNO_3. Cautiously mix 60 g of concentrated sulfuric acid with 40 g of concentrated nitric acid.

7.10. SPLP Leaching fluids

7.10.1. SPLP solutions are unbuffered and exact pH may not be attained. The pH of TCLP and SPLP fluids should be checked prior to use. If not within specifications, the fluid should be discarded and fresh fluid prepared.

7.10.2. SPLP fluid #1: Add 60/40 weight percent mixture of sulfuric and nitric acids to reagent water until the pH is 4.20 ± 0.05 This fluid is used for soils from a site that is east of the Mississippi River and for wastes and wastewaters.

7.10.3. SPLP fluid #2: Add 60/40 weight percent mixture of sulfuric and nitric acids to reagent water until the pH is 5.00 ± 0.05. This fluid is used for soils from a site that is west of the Mississippi River.

7.10.4. SPLP fluid #3: This fluid is reagent water and is used for leaching of volatiles. Additionally, any cyanide-containing waste or soil is leached with fluid #3 because leaching of cyanide containing samples under acidic conditions may result in the formation of hydrogen cyanide gas.

7.11. Methanol and methylene chloride - used to aid in cleaning oil contaminated equipment.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Samples being analyzed for non-volatile organic compounds should be collected and stored in glass containers with Teflon lid liners. Chemical preservatives shall NOT be added UNTIL AFTER leachate generation.

8.2. Samples being analyzed for metals only can be collected in either glass or polyethylene containers.

8.3. When the waste is to be evaluated for volatile analytes, care should be taken to minimize the loss of volatiles. Samples shall be collected and stored in a manner intended to prevent the loss of volatile analytes (e.g., samples should be collected in Teflon lined septum capped vials with minimal headspace and stored at 4 ± 2 °C). Samples should be opened only immediately prior to extraction.

8.4. Samples should be refrigerated to 4 ± 2 °C unless refrigeration results in irreversible physical changes to the waste. If precipitation occurs, the entire sample (including precipitate) should be extracted.

8.5. The minimum TCLP sample collection size is determined by the physical state or states of the waste and the analytes of concern. The amount of waste required varies with the percent solids. The lower the percent solids, the more waste will be required for preliminary and final testing. For aqueous samples containing between 0.5 and 10% solids, several kilograms of sample are required to complete the analyses. The general minimal requirements when the samples are 100% solids include: 1 - 32 oz jar for semi-volatile organic analysis and metals, and 1 - 4 oz jar for volatile organic analysis. Low density sample materials, such as rags or vegetation, will require larger volumes of sample. For liquid samples (less than *0.5%* solids), minimum requirements are 2 - 32 oz jars for semi-volatile organic analysis and metals, and 2 - 8 oz jars for volatile organic analysis. If volatile organic analysis is the only requested parameter, 2 separate jars are required. If matrix spike or duplicate control samples are requested, additional sample volume is required. If sufficient sample volumes were not received, analyses cannot be started and the client should be notified as soon as possible.

8.6. TCLP leachates should be prepared for analysis and analyzed as soon as possible following extraction. Leachates or portions of leachates for metallic analyte determinations must be acidified with nitric acid to a pH less than 2, unless precipitation occurs. If precipitation occurs upon addition of nitric acid to a small aliquot of the leachate, then the remaining portion of the leachate shall not be acidified and the leachate shall be analyzed as soon as possible. All other leachates should be stored under refrigeration (4 ± 2 °C) until analyzed. ZHE leachates must be stored in VOA vials filled to eliminate all headspace.

8.7. Samples are subject to appropriate treatment within the following time periods:

Table 1 - Holding Times (days)

Parameter	Collection to TCLP Leach	TCLP Leach to Preparation	Preparation to Analysis	Total Elapsed Time
Volatiles:	14	N/A	14	28
Semi-volatiles:	14	7	40	61
Mercury:	28	N/A	28	56
Other Metals:	180	N/A	180	360

NOTE: The initial holding time is measured from date of collection to date extraction started, and subsequent holding times are measured from the date extraction starts. If sample holding times are exceeded, the values obtained will be considered minimal concentrations. Exceeding holding times is not acceptable in establishing that a waste does not exceed the regulatory level. Exceeding the holding time will not invalidate characterization if the waste exceeds the regulatory limit.

9. QUALITY CONTROL

9.1. Quality Control Batch (QC Batch) - QA-003 defines a QC Batch as a set of up to 20 field samples of similar matrix that behave similarly and are processed using the same procedures, reagents and standards within the same time period. The same lot of reagents must be used within a batch. A minimum of one TCLP extraction blank (Method Blank), one Laboratory Control Sample (LCS), one Matrix Spike (MS), and one Matrix Spike Duplicate (MSD) will be prepared with each TCLP leachate batch.

9.2. Batching Samples - Groups of samples with visibly different bulk matrices (e.g., petroleum sludge and soil samples) must be batched separately for QC testing purposes.

9.3. TCLP Extraction Blanks - A minimum of one blank (using the same extraction fluid as used for the samples) must be prepared and analyzed for every batch of samples extracted in a particular vessel type. The blanks are generated in the same way as the samples (i.e:, blanks will be tumbled and filtered with the samples). Extraction vessels will be uniquely numbered. Each time a new batch is set up the blank should be rotated sequentially to the next vessel to ensure all vessels are periodically checked. Consult the Quanterra QC Program and the individual analysis SOPs for blank acceptance criteria.

9.4. Laboratory Control Sample (LCS) - A LCS is required with each batch of 20 or fewer samples. The LCS shall be generated after a batch of TCLP leachates have been generated (i.e., at the time of the preparative digestion or extraction) by spiking an

aliquot of the appropriate extraction fluid used for that batch. Consult the individual analysis SOPs for additional LCS guidance (i.e., spike amounts, spike levels, recovery criteria, etc.).

9.5. Matrix Spike (MS/MSD) - Matrix spikes are used to monitor the performance of the analytical methods on the matrix and to assess the presence of interferences. A MS/MSD pair are required with each batch of 20 or fewer samples.

9.5.1. Matrix spikes are to be added after filtration of the TCLP leachate. Spikes are not to be added prior to the TCLP leaching. For metals, matrix spikes are to be added before preservation with nitric acid.

9.5.2. The use of internal calibration or alternate methods may be needed when the recovery of the matrix spike is below the expected performance (see Section 9.6.2).

9.5.3. Consult the individual analysis SOPs for additional guidance on spike compounds and levels.

9.6. Corrective Actions

9.6.1. Consult the Quanterra QC Program and individual analysis SOPs for corrective action for blanks and LCS

9.6.2. Method of Standard Additions (MSA) shall be used for metals if all of the following conditions are met:

- Recovery of the analyte in matrix spike is not at least 50%,
- the concentration of the analyte does not exceed the regulatory level, and
- the concentration of the analyte measured in the sample is within 20% of the appropriate regulatory level.

If the matrix spike recovery is 5% or less due to dilution or matrix interference, contact the project manager and client for guidance. The client should also be contacted prior to initiation of any MSA steps. Refer to the individual analysis SOPs for details on how to perform MSA analysis.

10. CALIBRATION AND STANDARDIZATION

10.1. Refer to appropriate analysis SOPs.

11. PROCEDURE

11.1. GENERAL COMMENTS

11.1.1. One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented on a Nonconformance Memo kept in the project file and described in the final report. The variation must be approved by a project manager, Technical Specialist and QA Manager. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

11.2. PRELIMINARY SAMPLE EVALUATIONS (Refer to Flow Chart #1, Appendix D)

11.2.1. Preliminary TCLP evaluations (percent solids, particle size, selection of extraction fluid, and fluid/leachate compatibility) are required to be done using a minimum of a 100 gram aliquot of waste. This aliquot may also undergo the actual TCLP or SPLP extraction for Non-volatiles ONLY IF it has NOT been oven dried. If the solid portion is oven dried, a separate aliquot must be used for the actual leaching procedure.

11.2.2. Consult the holding times for the appropriate tests (Section 8.7) and prioritize extractions such that holding times are not exceeded.

11.2.3. Determine the total volume of TCLP leachate (solid phase leachate + liquid filtrate) that needs to be generated for analysis according to the following:

Table 2. Minimum Required Leachate Volume

Analysis	Required Volume (mL)
Volatiles	3 x 20
Semi-volatiles	400
Pesticides	200
Herbicides	200
Metals	300

11.2.3.1. SPLP - similar volumes are *required like TCLP* volatiles and metals.

11.2.3.2. For TCLP and SPLP samples used for matrix spike and matrix spike duplicate analysis, three times the listed volumes are required.

11.2.4. **Sample Description** (enter data on Worksheet 1)

11.2.4.1. Record the number of phases observed in the sample. It is common that when more than one container of multi-phasic materials is received from the field, each container will show different amounts of each phase.

11.2.4.1.1. If the sample has multiple phases and is received in more than 1 bottle then the contents of each bottle should be combined in a single larger container prior to processing the sample further. If this is not possible, then the alternate procedure described in the following section should be used.

11.2.4.1.2. Properly record the relative amounts of each phase by measuring the depth of the layers in each container after the contents have been allowed to settle. Determine the combined volume of each phase for all containers. Then mark the phase composition on a single container, mix thoroughly to obtain a representative subsample, and accurately measure the phase composition according to the following procedure. The two sets of values (combined volumes per phase and phase composition for one container) are used to determine the correct volume/mass adjustments on the final result. This procedure is not appropriate if testing will be done for volatile organic compounds.

11.2.4.2. Solid - record the visible presence of a solid material heavier than water. If the sample contains more than one solid phase (e.g., wood and sediment mixed with water), *document comment on benchsheet.*

11.2.4.3. Liquid - record the number of liquid phases observed in the sample according to apparent density. It may be impossible to distinguish apparent density if only one liquid phase is observed and there is no indication on the COC form. If this is the case,

record it as aqueous material and let the subsequent analytical record show if the liquid is organic.

11.2.5. **Percent Solid Phase** (enter data on Worksheet No. 1)

11.2.5.1. Percent Solids and ZHE Extractions - The ZHE filtration apparatus cannot accurately determine percent solids less than 5%. If an extraction is to be performed solely for volatile organic compounds and the percent solids concentration is apparently greater than 5%, proceed to Section 11.4 (Procedure: ZHE Extraction Procedure, Volatile Constituents). Otherwise, continue with the steps in this section. The aliquot of sample used here cannot be used again for the ZHE extraction.

11.2.5.2. Determine Type of Filtration Apparatus Needed

11.2.5.2.1. If the waste will obviously yield no free liquid when subjected to pressure filtration (i.e., it is 100% solid), then proceed to Section 11.2.6 (Particle-size Reduction).

11.2.5.2.2. If the sample is mostly a non-viscous liquid (water or non-viscous organic liquid) of low solids content (<10%) or a highly granular, liquid containing waste vacuum filtration may be used.

11.2.5.2.3. If the sample is viscous (sludge or has high solids content), use pressure filtration.

11.2.5.3. Weight of subsample and filtrate for percent solids measurement

11.2.5.3.1. Assemble the filtration apparatus (use blunt forceps to handle the 0.6 to 0.8 µm filter membrane).

11.2.5.3.2. Homogenize the waste, transfer a minimum of a 100 g subsample to the weighing vessel. Measure and record the gross weight.

11.2.5.3.3. Measure and record the tare weight of the filtration vessel.

11.2.5.3.4. Transfer the sample to the filtration device attempting to spread the waste sample evenly over the surface of the filter. Measure and record the tare weight of the empty weighing vessel and any residual sample.

11.2.5.3.5. Calculate and record the net weight of sample used for testing.

11.2.5.4. Filtration for percent solids

11.2.5.4.1. Slowly apply gentle pressure or vacuum of 10 psi to the filtration apparatus. Allow the sample to filter until no SIGNIFICANT additional liquid has passed through the filter during a 2 minute period.

11.2.5.4.2. Repeat previous step by increasing the pressure in 10 psi increments until a maximum of 50 psi is reached. Stop the filtration when no additional filtrate is generated within a 2 minute period.

Note: Some samples will contain liquid material that does not filter (e.g., oil). Do not attempt to filter the sample again by exchanging filters. Viscous oils or any wastes which does not pass through the filter is classified as a solid.

11.2.5.4.3. Remove the filtrate collection vessel, weigh and record the gross weight.

11.2.5.4.4. Calculate and record the net weight of filtrate This result will be used in the percent solids calculation.

11.2.5.4.5. Pour the filtrate into a graduated cylinder. Measure and record the volume of the aqueous phase. Measure and record the volume of any organic phase. If more than one organic phase is observed, enter "see below" and provide a description at the bottom of *benchsheet.* These results will be used in the final sample calculations.

11.2.5.4.6. Retain the filtrate for use in Section 11.2.8 (Determination of Filtrate/Extraction Fluid

Compatibility), and for possible recombination with the filtrate obtained in Section 11.3.

11.2.5.5. Percent of Wet Solids

11.2.5.5.1. Calculate the total weight of wet solids using Equation: $E = D\text{-}B$.

11.2.5.5.2. Calculate the weight percent of wet solids using Equation: $\% E = \frac{E}{C} x\ 100$.

11.2.5.5.3. If the percent wet solids result is ≥ 0.5% and < 5.0%, and it is noticed that a small amount of the aqueous filtrate is entrained in the wetting of the filter, proceed to Section 11.2.5.7 to complete the percent solids measurement on a dry-weight basis.

Note: If obviously oily (non-aqueous) material is entrained on the filter, do not dry the filter; proceed to Section 11.2.6 (Particle-Size Reduction).

11.2.5.5.4. If the percent wet solids result is greater than 5.0%, proceed to Section 11.2.6 (Particle-Size Reduction) and mark "≥0.5%" on *bench sheet.*

11.2.5.5.5. If the percent wet solids result is less than 0.5%, discard the solid phase. No leaching will be necessary; the filtrate is equivalent to the final leachate.

11.2.5.6. Weight percent of dry solids (skip this step for oily samples).

Note: These steps are required only if it is noticed that a small amount of the filtrate is entrained in wetting of the filter <u>and</u> the percent wet solids content is ≥ 0.5% and < 5.0 %.

11.2.5.6.1. Remove the filter with the wet solids from the filtration apparatus.

11.2.5.6.2. Dry the filter and solid phase at 100 ± 20 ° C.

11.2.5.6.3. Remove the filter from the oven and allow to cool in a desiccator.

11.2.5.6.4. Weigh and record the gross dry weight.

11.2.5.6.5. Repeat the drying step. Weigh and record the second gross dry weight. If the two weightings do not agree within 1%, perform additional drying and weighing until successive weighings agree within 1%.

11.2.5.6.6. Calculate the weight percent of dry solids using:

$$\textit{Weight Percent Dry Solids} = \frac{\textit{Gross Dry Weight}}{\textit{Weight of Sample}} \times 100$$

11.2.5.6.7. If the dry solids result is ≥ 0.5% and the sample will be extracted for non-volatile constituents, proceed to Section 11.2.6 (Particle Size Reduction) using a fresh wet portion of waste.

11.2.5.6.8. If the percent solids result is less than 0.5%, discard the solid phase. No leaching will be necessary; the filtrate is the TCLP leachate. Proceed to Section 11.2.8 (Determination of Filtrate/Leachate Compatibility) to determine whether or not the material is a non-aqueous, immiscible liquid.

11.2.6. **Particle-size Reduction for Fluid Selection** (enter data on Worksheet 2)

11.2.6.1. The subsample used for fluid selection must consist of particles less than 1 mm in diameter (versus the less than 1 cm requirement for the material used for the actual extraction). The method requires a smaller particle size to partially compensate for the shorter duration of contact time with the leachate solution as compared to the full extraction. Inappropriate use of coarser materials could result in the selection of the wrong fluid type.

11.2.6.2. Surface area exclusion - size reduction is not required if the sample surface area is greater than or equal to 3.1 cm^2 per gram.

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11.2.6.3. If the sample contains particles greater than 1 mm in diameter, crush, cut, or grind the solids to the required size.

11.2.6.4. Consult your supervisor or manager when dealing with unusual sample matrices (e.g., wood, cloth, metal, brick).

11.2.7. **Determination of Appropriate Extraction Fluid** (Worksheet 2)

11.2.7.1. If the solid content is greater than or equal to 0.5%, and if the sample is being analyzed for metals or nonvolatile organic compounds, the type of leaching solution must be determined.

11.2.7.2. Follow times, temperature, and particle size specified in this section as closely as possible. If reaction time between the acid solution and solid waste is too short or too long, the procedure may produce false pH readings.

11.2.7.3. For SPLP, refer to Section 7.10 for fluid selection. Matrix type must be specified by the client. Check special instructions or see the project manager, then put a check mark by the fluid type selected.

11.2.7.4. The TCLP leaching fluid for all volatiles is Fluid #1.

11.2.7.5. For TCLP leach fluid determination for non-volatile analytes, continue with the following steps.

11.2.7.6. Calibrate the pH meter with fresh buffer solution in accordance with the pH SOP.

11.2.7.7. Weigh out a 5.0 ± 0.1 g subsample (less than 1 mm particle size) of the solid phase into a 250-mL beaker. If 5.0 grams not used, enter the actual weight on line C2.

11.2.7.8. Add 96.5 ± 1.0 mL of reagent water, cover with a watchglass, and stir for 5 minutes on a stirrer. If a different volume used, enter the actual volume.

11.2.7.9. Measure and record the sample pH.

Note: To avoid damaging the pH probe when organic liquid is present, use narrow range pH indicator paper.

11.2.7.10. If the pH is less than or equal to 5.0, use Fluid #1 and proceed to Section 11.2.8 (Fluid Compatibility).

11.2.7.11. If the fluid pH is greater than 5.0, add 3.5 mL 1 N HCl, cover with a watchglass. Slurry the sample briefly then heat at 50 °C for 10 minutes.

Note: The heating cycle is a critical step. If the solid waste does not remain in contact with the acidic solution under specified time and temperature conditions, an erroneous pH may be measured.

11.2.7.12. Cool to room temperature.

11.2.7.13. Measure and record the pH immediately after the sample has reached room temperature.

11.2.7.13.1. If the pH is less than or equal to 5.0, use Fluid #1.

11.2.7.13.2. If the pH is greater than 5.0, use Fluid #2.

11.2.8. **Determination of Filtrate/Extraction Fluid Compatibility** (skip this step for SPLP extractions)

11.2.8.1. Place 5 mL of the appropriate leaching fluid (determined in the previous step) into a 20-25 mL vial.

Note: Use fluid type # 1 if simply testing the filtrate for a sample with less than 0.5% solids.

11.2.8.2. Add 5 mL of the initial filtrate, cap and shake.

11.2.8.3. If the phases are miscible, the initial filtrate and solid phase leachate will be physically recombined upon completion of the leachate generation.

11.2.8.4. If the phases are NOT miscible, document on bench sheet. The initial filtrate and the solid phase leachate will be prepared and analyzed separately and the results mathematically combined (see Section 12.1.4).

11.2.9. For samples requiring analysis for semi-volatile organics, pesticides, herbicides or metals proceed to Section 11.3.

11.2.10. For samples requiring analysis for volatile organics (ZHE), proceed to Section 11.4.

11.3. **BOTTLE EXTRACTION PROCEDURE: NON-VOLATILE CONSTITUENTS: SEMI-VOLATILES, PESTICIDES, HERBICIDES, METALS** (Refer to Flow Chart #2, Appendix D)

11.3.1. All masses should be recorded to the nearest 0.1 g.

11.3.2. The aliquot used in the Preliminary Evaluation MAY be used for this procedure ONLY if it was not oven dried. If the sample is 100% solid or if the preliminary aliquot was not oven dried proceed directly to Section 11.3.7 (Particle Size Reduction). If the Preliminary Evaluation aliquot was oven dried then, using a fresh aliquot of sample, continue as described in Sections 11.3.3 through 11.3.6.

11.3.3. Examine the sample and determine the type of filtration to employ per Section 11.2.5.2.

11.3.4. Repeat the steps outlined in Sections 11.2.5.3 through 11.2.5.5.3.

11.3.5. Determine and record the volume (mass) of the initial filtrate. Cover with aluminum foil and retain for use as defined in Section 11.3.18.

11.3.6. Determine and record the "solid" phase mass by subtracting the mass of the liquid filtrate from the mass of the subsample.

11.3.7. Evaluate the solid portion of the waste for particle size. If it contains particles greater than 1 cm in size, prepare the solid portion of the waste for leaching by crushing, cutting, or grinding such that all particles are less than 1 cm in size (i.e., capable of passing through a 9.5 mm, 0.375 inch, standard sieve). Size reduction is not required if the sample surface area is greater than or equal to 3.1 cm^2 per gram. If particle size reduction was required, record this on Worksheet 3, line E1.

11.3.7.1. Consult your supervisor or manager when dealing with unusual sample matrices (e.g., wood, cloth, metal, brick). Scissors or shears may be used to cut cloth, plastic or sheet metal. Saws may be used for wood or solid metal. Bricks, rocks, or other solids amenable to grinding should be subcontracted out for particle size reduction (Contact PA or PM). Note that size reduction to

fine powder is not appropriate, and could invalidate results. If necessary, consult client for guidance.

11.3.8. Determine the minimum total volume of solid phase leachate that needs to be generated. Refer to Section 11.2.3.

11.3.9. Divide the total volume of solid phase leachate required by 20 to determine the mass of solid phase required for leaching. Round this mass UP to the nearest 5 g.

11.3.10. Weigh the required mass of solid phase into an appropriate bottle (plastic for metals only, glass for all others) and **slowly** add 20 times its mass of appropriate leaching fluid as determined under Section 11.2.7 (e.g., 20 g of sample would require 400 g of leaching fluid). Record the weight of the sample aliquoted for the extraction and the amount of extraction fluid added.

11.3.11. Ensure any effervescence has stopped before capping the bottle tightly. Secure in a rotary agitator and rotate end-over-end at 28-32 rpm for 16-20 hours. The temperature of the room should be 23 ± 2°C. The room temperature and time should be checked at both the start and end of the extraction and recorded.

NOTE: As agitation continues, pressure may build up within the bottle for some types of wastes. To relieve excessive pressure, the bottle may be removed and opened periodically in a properly vented hood to relieve any built-up pressure.

11.3.12. Remove the bottle and filter the sample using vacuum or pressure filtration by filtering through a new glass fiber filter as discussed in Sections 11.2.5.5.1 - 11.2.5.5.2. For final filtration of the TCLP leachate, the glass fiber filter may be changed, if necessary, to facilitate filtration. Filters must be acid washed if metals are to be determined (see Section 6.3). The entire sample need not be filtered; however, sufficient volume should be generated to support the required analyses. Record the date and time the filtration is completed.

11.3.13. If the waste contained no initial filtrate, this solution from 11.3.12 is defined as the TCLP leachate.

11.3.14. If the waste did yield an initial filtrate, consult the worksheet for initial filtrate/leachate compatibility. If they are compatible, they are to be

combined in the correct proportions (see Section 12.1.4) and mixed well. This combined solution is defined as the TCLP leachate.

11.3.15. If the individual phases are NOT compatible, they are to be prepared and analyzed separately and the results combined mathematically. See Section 12.1.5.

11.3.16. Measure and record the pH of the TCLP leachate. (Do not attempt to measure the pH of oily samples as the probe may be rendered inoperable.)

11.3.17. Prepare subsamples for metals for MS/MSD quality control testing using the appropriate TCLP spiking solution (do not spike for organics). Refer to the appropriate determinative SOPs for further guidance on the spike components, levels and action criteria.

11.3.18. Immediately preserve the leachate as follows:

Metals	pH < 2 w/50% HNO_3 for non-oils (do not acidify oils)
All others	Refrigerate to 4 ± 2 °C

Note: Refer to Section 8.6 if precipitation occurs upon preservation.

11.3.19. Label each sample with the appropriate information and submit to the appropriate analytical groups for prep and analysis with copies of the TCLP preparation worksheets.

11.4. **ZHE EXTRACTION PROCEDURE: VOLATILE CONSTITUENTS** (Refer to Flow Chart #3, Appendix D)

11.4.1. Use the ZHE device to obtain a TCLP leachate for analysis of volatile compounds only. Leachate resulting from the use of the ZHE shall NOT be used to evaluate the mobility of non-volatile analytes (e.g., metals, pesticides, etc...).

11.4.2. Due to some shortcomings of the method, losses of volatile compounds may occur. Extra care should be observed during the ZHE procedure to ensure that such losses are minimized. Charge the ZHE with sample only once and do not open the device until the final extract has been collected. Do not allow the waste, the initial liquid phase or the extract to be exposed to the atmosphere any longer than necessary.

11.4.3. If the TCLP extraction is for volatile components only, refer to Section 11.2.5.1 before proceeding.

11.4.4. All masses should be recorded to the nearest 0.1 g.

11.4.5. Assemble the ZHE apparatus. Test for leakage by closing all valves except the gas inlet/outlet valve and pressurizing to 50 psi. Allow to stand for 15 minutes and check the pressure on the built-in gauge to make sure it is not leaking. If the pressure is NOT 50 psi, consult your supervisor.

11.4.6. Adjust the ZHE piston in the ZHE body to the appropriate height (slightly moisten the O-rings with leaching fluid if necessary).

11.4.7. Consult the worksheet and examine the sample. If the sample appears to be different from the preliminary information found on the worksheet, consult your supervisor.

11.4.8. If the preliminary evaluations indicated the need for particle size reduction, homogenize the waste, weigh out a sufficient size subsample and prepare for leaching by crushing, cutting, or grinding such that all particles are less than 1 cm in size as measured with a ruler (Do NOT sieve the sample). Size reduction is not required if the sample surface area is greater than or equal to 3.1 cm^2 per gram.

Note: To minimize loss of volatiles, samples for volatiles that require particle size reduction should be kept in sample storage (at 4 °C) until immediately before size reduction. Aggressive reduction which would generate heat should be avoided and exposure of the waste to the atmosphere should be avoided to the extent possible. Size reduction to a fine powder is not appropriate. Also see Section 11.3.11.

11.4.8.1. Consult your supervisor or manager when dealing with unusual sample matrices (e.g., wood, cloth, metal, brick). Scissors or shears may be used to cut cloth, plastic or sheet metal. Saws may be used for wood or solid metal. Bricks, rocks, or other solids amenable to grinding should be subcontracted out for particle size reduction (Contact PA or PM).

11.4.9. Place the ZHE apparatus on the balance and tare the balance.

11.4.10.

11.4.11. Determine the appropriate size subsample to weigh using the percent solids information from Section 11.2.5 and record the weight.

11.4.11.1. For wastes that are 100% solids, a 25 g sample is used.

11.4.11.2. For wastes containing < 0.5% solids the liquid portion of the waste, after filtration, is defined as the TCLP leachate. Filter enough of the sample to support all of the volatile analyses required.

11.4.11.3. For wastes containing ≥ 0.5% and < 5.0% solids, a 500 g subsample of waste is recommended.

11.4.11.4. If the sample has ≥ 5.0% solids, the appropriate sample size should be determined using the equation in Section 12.1.2.

Note: For wastes containing greater than 0.5% wet or dry solids (Section 11.2.5), the "solids" value from the ZHE filtration process may be used to determine the volume of fluid to load into the ZHE. This approach is recommended since the solids value from Section 11.2.5 may differ from the filtration solids due to sample variability or differences in the filtration apparatus.

11.4.12. Homogenize and transfer an appropriate size subsample of the waste into the ZHE and record the mass.

11.4.13. Carefully place the glass fiber filter between the support screens and secure to the ZHE. Tighten all the fittings.

11.4.14. Place the ZHE in a vertical position; open both the gas AND liquid inlet/outlet valves. Attach a gas line to the gas inlet/outlet valve.

11.4.15. If the waste is 100% solid, slowly increase the pressure to a maximum of 50 psi to force out as much headspace as possible and proceed to Section 11.4.18.

11.4.16. If the waste is < 100% solids, carefully apply gentle pressure of 10 psi (or more, if necessary) to force all headspace slowly out of the ZHE. At the FIRST appearance of liquid from the liquid inlet/outlet valve, quickly close the valve and discontinue gas pressure.

11.4.17. Assemble a syringe and place the plunger in all the way. Adjust the tension on the plunger to provide slight drag. Attach the pre-weighed syringe or Tedlar bag to the liquid inlet/outlet valve and open the valve. Record the tare weight of the collection device.

11.4.18. Carefully apply gas pressure of no more than 10 psi to force out the liquid phase. Allow the sample to filter until no SIGNIFICANT additional filtrate has passed in a 2 minute period.

Note: If the capacity of the syringe is reached, close the liquid inlet/outlet valve, discontinue gas pressure, remove the syringe and return to Section 11.4.15.

11.4.19. Repeat previous step increasing the pressure in 10 PSI increments until 50 psi is reached and no significant liquid has passed in a 2 minute period. Remove the collection device and record the total weight of the collection device with filtrate on Worksheet 4, Line 3a. Close the valve and discontinue gas pressure. Transfer the filtrate to VOA vials and label appropriately. Calculate the weight of filtrate collected.

Notes: If the original waste contained less than 0.5% solids (Section 11.2.5), this filtrate is defined as the TCLP leachate and you may proceed to Section 11.4.28. Otherwise, save the vials by storing at 4 C under minimal headspace conditions, for recombination as in Section 11.4.27.

The material remaining in the ZHE is defined to be the "solid" phase. Calculate the weight of the solid phase using Equation 4.

11.4.20. Based on the information from Sections 11.2.5 and 11.4.11 and using the formula in 12.1.3, determine the weight of fluid to load into the ZHE on the "solid" phase. The ZHE device has approximately a 500-mL capacity. Based on the need to add an amount of extraction fluid equal to 20 times the mass of the "solid" phase, the ZHE can therefore accommodate a maximum of 25 grams of "solid".

Note: The TCLP ZHE prep uses only TCLP fluid #1; the SPLP ZHE prep uses only SPLP fluid #1*(#3 at Santa Ana).*

11.4.21. Load the fluid transfer reservoir with an excess of Fluid #1 and preflush the transfer line to eliminate air pockets. Be sure the required volume remains.

11.4.22. Attach the transfer line to the liquid inlet/outlet valve and open the valve. Carefully pump the required volume into the ZHE and close the valve. Disconnect the transfer line.

11.4.23. Check the ZHE to make sure all the valves are closed and manually rotate the ZHE (end-over-end) 2 or 3 times. Reposition the ZHE in the vertical position.

11.4.24. Pressurize the ZHE to 5-10 psi. Allow to stand for 10 minutes, and then recheck the pressure. If the ZHE appears to be leaking, follow the corrective action protocols recommended by the manufacturer and repeat the analysis.

11.4.25. Slowly open the liquid inlet/outlet valve to bleed out any headspace that may have been introduced during the introduction of the Fluid. Upon the first sign of liquid from the valve, close the valve.

11.4.26. Repressurize the ZHE to 5-10 psi and place in the rotary agitator. Rotate at 28-32 rpm for 16-20 hours. Room temperature should be 23 ± 2 °C. The room temperature and time should be checked at both the start and end of the extraction and recorded.

11.4.27. Confirm that the pressure of 5-10 psi was maintained throughout the leaching. If it was NOT maintained, return to Section 11.4.1 and repeat the leachate with a new aliquot of sample.

11.4.28. Attach a syringe or Tedlar bag and open the liquid inlet/outlet valve to collect the aqueous leachate and proceed as outlined in 11.4.19 - 11.4.20. Record the volume/mass of the leachate and any oil. Record the date and time the filtration is completed.

Notes: If the waste contained an initial liquid phase, the liquid may be filtered directly into the same collection device holding the initial liquid phase of the waste.

A separate filtrate collection container must be used if combination would create multiple phases or there is not enough volume left within the filtrate collection container.

11.4.29. If the waste contained an initial filtrate (Section 11.4.18) that is miscible with the solid phase leachate (as determined in Section 11.2.8), the solid phase leachate and the initial filtrate are directly recombined in the correct

proportions (see Section 12.1.4). If the individual phases are NOT compatible, they are to be collected, prepped and analyzed separately.

Note: Chill the filtrate and receiving vessels before recombining.

11.4.30. Following collection, store the TCLP leachate in 3 20-mL VOA vials with minimal headspace at 4 ± 2 °C and prepare for analysis as soon as possible using the appropriate organic extraction procedure (see Section 16.3).

11.4.31. If the individual phases are analyzed separately, combine the results mathematically by using the recombination calculation in Section 12.1.5.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Calculations

12.1.1. Calculation of Percent Wet Solids:

$$Percent\ Wet\ Solids = 100\left(\frac{Mass,"solid"\ phase}{Mass, initial\ subsample}\right)$$

12.1.2. Calculation of weight of waste to charge to ZHE:

$$Weight\ of\ waste\ to\ charge\ to\ ZHE = 100\left(\frac{25}{\%\ wet\ solids}\right)$$

12.1.3. Calculation of weight of extraction fluid to use:

$$Weight\ of\ Extraction\ fluid = \frac{20\ x\ \%\ wet\ solids\ x\ weight\ of\ waste\ to\ be\ extracted}{100}$$

12.1.4. Calculation of volume of initial filtrate phase to recombine with solid phase leachate:

$$\text{Volume of filtrate for recombination} = \left(\frac{Weight\ of\ solids\ leached}{Total\ weight\ of\ solids}\right)\left(\frac{Leachate\ recovered}{Fluid\ added}\right)\left(Volume\ of\ initial\ aqueous\ filtrate\right)$$

12.1.5. Mathematical recombination of analytical results:

$$Final\ Analyte\ Concentration = \frac{(V_1 \times C_1) + (V_2 \times C_2)}{V_1 + V_2}$$

V_1 = total volume of the initial filtrate phase (L).
C_1 = analyte concentration in initial filtrate phase (mg/L).
V_2 = volume of the theoretical solid phase leachate (L).
C_2 = analyte concentration in solid phase leachate (mg/L).,

12.2. REPORTING REQUIREMENTS

12.2.1. Follow these reporting conventions for multi-phase samples:

12.2.1.1. If both phases have positive results, use the values from each phase to calculate the recombined result. Use the reporting limit for each phase to calculate the recombined reporting limit.

12.2.1.2. If both phases are "ND," not detected, the recombined result is "ND," and the reporting limit is calculated from the reporting limit for each phase.

12.2.1.3. If one phase is "ND" and the other phase has a positive result, use the reporting limit for the "ND" phase and the positive value for the other phase to calculate the combined result. The combined reporting limit is based on the reporting limit for both phases. If the combined result is less than the combined reporting limit, then supply a footnote to indicate that "a positive result was detected below the calculated detection limit."

12.2.2. Units - regardless of the nature of the sample, all TCLP and SPLP results are reported in units of mg/L.

12.2.3. For limits and significant figures, consult the appropriate analytical methods (Section 16.3).

12.2.4. Anomalies - all anomalies observed during the leach procedure must be noted on the worksheet or an anomaly form. Some examples of such anomalies are:

12.2.4.1. Sample was monolithic - subsample was obtained by crushing, cutting, grinding, sawing, etc.

12.2.4.2. Insufficient sample - less than the required 100 g minimum was available.

12.2.4.3. Multiple phases - "X" phases were present.

12.2.4.4. Sample was oil - single phase.

12.2.4.5. Sample contained liquid which did not filter under test conditions.

12.3. REVIEW REQUIREMENTS

12.3.1. Review all applicable holding times. If a holding time was exceeded, confirm that a holding time violation form was properly documented and routed.

12.3.2. If Total analysis results are available, those results may be compared with the TCLP analysis results according to the following:

$$Total \geq 20 \times TCLP$$

NOTE: Assumes the sample is 100% Solids.

12.3.3. Total constituent analysis results can be used to demonstrate the TCLP protocol is unnecessary. In performing a TCLP analysis, there is a 20:1 dilution of the original sample with the leaching solution. Thus, if the "total constituent" result is less than 20 times the TC level, it is impossible for the leachate to "fail" and the TCLP does not need to be performed. For example, the TC level for lead is 5.0 mg/L (ppm). Therefore, if a sample of lead-contaminated soil contains less than 100 ppm total lead, a TCLP test need not be run for lead.

13. METHOD PERFORMANCE

13.1. Refer to individual analysis SOPs.

13.2. Training Qualification:
The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience.

14. POLLUTION PREVENTION

14.1. This method does not contain any specific modifications that serve to minimize or prevent pollution.

15. WASTE MANAGEMENT

15.1. Waste generated in this procedure must be segregated and disposed according to the facility's hazardous wastes procedures. The Environmental Health and Safety Director should be contacted if additional information is required.

16. REFERENCES

16.1. Method 1311, Toxicity Characteristic Leaching Procedure, Revision 0, July 1992, SW-846 Final Update I.

16.2. Method 1312, Synthetic Precipitation Leaching Procedure, Revision 0, November 1992, SW-846 Proposed Update II.

16.3. Related Documents

16.3.1. Toxicity Characteristic: Corrections to Final Rule. Method 1311, Federal Register, Vol. 55, No. 126, Friday, June 29, 1990.

16.3.2. Toxicity Characteristic: Final Rule. Method 1311, Federal Register, Vol. 55, No. 61, Thursday, March 29, 1990.

16.3.3. Technical Background Document and Response To Comments, Method 1311, Toxicity Characteristic Leaching Procedure, USEPA/OSW, April, 1989.

16.3.4. QA-003, Quanterra QC Program

16.3.5. CORP-IP-0003: Acid Digestion of Aqueous Samples by SW846 and MCAWW 200 Series Methods.

16.3.6. CORP-MT-0001: Inductively Coupled Plasma-Atomic Emission Spectroscopy, Spectrometric Method for Trace Element Analysis, Method 6010A and Method 200.7.

16.3.7. CORP-MT-0003: Graphite Furnace Atomic Absorption Spectroscopy, SW846 Method 7000A and MCAWW 200 Series Methods.

16.3.8. CORP-MT-0005: Preparation and Analysis of Mercury in Aqueous Samples by Cold Vapor Atomic Absorption, SW-846 7470A and MCAWW 245.1.

16.3.9. CORP-IP-0003: Acid Digestion of Aqueous Samples by SW846 and MCAWW 200 Series Methods.

16.3.10. CORP-MS-0002: Determination of Volatile Organics by GC/MS based on Methods 8240B and 8260A.

16.3.11. CORP-MS-0001 : GC/MS Analysis Based on Method 8270B, SW846.

16.3.12. CORP-GC-0001: Gas Chromatographic Analysis Based on Methods 8000A, 8010B, 8020A, 8080A and 8150B, SW846.

16.3.13. CORP-OP-0001: Extraction and Cleanup of Organic Compounds from Waters and Soils, Based on SW846 3500 Series, 3600 Series, 8150 and 600 Series Methods.

17. MISCELLANEOUS

17.1. Modifications/Interpretations from Reference Methods

17.1.1. Section 8: Preliminary Evaluations. Section 7.1 of the source method states that the sample aliquot used for the preliminary evaluation "...may not actually undergo TCLP extraction." Section 7.1.5 of the source method indicates that the portion used for the preliminary evaluation may be used for either the ZHE or non-volatile extraction if the sample was 100% solid. Section 7.1.5 further indicates that if the sample was subjected to filtration (i.e., < 100% solid) that this aliquot may be used for the non-volatile extraction procedure only as long as sufficient sample is available (minimum 100 g). Samples which have been subjected to the oven drying step may not be used for TCLP extraction because solid phase degradation may result upon heating.

17.1.2. Section 11.2.5.6.3: Percent Solids Determination. Section 7.1.2 of the source method indicates that "if the percent wet solids is ≥ 0.5% and it is noticed that a small amount of the filtrate is entrained in wetting of the filter" that the filter should be oven dried to determine percent dry solids ". Drying of oil or organic matrices can both be hazardous and inappropriate. Additionally, it may be impossible to achieve a constant weight when performing this step. Due to safety concerns, if obviously oily or heavy organic matrices are entrained on the filter, the filter is not oven dried.

17.1.3. Section 11.2.8: Preliminary Determination of Filtrate/Extraction Fluid Compatibility. Section 7.2.13 of the source method provides no guidance as to how to make this determination. As a result, the procedure herein was developed and incorporated into the Preliminary Determinations section.

17.1.4. Section 9.2: TCLP Extraction Blanks. Section 8.1 of the source method states that a minimum of one blank for every 20 extractions "...that have been conducted in an extraction vessel." Quanterra has interpreted this to mean one blank per twenty samples leached per TYPE of leaching vessel (i.e., Bottle or ZHE) per leach fluid used.

17.1.5. Section 11.2.7.9: Determination of Appropriate Extraction Fluid. Method 1311 does not address the appropriate approach to take if the pH equals 5.0. This SOP requires that Fluid #1 must be used if the pH is less than or equal to 5.0.

17.1.6. Section 9.4: QA/QC - Matrix Spikes. Section 8.2 of the source method states "A matrix spike shall be performed for each waste type..." and "A minimum of one matrix spike must be analyzed for each analytical batch." Further, Section 8.2.3 of the source method also states "The purpose of the matrix spike is to monitor the performance of the analytical methods used, and to determine whether matrix interferences exist." The standard Quanterra QAPP is designed to address the performance monitoring of analytical methodology through the LCS program. A minimum of one MS and MSD will be prepared for each TCLP leachate batch. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, the MS/MSD results have immediate bearing only on the specific sample spiked and not all samples in the batch.

17.1.7. Section 8.2.2 of the source method states that "In most cases, matrix spikes should be added at a concentration equivalent to the corresponding regulatory level." The method also states "If the analyte concentration is less than one half the regulatory level, the spike concentration may be as low as one half of the analyte concentration but may not be less than five times the method detection limit". For several analytes, spiking at the regulatory level is inappropriate to the range of analysis afforded by the determinative methods. Due to the wide range in these levels, Quanterra spikes at the levels specified in the determinative SOPs.

17.2. Modifications from Previous SOP

None

17.3. Documentation and Record Management

The following documentation comprises a complete TCLP preparation raw data package:

- Completed worksheets (Appendix C)
- Non-conformance summary (if applicable).
- Anomaly documentation (if applicable).

APPENDIX A

TABLES

Table 3 - Toxicity Characteristic Analytes and Regulatory Levels (Final Rule)

Contaminant	mg/L
Arsenic	5.0
Barium	100.0
Benzene	0.5
Cadmium	1.0
Carbon tetrachloride	0.5
Chlordane	0.03
Chlorobenzene	100.0
Chloroform	6.0
Chromium	5.0
Total Cresols (used if isomers not resolved)	200.0
2,4-D	10.0
1,4-Dichlorobenzene	7.5
1,2-Dichloroethane	0.5
2,4-Dinitrotoluene	0.13
1,1-Dichloroethylene	0.7
Endrin	0.02
Heptachlor (& epoxide)	0.008
Hexachlorobenzene	0.13
Hexachlorobutadiene	0.5
Hexachloroethane	3.0
Lead	5.0
Lindane	0.4
Mercury	0.2
Methoxychlor	10.0
Methyl ethyl ketone	200.0
Nitrobenzene	2.0
Pentachlorophenol	100.0
Pyridine	5.0
Selenium	1.0
Silver	5.0
Tetrachloroethylene	0.7
Toxaphene	0.5
Trichloroethylene	0.5
2,4,5-Trichlorophenol	400.0
2,4,6-Trichlorophenol	2.0
2,4,5-TP (Silvex)	1.0
Vinyl chloride	0.2

APPENDIX B

FIGURES

Figure 1 & 2 - Rotary Agitation Apparatus and Zero Headspace Extraction Vessel (ZHE)

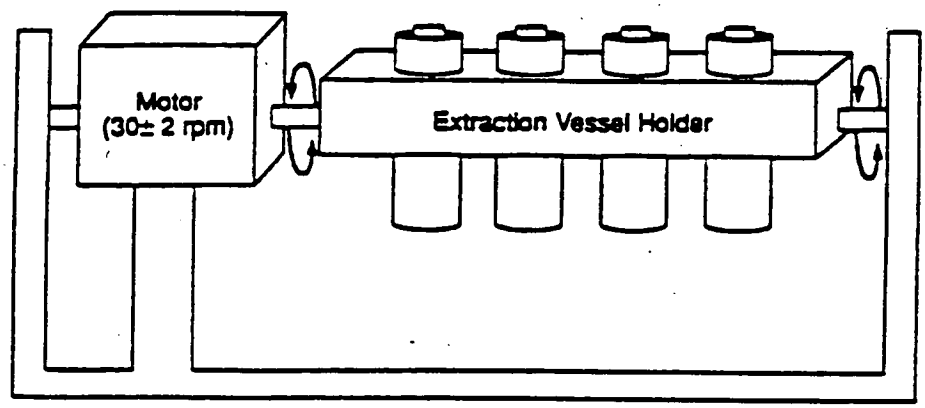

Figure 1. Rotary Agitation Apparatus

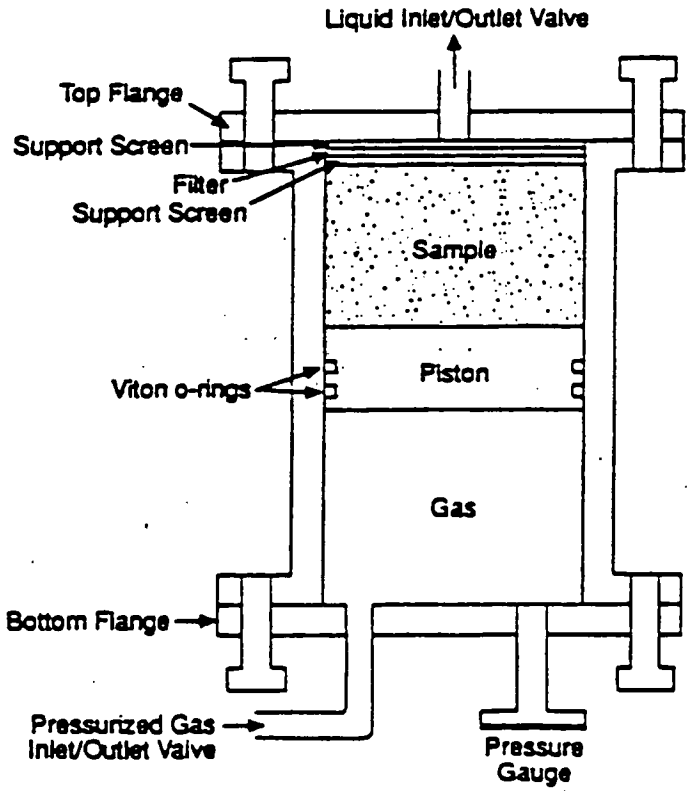

Figure 3 - US Environmental Protection Agency Memorandum #35, Page 1

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
WASHINGTON, D.C. 20460

OFFICE OF
SOLID WASTE AND EMERGENCY RESPONSE

MEMORANDUM # 35

DATE: June 12, 1992

SUBJECT: Notes on RCRA Methods and QA Activities

From: Gail Hansen, Chief Gail Hansen
Methods Section (OS-331)

This memo addresses the following topics:

- 1992 Symposium on Waste Testing and Quality Assurance
- SW-846 Update
  - Final Rule for January 23, 1989 Proposed Rule
  - Notice, Proposed Rulemaking for the Second Update to the Third Edition
- Chlorofluorocarbon 113 (CFC-113) Solvent Replacement Update
- Environmental Monitoring Methods Index (EMMI)
- Sampling Work Group Formation
- MICE Update
- Oily Waste Analysis
- Electronic SW-846 Availability.

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Figure 3 - US Environmental Protection Agency Memorandum #35, Page 10

Oily Waste Analysis

One of the most frequently asked questions on the MICE Service concerns the application of the TCLP, Method 1311, to oily wastes. Many callers request technical guidance on the extraction of oily wastes due to the difficulty in the filtration on these types of waste. In many cases, an oily waste does not filter completely due to premature clogging of the glass fiber filter. This can result in the retention of standing liquid on the glass fiber filter. Material that do not pass through the glass fiber filter at the conclusion of the filtration step is defined by the method as the solid phase of the waste. The solid phase is then subjected to the leaching procedure of the TCLP. For oily wastes, clogging of the glass fiber filter can result in an overestimation of the amount of solid material available for leaching.

To solve this problem, the Agency recommends a conservative approach, one that probably will overestimate the amount of leaching. Rather than performing the TCLP extraction on the unfiltered portion of the oily waste, assume the waste is 100% liquid (e.g., will pass through the glass fiber filter) and perform a totals analysis on the oily waste to determine if the oil exceeds the appropriate regulatory level.

Filterable waste oil generated during the TCLP must be analyzed for a variety of organic and inorganic analytes. The OSW recognizes the difficulty in achieving acceptable performance for the analysis of waste oil using methods currently provided in SW-846. As a result, the Agency will provide several new methods for the preparation and analysis of oil samples to the Organic Methods Workgroup in July. In addition, a microwave assisted digestion procedure should improve the analysis of metals and will be proposed as part of the Second Update of the Third Edition of SW-846. Brief descriptions of these techniques are provided below, for additional information on the organic procedures contact Barry Lesnik at (202) 260-7459. For additional information on microwave digestion contact Ollie Fordham (202) 260-4778.

The use of purge-and-trap (Method 5030) for volatiles in oil generally results in severe contamination of analytical instrumentation. Traps, transfer lines and chromatography columns may become contaminated with oil. This leads to elevated baselines, hydrocarbon background in subsequent analyses, and cross-contamination. Headspace (Method 3810) is currently allowed only as a screening procedure in SW-846. The Agency is evaluating the use of headspace in conjunction with isotope dilution mass spectrometry for the quantitative analysis of volatiles in oil. Headspace reduces interference problems encountered with purge-and-trap. However, headspace quantitation can be questionable because the distribution of analytes is not

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APPENDIX C

WORKSHEETS

TCLP/SPLP Worksheet 1 - Sample Description

Laboratory Sample No.					
Field Sample No.					
A. Sample Description					
Number of phases					
1.					
2.					
a. lighter than					
b. water					
c. heavier than water					
B. Percent Solid Phase					
1. weight of filter (g)					
2. weight of subsample					
a. gross weight (g)					
b. tare weight (g)					
c. net weight (g)					
3. weight of filtrate					
a. gross weight (g)					
b. tare weight (g)					
c. net weight (g)					
4. total weight wet solids[0]					
5. weight percent wet solids[1] (%)					
6. weight percent dry solids[2] (%)					
a. gross dry weight (g)					
b. final gross dry weight[2] (g)					
c. percent dry solids (%)					
7. volume of initial aqueous filtrate (mL)					
8. volume of initial organic filtrate (mL)					

[0] Total Weight of wet solids $= (2c - 3c)$

[1] Weight percent wet solids $= 100\left(\frac{\text{Total weight wet solids, B4}}{\text{Weight of subsample, B2c}}\right)$

[2] Weight percent dry solids $= 100\left(\frac{(\text{Gross dry weight, B6b}) - (\text{weight of filter, B1})}{\text{Weight of subsample, B2c}}\right)$

Comments: _____

_____

Analyst: _____ Date: _____

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TCLP/SPLP Worksheet 2 - Selection of Extraction Fluid

TCLP

Laboratory Sample No.					
Field Sample No.					
C. Extraction Fluid Determination—does not apply to determination of volatile organic components.					
1. particle size reduction? yes/no (<1 mm)					
2. sample weight, ✓ if 5.0 ± 0.1 g					
3. volume of water, ✓ if 96.5 ± 1.0 mL					
4. initial pH (after 5 min. mixing time)					
5. if pH >5.0, ✓ if 3.5 mL 1N HCL					
6. ✓ if heated and held at 50^{0}C for ten minutes					
7. secondary pH (at room temperature)					
D. Selection of Extraction Fluid					
1. ✓ if pH from § C (4) or § C (7) is <5.0, use extraction fluid No. 1.					
2. ✓ if pH from § C (7) IS >5.0, use extraction fluid No. 2					
3. ✓ if filtrate and fluid are miscible					

SPLP

Laboratory Sample No.					
Field Sample No.					
D. Selection of Extraction Fluid (✓ one)					
Fluid 1: Soils—East of the Mississippi River; Wastes; or Wastewaters.					
Fluid 2: Soils—West of Mississippi River					
Fluid 3: If VOCs or Cyanide containing wastes.					
1. ✓ if filtrate and fluid are miscible					

Comments: _____

Analyst: _____ Date: _____

TCLP/SPLP Worksheet 3 - Extraction for Metals, Semi-Volatile Organic Components and Pesticides/Herbicides

Laboratory Sample No.					
Field Sample No.					
E. Determination of Samples Size for Leach Testing—the method requires a minimum 100 gram sample size for extraction					
1. particle size reduction? yes/no (<9.5 mm)					
2. weight of wet solids after filtration (g)					
F. Determination of Amount of Extraction Fluid—the selection of the correct extraction fluid is found in Section D., Worksheet No. 2					
1. Fluid weight = 20 x solids weight (g)					
G. Record of Extraction Test—the extraction period is specified as 18 ± 2					
1. room temperature initial (°C) final (°C)					
2. vessel number					
3. extraction start time					
4. extraction stop time					
5. date and time filtration complete					
6. pH of leachate					
7. volume of leachate (for multiphase samples only) (mL)					
8. volume of initial aqueous filtrate for recombination[3] (mL)					
9. combined initial aqueous + leachate (mL)					

[3] Volume of filtrate used for recombination = $\left(\frac{\text{Weight of solids leached, E2}}{\text{Total weight of solids, B4}}\right)\left(\frac{\text{Leachate recovered, G6}}{\text{Fluid added, F1}}\right)\left(\text{Volume of initial aqueous filtrate, B7}\right)$

Comments: _____

_____

Analyst: _____ Date: _____

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TCLP/SPLP Worksheet 4 - Zero Headspace Extraction (ZHE)

Laboratory Sample No.					
Field Sample No.					
H. Determination of Sample Size for Leach Testing—maximum 25 grams					
1. particle size reduction? yes/no (<9.5 mm)					
2. weight of wet solid (g)					
I. Determination of Amount of Extraction Fluid No. 1					
1. weight of material added to ZHE (g)					
2. volume of filtrate in syringe (mL)					
3. weight of filtrate in syringe					
a. gross weight (g)					
b. tare weight (g)					
c. net weight (g)					
4. wet solids in ZHE[4] (g)					
5. weight of fluid to add[5] (g)					
J. Record of ZHE Extraction Text—the extraction period is as 18 ± 2 hours.					
1. room temperature initial (°C) final (°C)					
2. ZHE vessel number					
3. extraction start date & time					
4. extraction stop date & time					
5. ✓ if still under positive pressure					
6. filtration completed date & time					
7. volume of filtrate recovered after leaching (mL)					
a. volume of oil recovered after leaching (mL)					
8. volume of initial aqueous filtrate for recombination[6] (mL)					
9. combined initial aqueous + leachate (mL)					

[4] Solids remaining in ZHE = (*Material added, I1*) - (*Weight of filtrate in syringe, I3*)

[5] Weight of fluid to add = 20(*Wet solids in ZHE, I4*)

[6] Volume of filtrate used for recombination = $\left(\frac{\text{Weight of solids leached, I4}}{\text{Total weight of solids, B4}}\right)\left(\frac{\text{Leachate recovered, J7}}{\text{Fluid added, I5}}\right)$(*Volume initial aqueous filtrate, B7*)

Comments: _____

_____

Analyst: _____ Date: _____

TCLP/SPLP Worksheet 5 - Organic Results

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TCLP/SPLP ZHE Worksheet No. 5
Organic Results

Sample Number: _____

Organics Preparation		Volume	Units
Combined initial Aqueous/Leachate from § J9	V_A		mL
Oil phase Volume for Calculations[7]	V_O		mL

Analyte		Oil Phase			Combined Initial Aqueous/Leachate			Final Results	
Compound	Std. Reporting Limit (mg/L)	Dilution Factor	Result Co	Reporting Limit	Dilution Factor	Results C_A	Reporting Limit	Result C_{Final}	Reporting Limit

$$C_{Final} = \frac{(C_A \times V_A) + (C_O \times V_O)}{V_A + V_O}$$

[7] Volume of oil phase for calculation = $\left(\frac{\text{Wet solids leached, I4}}{\text{Total solid, B4}}\right)\left(\frac{\text{Leachate recovered, J7}}{\text{Fluid added, I5}}\right)(\text{Initial volume of oil, B8}) + (\text{Oil recovered from leaching, J7a})$

Comments: _____

_____

Analyst: _____ Date: _____

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TCLP/SPLP Worksheet 6 - Metals Results

Metals Results

Sample Number : _____

Metals Preparation		Volume	Units
Combined Initial Aqueous/Leachate from §G8	VA		mL
Oil phase volume for calculations[8]	VO		mL

Analyte		Oil Phase			Combined Initial Aqueous/Leachate			Final Results	
Element	Std. Reporting Limit (mg/L)	Dilution Factor	Result Co	Reporting Limit	Dilution Factor	Results C_A	Reporting Limit	Result C_{Final}	Reporting Limit
Arsenic	0.5								
Barium	10								
Cadmium	0.1								
Chromium	0.5								
Lead	0.5								
Mercury	0.0002								
Selenium	0.25								
Silver	0.5								

$$C_{Final} = \frac{(C_A \times V_A) + (C_O \times V_O)}{V_A + V_O}$$

[8] Volume of oil phase for calculation = $\left(\frac{\text{Wet solids leached, E2}}{\text{Total solid, B4}}\right)\left(\frac{\text{Leachate recovered, G7}}{\text{Fluid added, F1}}\right)(\text{Initial volume of oil, B8}) + (\text{Oil recovered from leaching, G6a})$

Notes: If sample contains no solids, volume of oil phase (V_0) is the volume of the initial organic filtrate

In cases where analytes are detected in only one phase, a footnote indicating a result reported below the reporting limit should be included.

Comments: _____

_____

Analyst: _____ Date: _____

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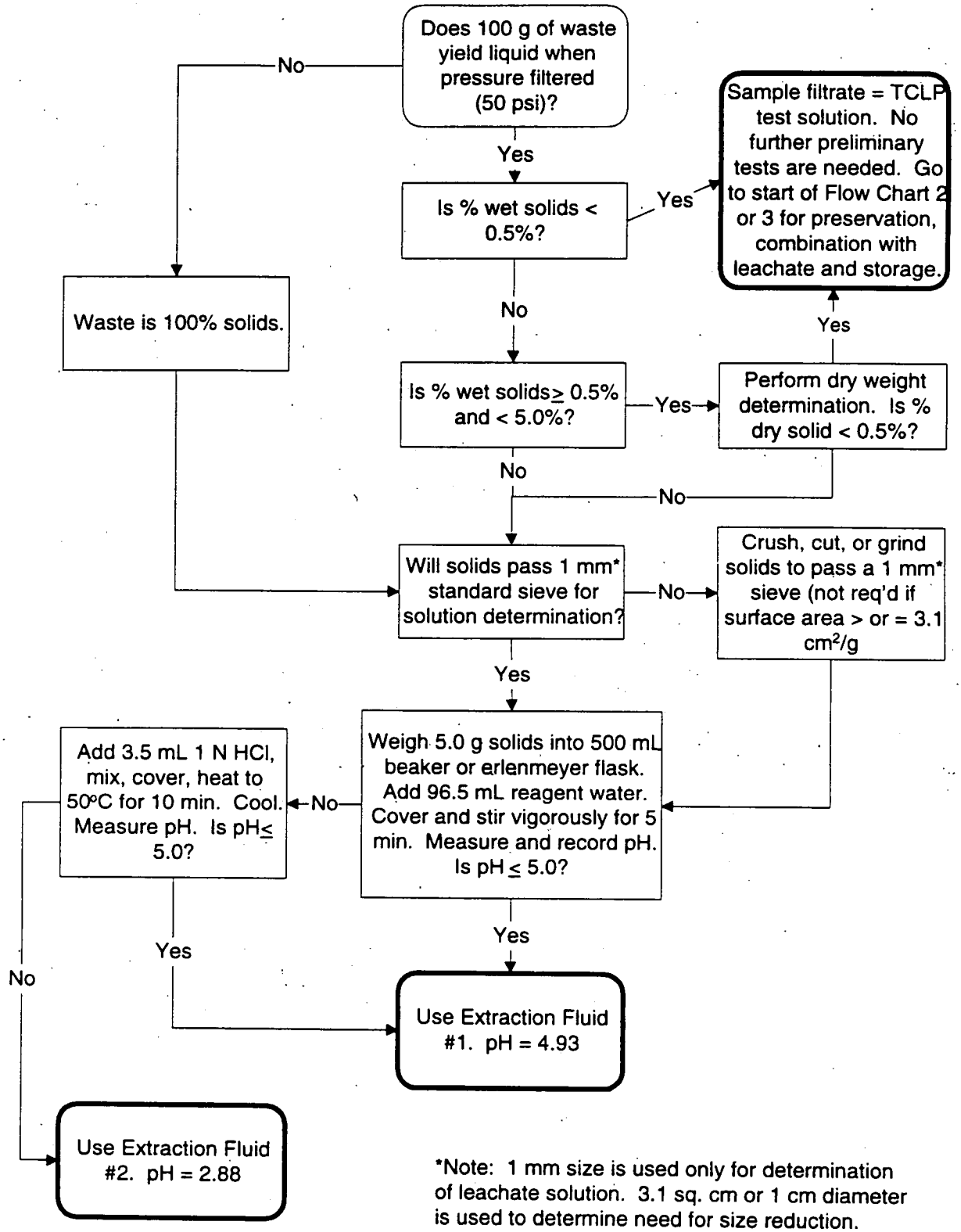
APPENDIX D

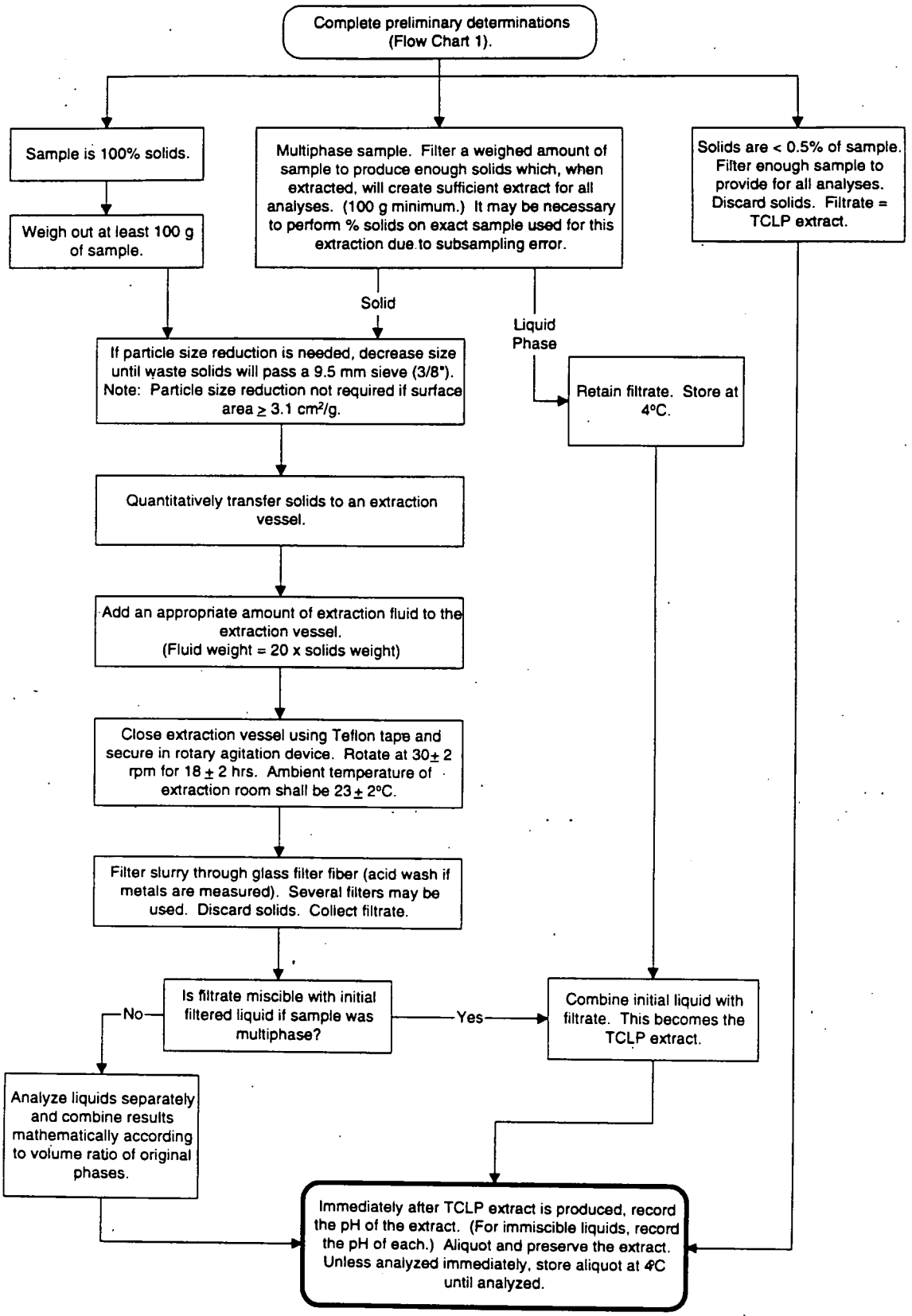
FLOW CHARTS

Flow Chart 1. Preliminary Sample Evaluation (Section 11.2)

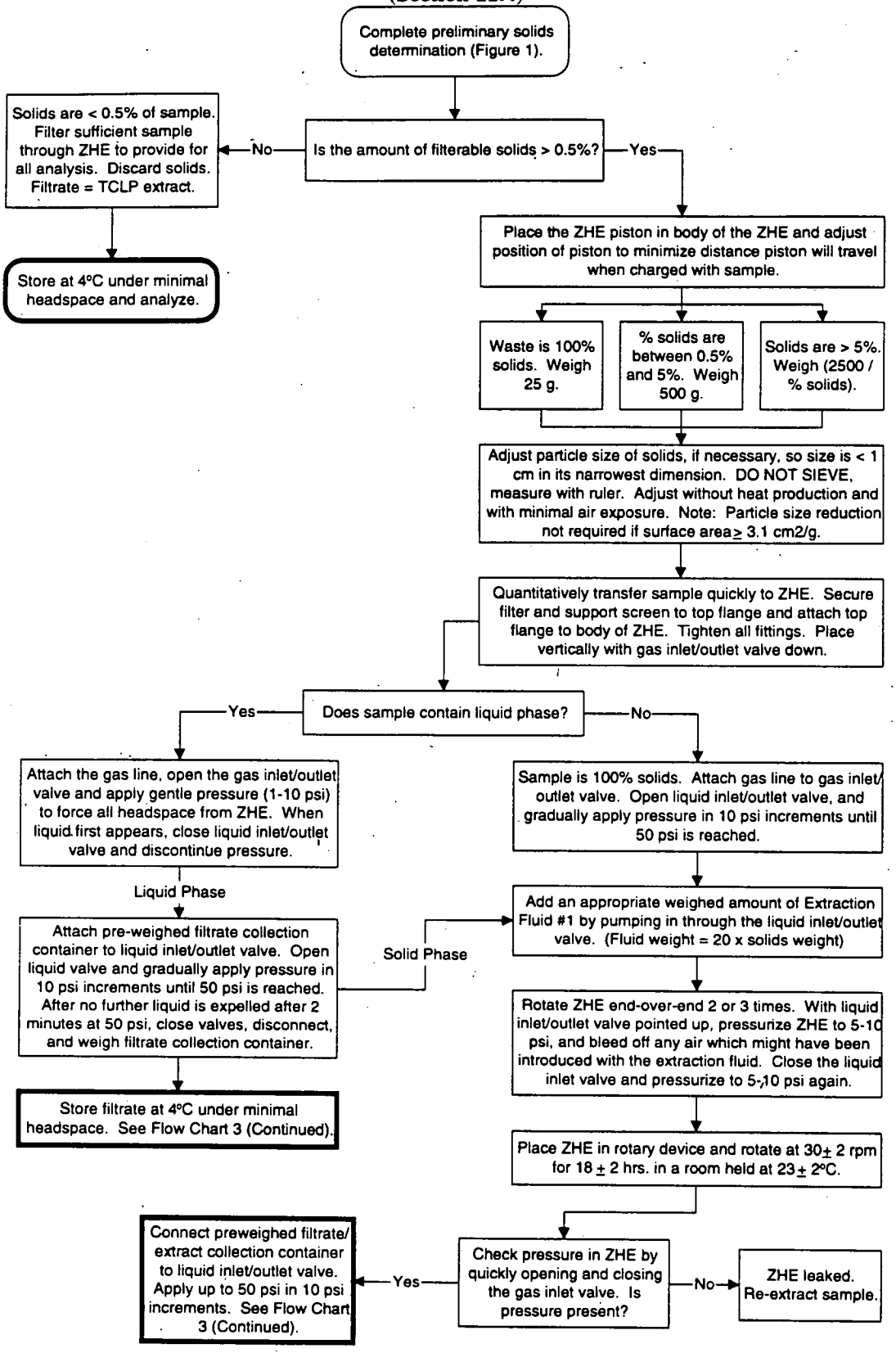


*Note: 1 mm size is used only for determination of leachate solution. 3.1 sq. cm or 1 cm diameter is used to determine need for size reduction.

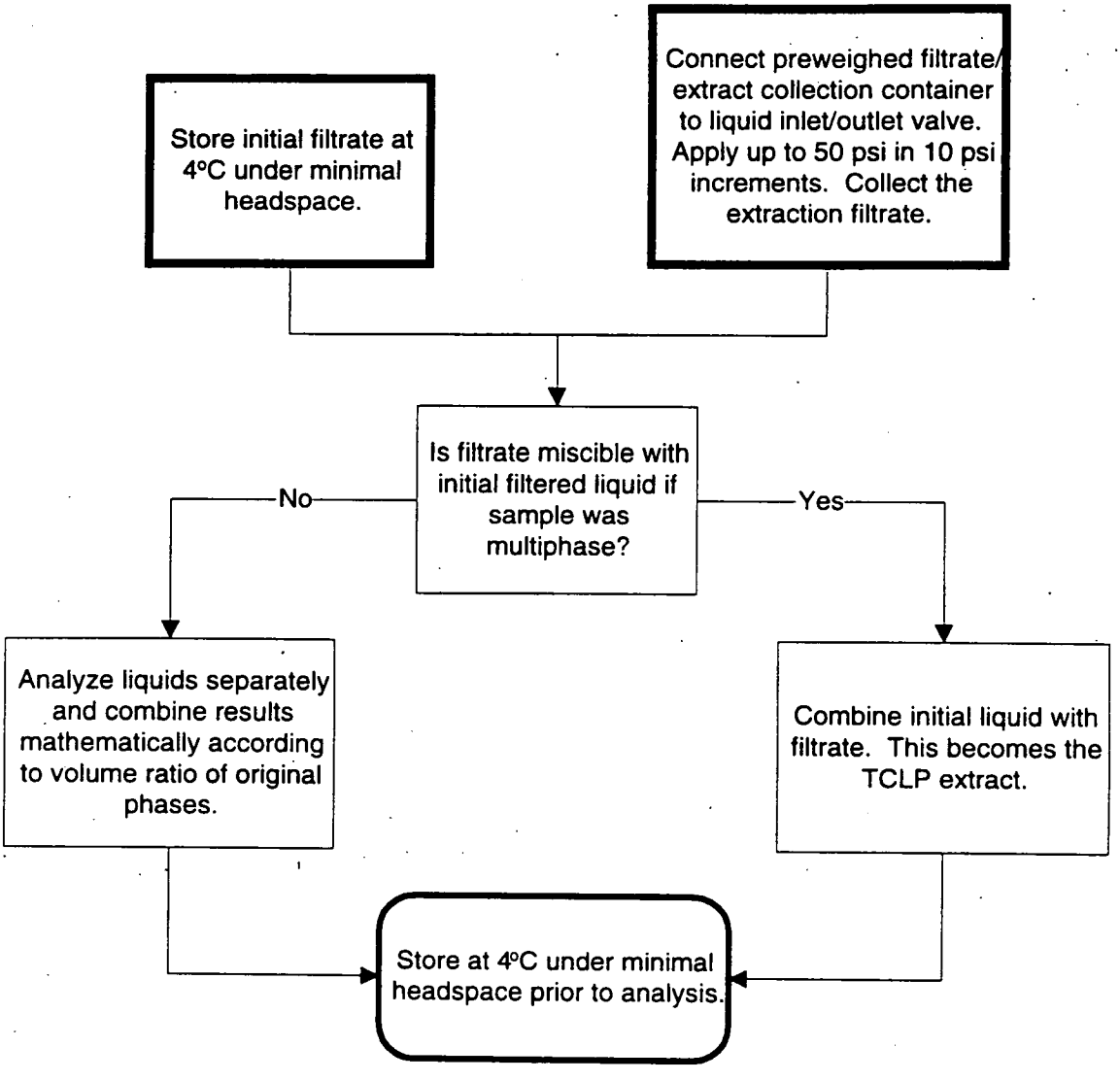
Flow Chart 2. Bottle Extraction, Non-Volatile Constituents (Section 11.3)



Flow Chart 3. ZHE Extraction, Volatile Constituents (Section 11.4)



Flow Chart 3. ZHE Extraction
(Continued)



QUANTERRA STANDARD OPERATING PROCEDURE

TITLE: DETERMINATION OF SOLIDS IN WATERS AND WASTES

(SUPERSEDES: CORP-WC-0002, REVISION 1)

Prepared by: Jeff Brenner

Reviewed by: _____
Technical Specialist, Josie Jones

Approved by: _____
Quality Assurance Manager, Gail Cho Celaschi

Approved by: _____
Environmental Health and Safety Coordinator, Cheryl Ann Stith

Approved by: _____
Laboratory Manager, Roger A. Freize II

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FIGURE 1: DECISION TREE FOR FAILED EC/TDS RATIO 30

TABLE 1: SUMMARY OF QUALITY CONTROL REQUIREMENTS FOR SOLIDS DETERMINATIONS 32

1. SCOPE AND APPLICATION

1.1. This SOP is applicable to the determination of total solids, total suspended solids, total dissolved solids, volatile solids, volatile dissolved solids, and volatile suspended solids using gravimetric techniques. This SOP is based on the residue methods contained in *Methods for Chemical Analysis of Waters and Wastes* (MCAWW) and *Standard Methods for the Examination of Water and Wastewater* (SM).

1.2. This SOP is applicable to drinking, surface, and saline waters and domestic and industrial wastes.

1.3. The Total Solids protocol is based on MCAWW Method 160.3 and Standard Method 2540B.

1.4. The Total Dissolved Solids protocol is based on MCAWW Method 160.1 and Standard Method 2540C.

1.5. The Total Suspended Solids protocol is based on MCAWW Method 160.2 and Standard Method 2540D.

1.6. The Volatile Solids protocol is based on MCAWW Method 160.4 and Standards Method 2540E.

1.7. The Volatile Dissolved Solids protocol is based on MCAWW Method 160.4 and Standards Method 2540E.

1.8. The Volatile Suspended Solids protocol is based on MCAWW Method 160.4 and Standards Method 2540E.

1.9. The methods cover a practical range of 10 mg/L to 20,000 mg/L (TSS: 4 mg/L - 20,000 mg/L). As a practical matter, the final residue weight should be limited to about 200 mg.

2. SUMMARY OF METHOD

2.1. Total Solids (TS): A well-mixed aliquot of the sample is quantitatively transferred to a preweighed evaporating dish and evaporated to dryness at 103-105 °C. The increase in weight over that of the empty dish represents the total solids.

2.2. Total Dissolved Solids (TDS): A well-mixed sample is filtered through a glass fiber filter. The filtrate is quantitatively transferred into a preweighed evaporating dish and is evaporated to dryness and then dried to constant weight at 180 °C. The increase in weight over that of the empty dish represents the total dissolved solids. The filter from this procedure may also be used for TSS/VSS determination.

2.3. Total Suspended Solids (TSS): A well-mixed sample is filtered through a pre-weighed glass fiber filter. The residue on the filter is dried to constant weight at 103-105 °C. The increase in weight over that of the pre-weighed filter represents the TSS content. The filtrate from this procedure may be used for TDS determination. The filter from this procedure may also be used for VSS analysis.

2.4. Volatile Solids (VS): The residue obtained from the determination of total solids is ignited at 550 °C in a muffle furnace. The loss of weight on ignition is reported as mg/L volatile solids.

2.5. Volatile Dissolved Solids (VDS): The residue obtained from the determination of total dissolved solids is ignited at 550 °C in a muffle furnace. The loss of weight on ignition is reported as mg/L volatile dissolved solids.

2.6. Volatile Suspended Solids (VSS): A well-mixed sample is filtered through a glass fiber filter to separate the suspended material. The filter is dried and weighed, then ignited at 550 °C and reweighed. Volatile suspended solids is determined from the weight loss after ignition. The filter from the analysis of TSS may be used for the determination of VSS.

3. DEFINITIONS

3.1. Total Solids (TS): The term applied to the residue left in the vessel after evaporation of a sample and its subsequent drying in an oven at 180 °C. Total solids includes "total suspended solids," the portion of solids retained by a filter, and "total dissolved solids," the portion that passes through the filter.

3.2. Total Dissolved Solids (TDS): Those solids capable of passing through a glass fiber filter and dried to constant weight at 180 °C. TDS is also referred to as filterable residue.

3.3. Total Suspended Solids (TSS): Those solids which are retained by a glass fiber filter and dried to constant weight at 103-105 °C. TSS is also referred to as non-filterable residue.

3.4. Volatile Solids (VS): The portion of total solids which is lost on ignition at 550 °C.

3.5. Volatile Dissolved Solids (VDS): The portion of total dissolved solids which is lost on ignition at 550 °C.

3.6. Volatile Suspended Solids (VSS): The portion of suspended solids which is lost on ignition at 550 °C.

3.7. Aliquot: A representative portion of a sample.

3.8. Reagent Water: Deionized water which is free of the analyte(s) of interest.

4. INTERFERENCES

4.1. Method interferences may be caused by contaminants, reagents, glassware, and other sample processing hardware. All these materials must be routinely demonstrated to be free from interferences under the conditions of analysis by running method blanks.

4.2. Non-homogeneous samples may give erroneous results. Samples should be mixed as thoroughly as possible before removing an aliquot for analysis.

4.3. Non-representative particulates such as leaves, sticks, fish, and lumps of fecal matter should be excluded from the sample if it is determined that their inclusion is not desired in the final result. The presence/removal of these artifacts should be noted on the benchsheet.

4.4. Samples containing large amounts of solids may filter slowly. Prolonged filtration times resulting from filter clogging may produce high TSS results due to increased colloidal materials captured on the clogged filter.

4.5. Oil and grease in the samples will cause unreliable results due to difficulty in drying to constant weight. Floating oil and grease, if present, should be included in the sample and dispersed by a blender device before aliquoting.

4.6. Filtration apparatus, filter material, pre-washing, post-washing, and drying temperatures are specified because these variables have been shown to affect the results.

4.7. The temperature at which the residue is dried has an important bearing on the results because weight losses due to volatilization of organic matter, mechanically

occluded water, water of crystallization, and gases from heat-induced chemical decomposition, as well as weight gains due to oxidation, depend on temperature and time of heating.

4.8. Each sample requires close attention to desiccation after drying. Minimize opening the desiccator because moist air enters. Some samples may be stronger desiccants than those used in the desiccator and may take on water.

4.9. Highly mineralized waters containing significant concentrations of calcium, magnesium, chloride, and/or sulfate may be hygroscopic and will require prolonged drying, desiccation and rapid weighing.

4.10. Samples containing high concentrations of bicarbonate may require careful and possibly prolonged drying to ensure that all the bicarbonate is converted to carbonate.

4.11. Too much residue in the drying vessel will crust over, entrapping water that will not be driven off during drying. Total residue should be limited to about 200 mg.

4.12. Some samples may have fine suspended solids which will pass through the glass fiber filter causing high TDS results.

4.13. Aluminum pans should not be used for TS or TDS analyses. Components in some samples may react to form aluminum compounds, causing unreliable results.

4.14. For samples high in dissolved solids, thoroughly wash the filter to ensure removal of dissolved material prior to TSS determination.

4.15. The volatile solids tests are subject to many errors due to the loss of water of crystallization, loss of volatile organic matter prior to combustion, incomplete oxidation of certain complex organics and decomposition of mineral salts during combustion. The results should not be considered an accurate measure of organic carbon in the sample.

5. SAFETY

5.1. Procedures shall be carried out in a manner that protects the health and safety of all Quanterra associates.

5.2. Eye protection that satisfies ANSI Z87.1 (as per the Chemical Hygiene Plan), laboratory coat, and chemically resistant gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have

been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.3. The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the Material Safety Data Sheets (MSDS) maintained in the laboratory.

5.4. Exposure to chemicals must be maintained **as low as reasonably achievable**; therefore, unless they are known to be non-hazardous, all samples should be opened, transferred, and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.5. The preparation of standards and reagents should be conducted in a fume hood with the sash closed as far as the operation will permit.

5.6. All work must be stopped in the event of a known or potential compromise to the health and safety of a Quanterra associate. The situation must be reported **immediately** to a laboratory supervisor.

5.7. If possible, place the oven or muffle furnace under a canopy hood or vent in to a fume hood. Do not stand directly in front of an oven or muffle furnace as the door is being opened. Stand behind the door to avoid breathing fumes that may be present.

5.8. Caution should be used when handling apparatus treated in the oven. Items will be hot. Heat resistant gloves and tongs must be used when placing material into or removing material from the muffle furnace. At a minimum, tongs must be used when placing material into or removing material from the oven.

6. EQUIPMENT AND SUPPLIES

6.1. Analytical balance capable of weighing to 0.0001 g.

6.2. Vacuum filtration apparatus.

6.3. Vacuum pump equipped with moisture trap.

6.4. Glass fiber filter disks, 47 mm, without organic binder (Gelman Type A/E) or equivalent.

Note: Commercially available pre-weighed filters may be used for TSS and VSS analyses.

6.5. Evaporating dishes, 100 mL, made of either porcelain, Vycor or platinum. These must be thoroughly cleaned, rinsed with deionized water, and baked at 180 ± 2 °C for at least one hour before use. Store in a desiccator.

Note: Glass beakers may also be used for TS or TDS determinations. Glass beakers may not be used for procedures requiring use of a muffle furnace.

6.6. Desiccators providing sufficient space for storage of samples in process separate from filters and evaporating dishes.

6.7. Desiccant containing a color indicator of moisture concentration or an instrumental indicator.

6.8. Drying ovens set at 103-105 °C and 180 ± 2 °C. Separate ovens should be maintained at appropriate temperatures if possible.

6.9. Muffle furnace (550 °C ± 50 °C).

6.10. Thermometers, NIST traceable.

6.11. Conductivity meter and associated apparatus.

6.12. Graduated cylinders, "to contain," assorted sizes.

6.13. Volumetric flasks, Class A, assorted sizes.

6.14. Glass weighing dishes large enough to hold a 47 mm filter.

6.15. Forceps for handling filters.

6.16. Crucible tongs.

6.17. Zetex gloves or other gloves capable of providing protection at 550 °C.

7. REAGENTS AND STANDARDS

7.1. Reagent water must be produced by a Millipore DI system or equivalent (see also Section 10.3).

7.2. LCS solution (ERA Mineral Standard for TSS and 500 mg/L NaCl for TDS and TS): Place 500 mg sodium chloride into a 1000 mL volumetric flask and dilute to volume with deionized water. Mix well. Prepare fresh every three months.

7.3. Spike solutions for Matrix Spikes for TDS analysis is 500 ppm TDS (5 g NaCl to diluted to a final volume of 1000 mL).

Note: Commercially available reference materials may also be used.

8. SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1. Samples should be collected in either plastic or glass bottles.

8.2. Samples must be stored at 4 °C ± 2 °C to minimize microbiological decomposition of solids. Bring samples to room temperature before analysis.

8.3. To achieve the reporting limits listed in Section 13, the recommended minimum volumes are as follows:

Parameter	Volume (mL)
TS	100
TDS	100
TSS	100
VS	100
VDS	100
VSS	250

8.4. Holding times for solids determinations are as follows:

Parameter	Holding Time (days)	Parameter	Holding Time (days)
TS	7	VS	7
TDS	7	VDS	7
TSS	7	VSS	7

9. QUALITY CONTROL

The Quanterra QC Program document (QA-003) provides further details of the QC and corrective action guidelines presented in this SOP. Refer to this document if additional guidance is required.

Table I (Section 17.7) provides a summary of quality control requirements including type, frequency, acceptance criteria, and corrective action.

9.1. Initial Demonstration of Capability

Prior to analysis of any samples using this SOP, the following requirements must be met:

9.1.1. Method Detection Limit (MDL): An MDL must be determined prior to analysis of any samples. The MDL is determined using seven replicates of reagent water, spiked with a reference material that has been carried through the entire analytical procedure. MDLs must be redetermined on an annual basis in accordance with 40 CFR Part 136 Appendix B requirements as detailed in Quanterra QA Policy QA-005. The result of the MDL determination must be below the Quanterra standard reporting limit.

9.1.2. Initial Demonstration Study: This requires the analysis of four QC check samples. The QC check sample is a well-characterized, laboratory-generated sample used to monitor method performance, which should contain the analyte(s) of interest. The results of the initial demonstration study must be acceptable before analysis of samples under this SOP may begin.

9.1.2.1. Four aliquots of the check sample (LCS) are prepared and analyzed using the procedures detailed in this SOP.

9.1.2.2. Calculations and acceptance criteria for the LCS are given in Section 9.4.

9.2. Batch Definition: Batch definitions are provided in the Quanterra Quality Control Program (QA-003).

9.3. Method Blank (MB): One method blank must be processed with each batch of 20 or less samples. The method blank consists of reagent water that is carried through the entire analytical procedure, including filtration as applicable. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data.

9.3.1. A reagent water blank is prepared and analyzed with each batch of samples. The volume of reagent water used should be the same as the minimum sample volume used for the test (Section 8.3).

9.3.2. Corrective Action for Blanks

9.3.2.1. If the analyte level in the method blank is ≥ the RL for the analyte of interest in the sample, all associated samples with reportable levels of analyte are reprepared and reanalyzed. If this is not possible due to limited sample quantity or other considerations, the corresponding sample data **must be addressed in the project narrative**. Refer to QA-003 for further details of corrective actions.

9.3.2.2. High blank results indicate contamination. Make sure the glassware and filtering apparatus is clean.

9.3.2.3. Low blanks typically indicate that the balance was not operating correctly or the filter/weighing dish was not completely dry when the initial weights were measured.

9.3.2.4. If there is no analyte greater than the RL in the samples associated with an unacceptable method blank, the data may reported with qualifiers. **Such action must be taken in consultation with the client and must be addressed in the project narrative.**

9.4. Laboratory Control Sample (LCS): One LCS must be processed with each batch of 20 or less samples. The LCS must be carried through the entire analytical procedure. The LCS is used to monitor the accuracy of the analytical process. On-going monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines. No LCS is available for volatile residue procedures.

9.4.1. An LCS is prepared and analyzed with each analytical batch of samples. Refer to Section 7.2 for instructions on how to prepare the LCS solution. The LCS should be the same volume as the method blank.

9.4.2. Corrective Action for LCS

9.4.2.1. A control limit of 80-120% recovery must be applied until in-house control limits are established. If the result is outside

established control limits, the system is out of control and corrective action must occur.

9.4.2.2. Corrective action will include repreparation and reanalysis of the batch unless the client agrees that another corrective action is acceptable. If this is not possible due to limited sample quantity or other considerations, the corresponding sample data **must be addressed in the project narrative**.

9.4.2.3. Poor LCS results are usually the result of contamination or poor mixing.

9.5. A Matrix Spike/Matrix Spike Duplicate (MS/MSD or MS/SD) pair must be extracted with every process batch of similar matrix, not to exceed twenty (20) samples. An MS/MSD pair are aliquots of a selected field sample spiked with analytes of known identity and concentration. The MS/MSD pair must be processed in the same manner and at the same time as the associated samples. Spiked analytes with recoveries or precision outside control limits must be within control limits in the LCS. Corrective actions must be documented on a Non-Conformance memo, then implemented when recoveries of any spike analyte is outside control limits provided on the LIMS or by the client. Re-extraction of the blank, an LCS, the selected field sample, the MS/MSD may be required after evaluation and review.

9.6. A duplicate control sample (LCSD or DCS) must be substituted when insufficient sample volume is provided to process an MS/MSD pair. The LCSD is evaluated in the same manner as the LCS. See Quanterra Inc. QA Policy 003 for specific acceptance criteria. Sample results should agree within 20% if both the sample and sample duplicate results are > 5x RL.

10. CALIBRATION AND STANDARDIZATION

10.1. Since this method is based on gravimetric techniques, there is no calibration in the usual sense. Proper balance operation will be verified daily or prior to sample analysis by following the lab-specific balance calibration SOP. Analytical balance calibration must be performed daily (every 24 hours).

10.2. Oven temperature must be checked daily and recorded either on the benchsheet or in an oven temperature logbook.

10.3. Conductivity of the water must be monitored and recorded in the Conductivity Logbook daily. The maximum permissible conductivity is 1.0 umhos/cm (at 25

°C). If the conductivity reading on the water system exceeds this level, do not use the water for these procedures and notify the supervisor immediately.

11. PROCEDURE

11.1. One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and must be approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

11.2. Any unauthorized deviations from this procedure must also be documented as a nonconformance with a cause and corrective action described.

11.3. All samples are to be checked out of sample control with the chain of custody documentation filled out completely.

11.4. Proper sample identification is extremely important in any analytical procedure. Labeling of evaporating dishes and filters holders must be done in a manner to ensure connection with the proper sample.

11.5. If possible, analyze all the samples of a project at the same time to minimize the QC required and streamline the flow of the project through the lab and reporting group.

11.6. Non-representative particulates such as leaves, sticks, fish, and lumps of fecal matter should be excluded from the sample if it is determined that their inclusion is not desired in the final result. The presence/removal of these artifacts should be noted on the benchsheet.

11.7. If samples are visibly oily, this should be noted on the benchsheet.

11.8. If there is limited sample volume or high solids content, smaller amounts of sample may need to be processed than detailed in the following sections. This occurrence must be noted on the benchsheet and reporting limits must be adjusted appropriately.

11.9. Proceed to the appropriate section for the desired method as follows:

Total Solids	11.10
Total Dissolved Solids	11.11
Total Suspended Solids	11.12
Volatile Solids	11.13
Volatile Dissolved Solids	11.14
Volatile Suspended Solids	11.15

11.10. Total Solids

11.10.1. Preparation of Evaporating Dish

11.10.1.1. If only total solids are to be measured, heat clean dish to 103-105 °C for one hour. If volatile solids are to be measured in addition to total solids, ignite the clean evaporating dish at 550 °C for one hour in a muffle furnace.

11.10.1.2. Remove the dish from the muffle furnace using tongs and heat resistant gloves.

11.10.1.3. Cool and store dish in desiccator until dish reaches room temperature or until needed.

11.10.1.4. Weigh immediately before use to the nearest 0.1 mg. Record the weight on the benchsheet.

11.10.2. Sample Analysis

11.10.2.1. Transfer a measured aliquot of well-mixed sample to the pre-weighed, labeled dish. Record the volume of sample (to the nearest mL) on the benchsheet. All benchsheet entries are entered into the Total Solids and Total Dissolved Solids Benchsheet Software program.

11.10.2.1.1. Choose an aliquot of sample sufficient to contain a residue of at least 25 mg but less than 200 mg. To obtain a weighable residue, successive aliquots of sample may be added to the same dish after evaporation.

11.10.2.1.2. If the sample is known to contain > 2000 mg/L dissolved solids, it should be diluted. Prescreening may be performed using a conductivity meter to determine the required sample volume or dilution. For this purpose, the following formula should be used to approximate the required sample volume:

(2000 x 100) / Conductivity = mL sample to use

Note: To avoid "oddball" reporting limits, round the determined sample volume to one of the following volume increments: 5, 10, 20, 25, 50, or 100 mL.

11.10.2.1.3. For the LCS, measure 100 mL of the LCS Solution (Section 7.2) and pour into the dish.

11.10.2.1.4. For the MB, measure 100 mL of reagent water and pour into the dish.

11.10.2.2. Evaporate the sample to dryness on a steam bath or in a drying oven. If evaporation is performed in a drying oven, the temperature should be lowered as necessary to prevent boiling and splattering of the sample.

11.10.2.3. Dry the evaporated sample for at least one hour at 103-105 °C.

11.10.2.4. Record the date, time, and oven temperature on the benchsheet when the samples are initially placed in the oven and again when they are removed from the oven.

11.10.2.5. Using tongs, remove the weighing dish from the oven and place in a desiccator and cool to room temperature.

11.10.2.6. Weigh the dish to the nearest 0.1 mg. Record the weight on the benchsheet. All benchsheet entries are entered into the Total Solids and Total Dissolved Solids Benchsheet Software program.

11.10.2.7. Return the samples to the oven for another hour, cool in a desiccator, and reweigh. Repeat the drying, cooling, desiccating, and weighing cycle until a constant weight is obtained or weight loss is less than 1.0 mg. If more than three cycles are required, prepare a Nonconformance Memo.

Note: When weighing dried sample, be alert to change in weight due to air exposure and/or sample degradation.

11.10.2.8. If volatile solids are to be determined, treat the residue according to Section 11.13.

11.10.2.9. Calculate results according to the equation provided in Section 12.1. All benchsheet entries are entered into the Total Solids and Total Dissolved Solids Benchsheet Software program. The software program completes all calculations. Use the lowest final weight achieved for calculating TS.

11.11. Total Dissolved Solids

Measure and record the conductance of each sample before preparation and analysis for TDS. Conductance multiplied by a factor between 0.55 and 0.81 gives a rough approximation of TDS. See calculations in Section 12.3. The measured TDS should be within the range of the expected TDS result calculated from conductance. If the measured TDS is not within the expected TDS range calculated from conductance, follow the corrective action protocol in Figure 1.

11.11.1. Preparation of Glass Fiber Filter Disc: Refer to Section 17.1.2.

11.11.2. Preparation of Evaporating Dishes

11.11.2.1. If only total dissolved solids are to be measured, heat clean dish to 180 ± 2 °C for one hour. If volatile dissolved solids are to be measured in addition to TDS, ignite the clean evaporating dish at 550 ± 50 °C for one hour in a muffle furnace.

11.11.2.2. Heat resistant gloves and tongs must be used when removing items from the muffle furnace.

11.11.2.3. Store and cool dish in desiccator until dish reaches room temperature or until needed.

Note: Always transfer the dish with gloves or tongs to prevent added weight due to oil from fingerprints.

11.11.2.4. Weigh immediately before use to the nearest 0.1 mg. All benchsheet entries are entered into the Total Solids and Total Dissolved Solids Benchsheet Software program.

11.11.3. Sample Analysis

11.11.3.1. Thoroughly rinse the entire filtration apparatus with reagent water before filtering each sample.

11.11.3.2. Assemble the filtering apparatus, place a glass fiber filter in the apparatus, pre-wet the filter using reagent water, and begin suction.

Note: If the sample also requires TSS, preweigh the prepared filter and refer to Section 11.12 for additional guidance.

11.11.3.3. Shake the sample vigorously and rapidly transfer 100 mL (or a smaller portion which will yield between 10 and 200 mg dried residue) to the funnel by means of a graduated cylinder. If total dissolved solids content is low, a larger volume may be filtered. If more than 10 minutes are required to complete filtration, decrease sample size.

Note: Multiple filters may be used if performing only TDS analysis.

11.11.3.3.1. The conductance of each sample may be used to determine the appropriate sample volume to process.

Note: TDS is typically 55-90% of the conductance result. The exact relationship depends on the compounds present in the samples and may not hold for very high concentrations or samples containing non-ionic species or samples with conductances

greater than 10,000 umho/cm or less than 50 umho/cm.

11.11.3.3.1.1. If the sample has a conductance less than 3,000 umhos/cm, 100 mL should be used.

11.11.3.3.1.2. If the conductance is 3,000 to 10,000 umhos/cm, use 20 mL of sample.

11.11.3.3.1.3. If the conductance is extremely high (> 10,000 umhos/cm) smaller volumes may be used.

11.11.3.3.2. All benchsheet entries are entered into the Total Solids and Total Dissolved Solids Benchsheet Software program.

11.11.3.3.3. For the method blank, process 100 mL of reagent water as the sample.

11.11.3.3.4. For the LCS, process 100 mL of the LCS Solution. Refer to Section 7.2 for instructions on how to prepare the LCS.

11.11.3.4. Filter the sample through the glass fiber filter.

11.11.3.5. Rinse the graduated cylinder, funnel walls, and filter with three successive 10 mL portions of reagent water and allow for complete drainage between washings. Continue to apply vacuum for about three minutes after filtration is complete to remove as much water as possible.

11.11.3.6. Transfer the filtrate (including the washings) to a pre-weighed evaporating dish. Rinse the receiving flask with 10-25 mL of reagent water and transfer washings into the dish to ensure complete transfer of the sample.

11.11.3.7. Evaporate the samples to dryness on a steam bath or in an oven set slightly below boiling. If filtrate volume exceeds

dish capacity, add successive portions to the same dish after evaporation.

11.11.3.8. Dry the evaporated sample in an oven for at least one hour at 180 ± 2 °C.

11.11.3.9. Record the date, time, and oven temperature on the benchsheet when the samples are initially placed in the oven and again when they are removed from the oven.

11.11.3.10. Using tongs, remove the weighing dish from the oven and place in a desiccator and cool to room temperature.

11.11.3.11. Weigh the dish to the nearest 0.1 mg. All benchsheet entries are entered into the Total Solids and Total Dissolved Solids Benchsheet Software program.

11.11.3.12. Return the samples to the oven for another hour, cool in a desiccator, and reweigh. Repeat the drying, cooling, desiccating and weighing cycle until a constant weight is obtained or weight loss is less than 1.0 mg. If more than three cycles are required, prepare a Nonconformance Memo.

11.11.3.13. Calculate results according to the equation in Section 12.2. All benchsheet entries are entered into the Total Solids and Total Dissolved Solids Benchsheet Software program. The software program completes all calculations. Use the lowest final weight achieved for calculating TDS.

11.12. Total Suspended Solids

11.12.1. Preparation of Glass Fiber Filter Disc

Note: As an alternative to the steps outlined in Sections 11.12.1.1 through 11.12.1.7, prepared and pre-weighed filters may be purchased for use under this method.

11.12.1.1. Place the glass fiber filter discs, one at a time, on the membrane filter apparatus or insert into bottom of a suitable Gooch crucible with wrinkled surface up.

11.12.1.2. While vacuum is applied, wash the disc with three successive 20 mL volumes of distilled water.

11.12.1.3. Remove all traces of water by continuing to apply vacuum after water has passed through and discard washings.

11.12.1.4. Remove filter from membrane filter apparatus and place in a labeled, glass weighing dish (or remove both the crucible and filter if a Gooch crucible is used), and dry in an oven at 103-105 °C for one hour.

Note: If volatile suspended solids (VSS) are to be determined as well as total suspended solids, the filter must be heated at 550 °C for one hour, instead of 103-105 °C.

11.12.1.5. Using tongs, remove the weighing dish from the oven and place in a desiccator and cool to room temperature.

11.12.1.6. Weigh the cooled filter using an analytical balance to the nearest 0.1 mg. Handle the filters or crucible/filter only with forceps. All benchsheet entries are entered into the Total Solids and Total Dissolved Solids Benchsheet Software program.

11.12.1.7. Repeat the drying, cooling, and weighing cycle until a constant weight is obtained or weight loss is less than 1.0 mg.

11.12.2. Selection of Sample Volume

11.12.2.1. For a 47 mm diameter filter, filter 100 mL of sample.

11.12.2.2. If during filtration of this initial volume, the filtration rate drops rapidly or if filtration time exceeds 5-10 minutes, a smaller volume of sample should be processed.

Note: If the sample appears high in TSS, start with a sample volume < 100 mL.

11.12.3. Sample Analysis

11.12.3.1. Assemble the filtering apparatus, place the pre-weighed glass fiber filter in the apparatus, pre-wet the filter using reagent water and begin suction.

Note: Handle the filters or crucible/filter only with forceps.

11.12.3.2. Shake the sample vigorously and quantitatively transfer 100 mL (or an appropriate aliquot) of the sample to the filter using a graduated cylinder or pipet. A smaller amount should be filtered if the sample is high in TSS or is otherwise slow to filter. Filter 25 mL at a time until filtration slows. Record the volume of sample filtered (to the nearest mL) on the benchsheet.

Note: If Total Dissolved Solids (TDS) is also required, the filtrate may be used. Refer to Section 11.11 for additional guidance.

11.12.3.3. Remove all traces of water by continuing to apply vacuum after the sample has passed through.

11.12.3.4. With suction on, rinse the graduated cylinder, filter, suspended solids residue, and filter funnel wall with three 10 mL portions of reagent water allowing complete drainage between washings.

11.12.3.5. Remove all traces of water by continuing to apply vacuum for about three minutes after the sample has passed through.

11.12.3.6. Carefully remove the filter from the filter support and transfer to an glass weighing dish. Alternatively, remove the crucible and filter from the crucible adapter.

11.12.3.7. Dry the filter at least one hour at 103-105 °C.

11.12.3.8. Record the date, time, and oven temperature on the benchsheet when the samples are initially placed in the oven and again when they are removed from the oven.

11.12.3.9. Using tongs, remove the weighing dish from the oven and place in a desiccator and cool to room temperature.

11.12.3.10. Cool the samples in a desiccator, weigh (to the nearest 0.1 mg). Enter weight into the Total Solids and Total Dissolved Solids Benchsheet Software program.

11.12.3.11. Return the samples to the oven for another hour, cool in a desiccator, and reweigh. Repeat the drying, cooling, desiccating, and weighing cycle until a constant weight is obtained or weight loss is less than 1.0 mg. If more than three cycles are required, prepare a Nonconformance Memo.

11.12.3.12. If volatile suspended solids are to be determined, treat the residue according to Section 11.15.

11.12.3.13. Calculate the results using the formula given in Section 12.3. All benchsheet entries are entered into the Total Solids and Total Dissolved Solids Benchsheet Software program. Use the lowest final weight achieved for calculating TSS.

11.13. Volatile Solids

11.13.1. Heat muffle furnace up to temperature (550 ± 50 °C).

11.13.2. Place evaporating dish containing residue generated by Total Solids protocol (Section 11.10) in muffle furnace to ignite the residue.

11.13.3. Record the date, time, and oven temperature on the benchsheet when the samples are initially placed in the oven and again when they are removed from the oven.

11.13.4. Typically, 15-20 minutes ignition are required for 200 mg of residue. However, more than one sample and/or heavier residues may necessitate longer ignition times.

11.13.5. Let dish cool partially in air until most of the heat has dissipated before transferring to a desiccator for final cooling.

11.13.6. Using tongs and heat resistant gloves, remove the weighing dish from the muffle furnace and place in a desiccator and cool to room temperature.

11.13.7. Weigh dish as soon as it has cooled to room temperature.

11.13.8. Return the samples to the oven for another hour, cool in a desiccator and reweigh. Repeat the drying, cooling, desiccating, and weighing cycle until a constant weight is obtained or weight loss is less than 1.0 mg. If more than three cycles are required, prepare a Nonconformance Memo.

11.13.9. Calculate the results using the formula given in Section 12.4. Use the lowest final weight achieved for calculating VS.

11.14. Volatile Dissolved Solids

11.14.1. Heat muffle furnace up to temperature (550 ± 50 °C).

11.14.2. Place evaporating dish containing residue generated by Total Dissolved Solids protocol (Section 11.3) in muffle furnace to ignite the residue.

11.14.3. Record the date, time, and oven temperature on the benchsheet when the samples are initially placed in the oven and again when they are removed from the oven.

11.14.4. Typically, 15-20 minutes ignition are required for 200 mg of residue. However, more than one sample and/or heavier residues may necessitate longer ignition times.

11.14.5. Let dish cool partially in air until most of the heat has dissipated before transferring to a desiccator for final cooling.

11.14.6. Using tongs and heat resistant gloves, remove the weighing dish from the muffle furnace and place in a desiccator and cool to room temperature.

11.14.7. Weigh dish as soon as it has cooled to room temperature.

11.14.8. Return the samples to the oven for another hour, cool in a desiccator and reweigh. Repeat the drying, cooling, desiccating and weighing cycle until a constant weight is obtained or weight loss is less than 1.0 mg. If more than three cycles are required, prepare a Nonconformance Memo.

11.14.9. Calculate the results using the formula given in Section 12.5. Use the lowest final weight achieved for calculating VDS.

11.15. Volatile Suspended Solids

11.15.1. Heat muffle furnace up to temperature (550 ± 50 °C).

11.15.2. Place glass fiber filter disc containing residue generated by Total Suspended Solids protocol (Section 11.12) in muffle furnace to ignite the residue.

11.15.3. Record the date, time, and oven temperature on the benchsheet when the samples are initially placed in the oven and again when they are removed from the oven.

11.15.4. Typically, 15-20 minutes ignition are required for 200 mg of residue. However, more than one sample and/or heavier residues may necessitate longer ignition times.

11.15.5. Let filter cool partially in air until most of the heat has dissipated before transferring to a desiccator for final cooling.

11.15.6. Using tongs and heat resistant gloves, remove the weighing dish from the muffle furnace and place in a desiccator and cool to room temperature.

11.15.7. Weigh filter as soon as it has cooled to room temperature.

11.15.8. Return the samples to the oven for another hour, cool in a desiccator, and reweigh. Repeat the drying, cooling, desiccating, and weighing cycle until a constant weight is obtained or weight loss is less than 1.0 mg. If more than three cycles are required, prepare a Nonconformance Memo.

11.15.9. Calculate the results using the formula given in Section 12.6. Use the lowest final weight achieved for calculating VSS.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Calculate **Total Solids** as follows:

$$\text{Total Solids, mg / L} = \frac{(A - B) \times 1000}{C}$$

where: A = weight of dried residue + dish (mg)
B = weight of dish (mg)
C = volume of sample (mL)

12.2. Calculate **Total Dissolved Solids** as follows:

$$\text{Total Dissolved Solids, mg / L} = \frac{(A - B) \times 1000}{C}$$

where: A = weight of dried residue + dish (mg)
B = weight of dish (mg)
C = volume of sample (mL)

12.3. Calculate **Total Suspended Solids** as follows:

$$\text{Total Suspended Solids, mg / L} = \frac{(A - B) \times 1000}{C}$$

where: A = weight of filter + residue (mg)
B = weight of filter (mg)
C = volume of sample filtered (mL)

Calculate the expected TDS range from conductance (as umho/cm).

Expected TDS_{low} (mg/L) = (Conductance x 0.55)

Expected TDS_{high} (mg/L) = (Conductance x 0.81)

12.4. Calculate **Volatile Solids** as follows:

$$\text{Volatile Solids, mg / L} = \frac{(A - B) \times 1000}{C}$$

where: A = weight of residue + dish before ignition (mg)
B = weight of residue + dish after ignition (mg)
C = volume of sample (mL)

12.5. Calculate **Volatile Dissolved Solids** as follows:

$$\text{Volatile Dissolved Solids, mg / L} = \frac{(A - B) \times 1000}{C}$$

where: A = weight of residue + dish before ignition (mg)
B = weight of residue + dish after ignition (mg)
C = volume of sample (mL)

12.6. Calculate **Volatile Suspended Solids** as follows:

$$\text{Volatile Suspended Solids, mg / L} = \frac{(A - B) \times 1000}{C}$$

where: A = weight of residue + filter before ignition (mg)
B = weight of residue + filter after ignition (mg)
C = volume of sample (mL)

12.7. The relative percent difference (RPD) of duplicates are calculated according to the following equation:

$$RPD = 100\left[\frac{|DU1 - DU2|}{\left(\frac{DU1 + DU2}{2}\right)}\right]$$

Where: DU1 = Sample result
DU2 = Sample duplicate result

12.8. If multiple weighing cycles are required, the lowest final sample weight is used for calculating solids content.

12.9. If smaller or larger sample volumes are processed than are specified in the method, the reporting limit must be adjusted accordingly.

12.10. Sample results should be reported according to the following significant figure rules:

Significant Figures	Sample Result
2	< 10
3	≥ 10

13. METHOD PERFORMANCE

13.1. The reporting limit is 10 mg/L for TS, TDS, VS, VDS, and VSS.

13.2. The reporting limit is 5 mg/L for TSS.

13.3. The initial demonstration study as detailed in Section 9.1.2 must be acceptable before the analysis of field samples under this SOP may begin.

13.4. The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required expertise.

14. POLLUTION PREVENTION

14.1. No solvents of any kind or in any amount are to be disposed of in the sinks or evaporated in the hoods.

15. WASTE MANAGEMENT

15.1. Waste management in the procedure must be segregated and disposed according to the facility hazardous waste procedures. The Environmental Health and Safety Coordinator or Hazardous Materials Technician should be contacted if additional information is required.

15.2. All waste must be disposed of according to the facility hazardous waste management procedures, Attachment C of the Chemical Hygiene Plan, Section WS002, Table 1.

15.3. Samples and other solutions containing high concentrations of toxic materials must be disposed of according to the facility hazardous waste management procedures, Attachment C of the Chemical Hygiene Plan, Section WS003, Disposal of Samples After Analysis.

16. REFERENCES

16.1. Methods for the Chemical Analysis of Water and Wastes, EPA-600/4-79-020, March 1979: Methods 160.1, 160.2, 160.3, and 160.4.

16.2. Standard Methods for the Examination of Water and Wastewater, 18th Edition, 1992: Methods 2540A, 2540B, 2540C, 2540D, and 2540E.

16.3. QA Policy QA-003, Quanterra QC Program.

16.4. QA Policy QA-004, Rounding and Significant Figures.

16.5. QA Policy QA-005, Method Detection Limits.

16.6. SAC-QA-0041 Calibration and Calibration Check of Balances, Revision 1.1.

16.7. LP-CAL-0001 Laboratory Documentation Practices, Revision 3.0.

16.8. QA Policy 003 Quanterra Quality Control Program, Revision 1.

16.9. QA Policy 008 Data Recording Requirements, Rev. 0.

16.10. Chemical Hygiene Plan, Attachment C, Hazardous Waste Management Procedures, Revision 0.

17. MISCELLANEOUS (TABLES, APPENDICES, ETC.)

17.1. Modifications/Interpretations From Reference Method

17.1.1. Samples are not taken through the drying, cooling, and weighing cycle more than three times. If the sample fails to reach constant weight within three cycles, this is documented in a Nonconformance Memo and the lowest final weight is used for the calculation.

17.1.2. The filter cleaning procedure outlined in Method 160.1 is not applied. This SOP requires the use of method blanks to document system cleanliness.

17.2. Modifications From Previous SOP

17.2.1. Constant weight shall be determined after weight loss is less than 1.0 mg rather than 0.5 mg as specified in Sections 11.10.2.7 and 11.11.3.12.

17.2.2. All entries shall be entered into the TDS and TS Benchsheet Software program. Software program completes all calculations.

17.2.3. Section 7.2 includes LCS solutions used at Quanterra, West Sacramento. Sections 7.3 and 9.6 describe the spike solutions and quality control for matrix spikes.

17.2.4. Sections 8.3, 13.2, 11.12.2.1, and 11.12.3.2 describe the 100 mL sample used for TSS rather than 250 mL used in previous SOP and raises the Reporting Limit to 5 ppm, rather than 4 ppm.

17.2.5. TSS filters are placed in a glass weighing dish rather than an aluminum one in Sections 11.12.1.4 and 11.12.3.6.

17.2.6. Figure 1: Decision Tree for Failed EC/TDS Ratio has been included.

Figure 1: Decision Tree for Failed EC/TDS Ratio

STEP 1: Investigate the physical properties of the sample.

1. Take good notes on your benchsheet during pouring and after drying.

   1.1. Floatables, sediment content, viscosity, color, reaction to air exposure, smell, etc. are enough to cause a failed EC/TDS ratio.

   1.2. What happened during the process? Did the sample spill, splatter, too much sediment?

      1.2.1. If analyst error occurs, stop and reanalyze immediately.

   1.3. What is the sample's appearance after drying? Hygroscopic crystals apparent? Hard to dry? Ring around crucible? Fine sediment persistent?

   NOTE: For samples high in sediment, do not shake the samples prior to analysis.

STEP 2: Always check initial EC, when in doubt reanalyze for EC.

1. If EC is < 20 umhos/cm with TDS of < 100 ppm: Report result with an anomaly stating the EC value is <100 umhos/cm and therefore the EC/TDS ratio criteria does not apply.

2. If EC is between 20 umhos/cm and 100 umhos/cm with TDS < 150% of EC: Report result with an anomaly stating the EC value is <100 umhos/cm and therefore the EC/TDS ratio criteria does not apply.

3. If EC is between 20 umhos/cm and 100 umhos/cm with TDS > 150% of EC: Reanalyze TDS.

   3.1. If reanalysis confirms the original TDS result, report the original run with documentation on both datasheets that reanalysis confirms TDS results with failed EC/TDS ratio.

   3.2. If reanalysis does not confirm the original TDS result, and the rerun analysis passes EC/TDS criteria, report the rerun. If holding times were violated, file an anomaly stating the internal QC failed in the original run.

4. If EC is > 20,000 umhos/cm (oversaturated), dilute the sample at 10x and rerun for EC. Recalculate EC/TDS ratio using the rerun results for EC.

   4.1. If new ratio passes, report results with documentation in the data that the sample was diluted and rerun for EC due to high EC and possibly saturation of salts.

   4.2. If new ratio still fails, rerun TDS.

      4.2.1. If reanalysis confirms the original TDS result, report the original run with notes on both datasheets that reanalysis confirms TDS results with failed EC/TDS ratio.

4.2.2. If reanalysis does not confirm the original TDS result, and the rerun analysis passes EC/TDS criteria, report the rerun. If holding times were violated, file an anomaly stating the internal QC failed in the original run.

5. For failed TDS with EC > 100 umhos/cm, but < 20,000 umhos/cm, rerun EC first before reanalyzing TDS. Keep TDS holding times (7 days) in mind.

5.1. If rerun EC confirms original, and EC/TDS ratio still fails, rerun TDS.

5.2. If rerun EC > 20% RPD from the original EC result, and EC/TDS ratio passes, report TDS result and record new EC result.

5.3. If EC/TDS still fails, rerun TDS.

STEP 3: Only when the EC evaluations and reanalyses are complete should you determine whether to reanalyze for TDS.

STEP 4: If none of the above scenarios apply and you need additional guidance, see your supervisor.

17.3. Tables

Table 1: Summary of Quality Control Requirements for Solids Determinations

QC Samples	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	1 per batch of up to 20 samples	< ± RL	Reanalyze all samples associated with unacceptable MB
Laboratory Control Sample (LCS)	1 per batch of up to 20 samples	80 - 120 % until in-house limits established	Reanalyze all samples associated with unacceptable LCS
Matrix Spikes (MS/MSD)	1 pair per batch of up to 20 samples	75-125% , RPD ≤ 20%	Spiked analytes must be within control limits in the LCS. Re-extraction of the blank, an LCS, the selected field sample, the MS/MSD may be required after evaluation and review.
Duplicate (D)	1 per batch of up to 20 samples, substituted when insufficient sample volume is provided to process an MS/MSD pair	≤ 20% RPD when results > 5x RL	Narrate the results

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STANDARD OPERATING PROCEDURE

Subject or Title: Chemical Oxygen Demand (Method 410.4)		Page 1 of 6
SOP No.: LM-CAL-1019	Revision No.: Original	Effective Date: March 19, 1990
Supersedes: None		

1. SCOPE AND APPLICATION

1.1 Analytes

This method measures the oxygen equivalent of materials present in water that are subject to oxidation by a strong chemical oxidant.

1.2 Reporting Limit

The reporting limit is 5 mg/L.

1.3 Applicable Matrices

This method is applicable to surface waters, as well as industrial and domestic wastewaters.

1.4 Dynamic Range

This method covers the ranges between 5 and 1500 mg/L COD. Higher concentrations can be determined by dilution.

1.5 Analysis Time

The analytical time is approximately 4 hours for a batch of 20 samples.

Prepared By: John Disney	Date: 3/16/90
Management Approval:	Date: 3/19/90
QA Officer Approval:	Date: 3/19/90

2. SUMMARY OF METHOD

2.1 Organic and oxidizable inorganic substances in an aqueous sample are digested and oxidized by potassium dichromate in 50% sulfuric acid in a sealed vial at 150°C. After two hours of digestion, tubes are removed from the digestor, cooled and measured colorimetrically at both high and low level ranges. The measurement is an indication of excess dichromate remaining in the vial after digestion.

2.2 The low level curve ranges from 5 to 150 mg/L and is read at 420 nm. The high level curve ranges from 100 to 1500 mg/L and is read at 620 nm wavelength. Different Hach COD vials are used for each level.

3. COMMENTS

3.1 Interferences

3.1.1 Low levels of organic matter contamination in water or on glassware may give bad results.

3.1.2 Volatile materials may be lost during sampling and storage. Samples should completely fill a glass bottle and be preserved to pH <2 with H_2SO_4.

3.1.3 Chloride may give false positive results. Mercuric sulfate in the Hach vials will prevent interference of up to 2000 ppm chloride. If chloride is higher than 2000 ppm, dilutions must be made to reduce this interference.

3.2 Helpful Hints

3.2.1 This method is based on the reduction of Cr (VI) to Cr (III). Readings at 420 nm represent the reduction of Cr (VI) in the low curve. The high curve reads at 620 nm to represent the increase in Cr (III).

3.2.2 COD is related to TOC and BOD. For rough comparison purposes, COD = 1.6 x BOD; BOD = 1.6 x TOC.

3.2.3 Some inorganic compounds may contribute to COD thus indicating a higher level of organic contamination.

4. SAFETY ISSUES

4.1 All Cal employees are expected to follow the Enseco GGC policy. Lab coats, gloves, and glasses are required when working in the lab.

4.2 COD vials contain sulfuric acid as well as salts of mercury, silver, and dichromate. Consult MSDS and handle with extreme care. Return used tubes to the original containers so they can be sent off-site for disposal.

4.3 Run analysis in a fume hood.

4.4 When mixing samples in the vials, wrap paper towels around the vials to avoid burns and possible breakage.

5. SAMPLE COLLECTION AND PRESERVATION

5.1 Samples should be collected in glass bottles, preserved to pH less than 2 and stored at 4°C. Minimize head space.

5.2 Holding time for COD is 28 days.

6. APPARATUS

6.1 Hach block digestor for 16 mm tubes set at 150°C ± 2°C.

6.2 Spectrophotometer to measure 16 mm tubes at 420 and 620 nm.

7. REAGENTS AND STANDARDS

7.1 Premixed Hach low range COD tubes #21258-15.

7.2 Premixed Hach high range + COD tubes #21259-15.

7.3 Deionized water which contains less than 0.3 mg/L organic carbon. Do not store water in plastic bottles. Distilled water may be used.

7.4 1500 ppm COD stock standard. Dry potassium phthalate (KHP) and desiccate. Use 1.275 g to 1000 mL and preserve with 2 mL H_2SO_4.

7.5 1000 ppm COD stock standard. Use 0.85 g KHP to 1 liter with 2 mL H_2SO_4.

7.6 COD working standards. Dilute stock solutions in volumetrics as follows:

Concentration (mg/L)	Volume of Stock (mL)	Final Volume (mL)
0	0	100
5	5 mL 100 ppm	100
10	10 mL 100 ppm	100
50	50 mL 100 ppm	100
100	100 mL 100 ppm	100
150	15 mL 1000 ppm	100
500	50 mL 1000 ppm	100
1000	100 mL 1000 ppm	100
1500	100 mL 1500 ppm	100

7.7 Reference Solution

Use ERA demand sample diluted according to instructions supplied with the vial.

8. PROCEDURE

8.1 Preheat block digestor to 150°C.

8.2 Prepare both a high range curve consisting of a blank, 100, 150, 500, 1000, and 1500 mg/L standards and a low range curve consisting of a blank, 5.0, 10, 50, 100, and 150 mg/L. Mid-range ERA reference standards are to be run on both ranges.

8.3 Pipet 2.0 mL of standard/sample into each vial (one low range, one high range). Replace cap tightly, wrap in paper towel and shake vigorously. Place vials in digestor block for 2 hours.

8.4 Turn on spectrophotometer and allow 20 minutes for warm-up.

8.5 Remove vials to a cooling rack for approximately 20 minutes.

8.6 Read absorbance for the low range vials at 420 nm and high range vials at 620 nm.

8.7 Record absorbances to 0.001 units. Perform standard checks every 10 samples for each curve.

9. QA/QC REQUIREMENTS

9.1 A blank is required for each curve and must accompany no more than 20 samples.

9.2 Duplicate Control Samples (DCS) are required for every 20 samples. Single Control Samples (SCS) are required for every batch which doesn't include DCS. Separate DCS/SCS are prepared for each curve.

9.3 A blank and standard check must bracket every run and be after every 10 samples.

9.4 If QC spikes are required, use a spike concentration of 100 mg/L.

9.5 Acceptance Criteria

9.5.1 Blanks must fall below the reporting limit (5 mg/L).

9.5.2 DCS and SCS recoveries must fall within 85-115%.

9.5.3 Relative Percent Difference (RPD) of the DCS must be less than 10 for the high curve and less than 15 for the low curve.

9.5.4 Standard checks must be within 10%.

9.5.5 Calibration standards must give a correlation coefficient of ± 0.995 or better.

10. CALCULATIONS

10.1 Enter concentrations and absorbances of the standards into a linear least squares program. Notice the slope of the low range curve is negative while that of the high range curve is positive.

10.2 Calculate sample concentrations from the calibration curves and multiply by dilution factors.

10.3 Note that negative absorbances on the low range curve indicate an over-range reading. Therefore the high range reading should be used.

11. REPORTING

11.1 Results are reported in mg/L.

11.2 For all dilutions, the reporting limit is multiplied by the dilution factor.

11.3 Use 3 significant figures for values of 10 or greater. Report all DCS and SCS with 3 significant figures.

11.4 Document all non-standard procedures such as odd dilutions, interference problems, etc.

12. REFERENCES

12.1 Sources

12.1.1 EPA Method 410.4.

12.1.2 The Hach Corporation recommended method.

SOP FOR BIOCHEMICAL OXYGEN DEMAND (BOD)

SCOPE: This SOP covers determination of Biochemical Oxygen Demand (BOD) in samples. BOD is the amount of dissolved oxygen required by bacteria to consume biodegradable material over a period of five days at 20 degrees C. Several dilutions of a sample are prepared with nutrient-rich dilution water. The initial dissolved oxygen is measured with an electrode. The samples are then incubated at 20 degrees C for five days. The final dissolved oxygen is measured by electrode and the BOD is calculated.

REFERENCES: EPA 405.1

LIMS PRODUCT NAME: BOD
LINKED SPREADSHEETS: NONE
REPORTING LIMIT: 5.0 mg/L

QC REQUIREMENTS: Two glucose/glutamic acid blank spikes (LCS's) are prepared and reported with each batch. A seeded blank is prepared with each batch. Final DO must be at least 1 mg/L for a dilution to be used in reporting. Oxygen depletion must be at least 1 mg/L for a dilution to be used in reporting. A duplicate sample must be run with each batch and the %RPD must be less than 10%. The temperature of the incubator must be maintained at 20 degrees C +/- 1 degree. A seed control blank must be analyzed with each batch.

SAFETY: Wear protective gloves when preparing the sample. Avoid skin and eye contact with all samples.

APPARATUS:
1. 300 mL BOD bottles with pointed ground-glass stoppers.
2. Racks for transporting BOD bottles.
3. Incubator, at 20 degrees C +/- 1 degree.
4. Class A volumetric pipets.
5. Volumetric glassware.
6. Magnetic stirrer with stir bars.
7. Graduated cylinders
8. 19-liter plastic container for dilution water.
9. Tygon tubing for siphon.
10. Aerator with pump
11. pH meter
12. Dissolved Oxygen electrode
13. 500 mL Erlenmeyer flasks.

14. Buret for sodium thiosulfate titrant.
15. Hach DPD test kit for chlorine residual determination.

REAGENTS:

1. Milli-Q water
2. Nutrient buffer pillows for BOD, commercially available from HACH; add contents of one pillow to 19 liters of Milli-Q water.
3. Glucose/glutamic acid solution:
   Dry reagent-grade glucose and glutamic acid in the 105 degree oven for one hour. Dissolve 0.15 g glucose and 0.15 g glutamic acid in deionized water and dilute to 1 L in a volumetric flask. Mix well. Store in a refrigerator.
4. Seed:
   BOD Seed Inoculum (Polyseed). Dissolve one capsule in 500 mL of the dilution water. Stir for 1 hour. prepare daily; solution will be usable for only six hours.

PROCEDURE:

1) Dilution water vessel preparation:

a. Fill vessel with hot tap water and a cup of bleach. Put the tygon tubing in the vessel.
b. Let soak for 30 minutes.
c. Empty and rinse thoroughly with hot water. It is extremely important that no trace of bleach remains.
d. Rinse thoroughly with organic free DI water.
e. Perform cleaning once per month or whenever the average dissolved oxygen depletion in the blanks exceeds 0.2.

2) Dilution water preparation:

a. Dispense the Milli-Q water into a 19 liter container,
b. Add contents of nutrient powder pillow.
c. Aerate for 45 minutes.

3) Seed Preparation:

a. Add contents of one polyseed capsule to 500 mL of prepared dilution water.
b. Mix with stir bar and aerate for one hour minimum.

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4) Sample dilution:

a. Determine COD on sample - one hour reaction time is sufficient as a screen for BOD analysis.
b. Determine the pH of the sample. It should lie between 6.5 and 8.0. If it does not, adjust the H with sulfuric acid or sodium hydroxide.
c. check for residual chlorine with potassium iodide/starch test paper, or use Hach DPD test kit, following kit instructions.
d. If residual chlorine is found, treat the sample with a predetermined amount of sodium sulfite solution.
e. Collect enough BOD bottles for the batch. Allow 2 bottles for blank, 1 bottle for a seeded blank, 2 bottles for an LCS, 3 bottles for each sample, and 3 bottles for each sample duplicate.
f. Documentation for benchbook includes: The "date off" should be five days after the "date set". Include columns for the bottle numbers, the sample ID, the initial DO, the sample volume, the depletion, and the BOD (mg/L) for each bottle.
g. Allow the sample to reach room temperature.
h. mix each sample well.
i. Prepare serial dilutions in accordance with COD values, see Appendix A.
j. Use Class A volumetric pipets to deliver difference volumes of each sample to three BOD bottles. The volumes can be selected by referring to historical results. The goal is to obtain oxygen depletions of at least 1 mg/L with a final DO of at least 1 mg/L.
k. Add 25 mL of seed directly to the seed blank.
l. Add 5 mL of the glucose/glutamic acid solution the first LCS and 10 mLs of glucose/glutamic acid solution to the second LCS.
m. Add 2 mL of seed to each of the LCS bottles and each sample bottle. Do not add seed to the blanks.
n. Carefully fill each bottle with the prepare dilution water using a siphon. Let the open end of the siphon extend below the surface of the water in the bottles during filling. Take care not to introduce air bubbles.

4. Determination of initial dissolved oxygen:

a.Connect the dissolved oxygen electrode to the pH meter.

Note: The oxygen electrode nests in a funnel with a built-in stirrer. The funnel provides an overflow reservoir for water displaced when the electrode is inserted in a bottle.

Note: There is a five position switch at the top of the electrode: OFF, BT CK (for battery check), ZERO (for zero calibration), AIR and H2O (to take readings).

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Note: When not in use, the electrode should be stored in a BOD bottle partially filled with DI water. Do not immerse the electrode. Switch the electrode to the "OFF" position.

b. With the electrode switch in the "OFF" position, switch the pH meter to pH mode. The meter will read 7.00.
c. Turn the electrode switch to "BT CK" and verify that the battery reading is 13 or greater. If less than 13, replace battery.
d. Turn the electrode switch to "zero". Use the "zero calibration control" to set the meter to read 0.00.
e. Turn the electrode switch to "AIR". Read out should be 7.60. If not, adjust with knob, wait for stable reading and probe is now calibrated.
f. Turn electrode switch to "H2O".
g. Place the BOD bottles containing the seed blanks, LCS, samples, and replicates on the magnetic stirrer one by one. Carefully insert the funnel and electrode and record the initial DO reading for each in the benchbook.
h. Carefully stopper each bottle. Make sure that the BOD bottles are water sealed and that there are no air bubbles inside. Cover each stopper with a plastic cap and write the "date off" on the cap. The "date off" is five days after today's date. Transport all BOD bottles to the incubator.

5. Determination of final dissolved oxygen:

a. Five days after they were wet, retrieve the BOD bottles from the incubator.
b. Follow meter and probe calibrations as outlined in E.4.a through f.
c. Document in benchbook.
d: Place the BOD bottles containing the seed blanks, LCS samples and replicates on the magnetic stirrer one by one. Carefully insert the funnel and electrode and record the final DO reading for each in the benchbook.

CALCULATION and Data Review:

1. Subtract the final DO from the initial DO for each bottle. Record the results in the "depletion" column in the benchbook

2. Calculate the average initial DO of the regular blanks (DO.AVG). Calculate the average final DO of the seeded blanks (SEED.AVG). Calculate the seed correction factor (SCF) as follows:

$$SCF = \frac{(DO.AVE) - (SEED.AVG)}{5}$$

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3. Calculate the BOD for each bottle as follows:

$$BOD = \frac{(\text{Initial DO}) - (\text{Final DO}) - (\text{SCF})}{\text{Sample volume (in mL)}} \times 300$$

4. The reported BOD for a sample is the average of the BOD's for each sample dilution that satisfies the QC requirements. If only one dilution is satisfactory, its BOD will be the reported BOD.

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QUANTERRA STANDARD OPERATING PROCEDURE

TITLE: GC/MS ANALYSIS BASED ON METHODS 8270C AND 625

(SUPERSEDES: Revision 1)

Prepared by: Richard Burrows

Reviewed by:_____
Technology Standardization Committee

Approved by:_____
Director, Quality Assurance

Approved by:_____
Director, Environmental Health and Safety

Approved by:_____
Management

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SCOPE AND APPLICATION

1.1 This method is based upon SW846 8270C, and is applicable to the determination of the concentration of semivolatile organic compounds in extracts prepared from solid and aqueous matrices. The modifications presented in Attachment A may be followed for analysis of wastewater following method 625. Direct injection of a sample may be used in limited applications. Refer to Tables 1, 2, 3 and 4 for the list of compounds applicable for this method. Note that the compounds are listed in approximate retention time order. Additional compounds may be amenable to this method. If non-standard analytes are required, they must be validated by the procedures described in section 13 before sample analysis.

1.2 The following compounds may require special treatment when being determined by this method:

- Benzidine can be subject to oxidative losses during solvent concentration and exhibits poor chromatography. Neutral extraction should be performed if this compound is expected.
- Hexachlorocyclopentadiene is subject to thermal decomposition in the inlet of the gas chromatograph, chemical reaction in acetone solution, and photochemical decomposition.
- N-Nitrosodiphenylamine decomposes in the gas chromatographic inlet and cannot be distinguished from diphenylamine.
- Pentachlorophenol, 2,4-dinitrophenol, 4-nitrophenol, 4,6-dinitro-2-methylphenol, 4-chloro-3-methylphenol, benzoic acid, 2-nitroaniline, 3-nitroaniline, 4-chloroaniline, and benzyl alcohol are subject to erratic chromatographic behavior, especially if the GC system is contaminated with high boiling material.
- Hexachlorophene is not amenable to analysis by this method.
- 3-Methylphenol cannot be separated from 4-methylphenol by the conditions specified in this method.

1.3 The standard reporting limit (SRL) of this method for determining an individual compound is approximately 0.33 mg/kg (wet weight) for soil/sediment samples, 1 - 200 mg/kg for wastes (dependent on matrix and method of preparation), and 10 µg/L for groundwater samples. Some compounds have higher reporting limits. Refer to Tables 1 and 2 for specific SRLs. Reporting limits will be proportionately higher for sample extracts that require dilution.

2 SUMMARY OF METHOD

2.1 Aqueous samples are extracted with methylene chloride using a separatory funnel, a continuous extractor or Accelerated One-Step™. Solid samples are extracted with methylene chloride / acetone using sonication, soxhlet, accelerated soxhlet or

pressurized fluid extraction. Waste dilution is used for samples that are miscible with the solvent. The extract is dried, concentrated to a volume of 1 mL, and analyzed by GC/MS. Extraction procedures are detailed in SOP# CORP-OP-0001. Qualitative identification of the parameters in the extract is performed using the retention time and the relative abundance of characteristic ions. Quantitative analysis is performed using the internal standard technique with a single characteristic ion.

3 DEFINITIONS

3.1 CCC (Calibration Check Compounds) - A subset of target compounds used tc evaluate the calibration stability of the GC/MS system. A maximum percent deviation of the CCC's is specified for calibration acceptance.

3.2 SPCC (System Performance Check Compounds) - Target compounds designated to monitor chromatographic performance, sensitivity, and compound instability or degradation on active sites. Minimum response factors are specified for acceptable performance.

3.3 Batch - The batch is a set of up to 20 samples of the same matrix processed using the same procedures and reagents within the same time period. The Quality Control batch must contain a matrix spike / spike duplicate (MS/MSD), a Laboratory Control Sample (LCS), and a method blank. In some cases, at client request, the MS/MSD may be replaced with a matrix spike and sample duplicate. Batches are defined at the sample preparation stage. Batches should be kept together through the whole analytical process to the extent possible, but it is not mandatory to analyze prepared extracts on the same instrument or in the same sequence. Refer to the Quanterra QC Program document (QA-003) for further details of the batch definition.

3.4 Method Blank - An analytical control consisting of all reagents, internal standards and surrogate standards, that is carried through the entire analytical procedure. The method blank is used to define the level of laboratory background and reagent contamination.

3.5 LCS (Laboratory Control Sample) - A blank spiked with the parameters of interest that is carried through the entire analytical procedure. Analysis of this sample with acceptable recoveries of the spiked materials demonstrates that the laboratory techniques for this method are acceptable.

3.6 MS (Matrix Spike)- aliquot of a matrix (water or soil) fortified (spiked) with known quantities of specific compounds and subjected to the entire analytical procedure in order to indicate the appropriateness of the method for the matrix by measuring recovery.

3.7 MSD (Matrix Spike Duplicate)- a second aliquot of the same sample as the matrix spike (above) that is spiked in order to determine the precision of the method.

4 INTERFERENCES

4.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Raw GC/MS data from all blanks, samples, and spikes must be evaluated for interferences. If an interference is detected it is necessary to determine if the source of interference is in the preparation and/or cleanup of the samples; then take corrective action to eliminate the problem.

4.2 The use of high purity reagents, solvents, and gases helps to minimize interference problems.

4.3 Matrix interferences may be caused by contaminants that are coextracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature of the sample.

4.4 Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. To reduce carryover, the sample syringe must be rinsed with solvent between samples. Whenever an unusually concentrated sample is encountered, it should be followed by the analysis of solvent to check for cross contamination.

4.5 Phthalate contamination is commonly observed in this analysis and its occurrence should be carefully evaluated as an indicator of a contamination problem in the sample preparation step of the analysis.

5 SAFETY PRECAUTIONS

5.1 Procedures shall be carried out in a manner that protects the health and safety of all Quanterra associates. The following requirements must be met:

5.1.1 Eye protection that satisfies ANSI Z87.1 (as per the Chemical Hygiene Plan), laboratory coat, and appropriate gloves must be worn while samples, standards, solvents and reagents are being handled. Disposable gloves that have become contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.1.2 The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the MSDS files maintained in the laboratory. The following specific hazards are known:

5.1.3 Chemicals that have been classified as carcinogens, or potential carcinogens,

under OSHA include: Benzo(a)anthracene, benzidine, 3,3'-dichlorobenzidine, benzo(a)pyrene, dibenzo(a,h)anthracene, and n-nitrosodimethylamine. Primary standards should be purchased in solution. If neat materials must be obtained, they shall be handled in a hood.

5.1.4 Exposure to chemicals must be maintained as low as reasonably achievable; therefore, unless they are known to be non-hazardous, all samples should be opened, transferred, and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers should be kept closed unless transfers are being made.

5.1.5 All work must be stopped in the event of a known or potential compromise to the health and safety of a Quanterra associate. The situation must be reported immediately to a laboratory supervisor.

6 EQUIPMENT AND SUPPLIES

6.1 Gas Chromatograph/Mass Spectrometer System: An analytical system complete with a temperature-programmable gas chromatograph suitable for split/splitless injection and all required accessories, including syringes, analytical columns, and gases. The capillary column should be directly coupled to the source.

6.2 Column: 30 m x 0.32 mm I.D. (or 0.25 mm I.D.) 0.5-µm film thickness silicon-coated fused-silica capillary column (J & W Scientific DB-5.625 or equivalent). Alternate columns are acceptable if they provide acceptable performance.

6.3 Mass Spectrometer: Capable of scanning from 35 to 500 AMU every one second or less, using 70 volts (nominal) electron energy in the electron impact ionization mode. The mass spectrometer must be capable of producing a mass spectrum for decafluorotriphenylphosphine (DFTPP) which meets all of the criteria in Table 6 when 50 ng of the GC/MS tuning standard is injected through the GC.

6.4 GC/MS Interface: Any GC-to-MS interface that gives acceptable calibration points and achieves acceptable tuning performance criteria may be used.

6.5 Data System: A computer system must be interfaced to the mass spectrometer. The system must allow the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer must have software that can search any GC/MS data file for ions of a specific mass and that can plot such ion abundances versus time or scan number. This type of plot is defined as the Extracted Ion Current Profile (EICP). Software must also be available that allows integrating the abundances in any EICP between specified time or scan-number limits. The most recent version of the EPA/NIH Mass Spectral Library is recommended.

6.6 Syringe: 10 µL Hamilton Laboratory grade syringes or equivalent.

6.7 Carrier gas: Ultra high purity helium.

7 REAGENTS AND STANDARDS

7.1 A minimum five point calibration curve is prepared. The low point should be at or below the reporting limit. Refer to Tables 12 and 13 for typical calibration levels for all analytes. Other calibration levels may be used, depending on instrument capability, but the low standard must support the reporting limit and the high standard defines the range of the calibration.

7.2 An Internal Standard solution is prepared. Compounds in the I.S. Mix are: acenaphthene-d10, chrysene-d12, 1,4-dichlorobenzene-d4, naphthalene-d8, perylene-d12, and phenanthrene-d10.

7.2.1 Internal Standards are added to all standards and extracts to result in 40ng injected onto the column. For example, if the volume of an extract used was 200 µL, 20 µL of a 400 µg/mL internal standard solution would be added for a 1 µL injection.

7.3 Surrogate Standard Spiking Solution: Prepare as indicated in the preparative methods. See appropriate preparation SOP. Surrogate compounds and levels are listed in Table 11.

7.4 GC/MS Tuning Standard: A methylene chloride solution containing 50 µg/mL of decafluorotriphenylphosphine (DFTPP) is prepared. Pentachlorophenol, benzidine, and DDT, should also be included in the Tuning Standard at 50 µg/mL.

7.5 Laboratory Control Spiking Solution: Prepare as indicated in the preparative methods. See appropriate preparation SOP. LCS compounds and levels are listed in Tables 9 and 10.

7.6 Matrix Spike Solution: Prepare as indicated in the preparative methods. See preparation SOP. The matrix spike compounds and levels are the same as the LCS compounds.

7.7 The standards listed in 7.1 to 7.6 should be refrigerated at $\leq$ 6°C when not in use. Refrigeration at -10°C to -20°C may be used if it can be demonstrated that analytes do not fall out of solution at this temperature. The standards must be replaced at least once a year. The continuing calibration standard must be replaced every week and is stored at $\leq$ 6°C.

8 SAMPLE PRESERVATION AND STORAGE

8.1 Reference appropriate facility SOP for sample bottle preservation and storage.

8.2 Samples are stored at 4 $\pm$ 2°C. Samples and extracts should be stored in suitable glass containers with Teflon lined caps. (Extracts will normally be stored for 30 days after invoicing.)

8.3 Water samples are extracted within seven days of sampling and the extracts are analyzed within forty days of extraction. Solids, sludges, and organic liquids are extracted within fourteen days of sampling and the extracts are analyzed within forty days of extraction.

9 QUALITY CONTROL

9.1 Initial Demonstration of Capability

9.1.1 For the standard analyte list, the initial demonstration and method detection limit (MDL) studies described in section 13 must be acceptable before analysis of samples may begin. Refer to the flow chart in section 17.4.1.

9.1.2 For non-standard analytes an MDL study should be performed and calibration curve generated before analyzing any samples, unless lesser requirements are previously agreed to with the client. In any event, the minimum initial demonstration required is analysis of an extracted standard at the reporting limit and a single point calibration.

9.2 Control Limits

In-house historical control limits must be determined for surrogates, matrix spikes, and laboratory control samples (LCS). These limits must be determined at least annually. The recovery limits are mean recovery +/- 3 standard deviations for surrogates, MS and LCSPrecision limits for matrix spikes / matrix spike duplicates are mean relative percent difference +/- 3 standard deviations.

9.2.1 These limits do not apply to dilutions (except for tests without a separate extraction), but surrogate and matrix spike recoveries will be reported unless the dilution is more than 5X.

9.2.2 All surrogate, LCS, and MS recoveries (except for dilutions) must be entered into QuantIMS (when available) or other database so that accurate historical control limits can be generated. For tests without a separate extraction, surrogates and matrix spikes will be reported for all dilutions.

9.2.3 Refer to the QC program document (QA-003) for further details of control limits.

9.3 Method Blank

A method blank is prepared and analyzed with each batch of samples. The method blank consists of reagent water for aqueous samples, and sodium sulfate for soil samples (Refer to SOP No. CORP-OP-0001 for details). Surrogates are added and the method blank is carried through the entire analytical procedure. The method blank must not contain any analyte of interest at or above the reporting limit (except common laboratory contaminants, see below) or at or above 5% of the measured concentration of that analyte in the associated samples, whichever is higher.

- If the analyte is a common laboratory contaminant (phthalate esters), the data may be reported with qualifiers if the concentration of the analyte is less than five times the RL. Such action must be taken in consultation with the client.

- Reanalysis of any samples with reportable concentrations of analytes found in the method blank is required unless other actions are agreed with the client.

- If there is no target analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. Such action should be taken in consultation with the client.

9.3.1 The method blank must have acceptable surrogate recoveries. If surrogate recoveries are not acceptable, the data must be evaluated to determine if the method blank has served the purpose of demonstrating that the analysis is free of contamination. If surrogate recoveries are low and there are reportable analytes in the associated samples, re-extraction of the blank and affected samples will normally be required. Consultation with the client should take place.

9.3.2 If reanalysis of the batch is not possible due to limited sample volume or other constraints, the method blank is reported, all associated samples are flagged with a "B", and appropriate comments may be made in a narrative to provide further documentation.

9.3.3 Refer to the Quanterra QC Program document (QA-003) for further details of the corrective actions.

9.3.4 Sample results are NOT blank subtracted unless specific requests and arrangements have been made with a client or agency.

9.4 Instrument Blank

9.4.1 Instruments must be evaluated for contamination during each 12 hour analytical run. This may be accomplished by analysis of a method blank. If a method blank is not available, an instrument blank must be analyzed. An instrument blank consists of methylene chloride with the internal standards added. It is evaluated in the same way as the method blank.

9.5 Laboratory Control Sample (LCS)

9.5.1 A laboratory control sample (LCS) is prepared and analyzed with every batch of samples. All analytes must be within established control limits. The LCS is spiked with the compounds listed in Tables 9 and 10 unless specified by a client or agency. The compounds must be spiked at a concentration equivalent to 100 or 150 ng on-column depending on the analyte.

9.5.2 If any analyte in the LCS is outside the laboratory established historical control limits, corrective action must occur. Corrective action may include re-extraction and reanalysis of the batch.

- If the batch is not re-extracted and reanalyzed, the reasons for accepting the batch must be clearly presented in the project records and the report. (An example of acceptable reasons for not reanalyzing might be that the matrix spike and matrix spike duplicate are acceptable, and sample surrogate recoveries are good, demonstrating that the problem was confined to the LCS).

- If re-extraction and reanalysis of the batch is not possible due to limited sample volume or other constraints, the LCS is reported, all associated samples are flagged, and appropriate comments are made in a narrative to provide further documentation.

9.5.3 Ongoing monitoring of the LCS provides evidence that the laboratory is performing the method within accepted QC guidelines for accuracy and precision.

9.6 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

A matrix spike/matrix spike duplicate (MS/MSD) is prepared and analyzed with every batch of samples. The MS/MSD is spiked with the same subset of analytes as the LCS (See Tables 9 and 10). Compare the percent recovery and relative percent difference (RPD) to that in the laboratory specific historically generated limits.

- If any individual recovery or RPD falls outside the acceptable range, corrective action must occur. The initial corrective action will be to check the recovery of that analyte in the Laboratory Control Sample (LCS). Generally, if the recovery of the analyte in the LCS is within limits, then the laboratory operation is in control and analysis may proceed. The reasons for accepting the batch must be documented.

- If the recovery for any component is outside QC limits for both the Matrix spike / spike duplicate and the LCS, the laboratory is out of control and corrective action must be taken. Corrective action will normally include repreparation and reanalysis of the batch.

- If a MS/MSD is not possible due to limited sample, then a LCS duplicate should be analyzed. RPD of the LCS and LCSD are compared to the matrix spike limits.

- The matrix spike / duplicate must be analyzed at the same dilution as the unspiked sample, even if the matrix spike compounds will be diluted out.

9.7 Surrogates

9.7.1 Every sample, blank, and QC sample is spiked with surrogate standards. Surrogate spike recoveries must be evaluated by determining whether the concentration (measured as percent recovery) falls within the required recovery limits. Surrogate compounds must be spiked at either 100 or 150 ng on-column, depending on the surrogate. The compounds routinely included in the surrogate spiking solution, along with recommended standard concentrations, are listed in Table 11.

9.7.2 If any surrogates are outside limits the following corrective actions must take

place (except for dilutions):

- Check all calculations for error.
- Ensure that instrument performance is acceptable.
- Recalculate the data and/or reanalyze the extract if either of the above checks reveal a problem.
- Re-extract and reanalyze the sample or flag the data as "Estimated Concentration" if neither of the above resolves the problem.

The decision to reanalyze or flag the data should be made in consultation with the client. It is only necessary to reprepare / reanalyze a sample once to demonstrate that poor surrogate recovery is due to matrix effect, unless the analyst believes that the repeated out of control results are not due to matrix effect.

9.7.3 If the sample with surrogate recoveries outside the recovery limits was a sample used for an MS/MSD and the surrogate recoveries in the MS/MSD are also outside of the control limits, then the sample, the MS, and the MSD do not require reanalysis as this phenomenon would indicate a possible matrix problem.

9.7.4 If the sample is reanalyzed and the surrogate recoveries in the reanalysis are acceptable, then the problem was within the analyst's control and only the reanalyzed data should be reported. (Unless the reanalysis was outside holding times, in which case reporting both sets of results may be appropriate.)

9.7.5 If the reanalysis does confirm the original results, the original analysis is reported and the data flagged as estimated due to matrix effect.

9.8 Nonconformance and Corrective Action

9.8.1 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the facility QA Manager.

9.9 Quality Assurance Summaries

Certain clients may require specific project or program QC which may supersede these method requirements. Quality Assurance Summaries should be developed to address these requirements.

9.10 Quanterra QC Program

Further details of QC and corrective action guidelines are presented in the Quanterra

QC Program document (QA-003). Refer to this document if in doubt regarding corrective actions.

10 CALIBRATION AND STANDARDIZATION

10.1 Summary

10.1.1 The instrument is tuned for DFTPP, calibrated initially with a five-point calibration curve, and verified each 12-hour shift with one or more continuing calibration standard(s). Recommended instrument conditions are listed in Table 5.

10.2 All standards and extracts are allowed to warm to room temperature before injecting.

10.3 Instrument Tuning

At the beginning of every twelve hour shift when analyses are to be performed, the GC/MS system must be checked to see if acceptable performance criteria (Table 6) is achieved for DFTPP (decafluorotriphenylphosphine).

10.3.1 Inject 50 ng of the GC/MS tuning standard (Section 7.4) into the GC/MS system. Obtain a background-corrected mass spectra of DFTPP and confirm that all the key m/z criteria in Table 6 are achieved. If all the criteria are not achieved, the analyst must retune the mass spectrometer and repeat the test until all criteria are achieved. The performance criteria must be achieved before any samples, blanks, or standards are analyzed.

10.3.2 The GC/MS tuning standard should also be used to evaluate the inertness of the chromatographic system. Benzidine and pentachlorophenol should not exhibit excessive tailing. If DDT is an analyte of interest, it must be included in the tuning standard, and its breakdown must be < 20%. Refer to section 12 for the appropriate calculations.

10.4 Initial Calibration

10.4.1 Internal Standard Calibration Procedure: Internal standards are listed in Table 7. Use the base peak m/z as the primary m/z for quantitation of the standards. If interferences are noted, use one of the next two most intense masses for quantitation.

10.4.2 Compounds should be assigned to the IS with the closest retention time.

10.4.3 Prepare calibration standards at a minimum of five concentration levels for each parameter of interest. Six standards must be used for a quadratic least squares calibration. It may also be useful to analyze six calibration levels and use the

lower five for most analytes and the upper five for analytes that have poor response. Add the internal standard mixture to result in 40 ng on column. (For example, if the volume of the calibration standard used is 1 mL, add 100 µL of the 400 µg/mL internal standard solution for a 1 µL injection). The concentrations of all analytes are listed in tables 12 and 13.

10.4.4 Analyze each calibration standard and tabulate the area of the primary characteristic m/z against concentration for each compound and internal standard. Calculate response factors (RF), average response factors, and the percent RSD of the response factors for each compound using the equations in section 12 and verify that the CCC and SPCC criteria in section 10.4.5 and 10.4.6 are met. **No sample analysis may be performed unless these criteria are met.**

10.4.5 System Performance Check Compounds (SPCCs): The minimum average RF for semivolatile SPCCs is 0.050. If the minimum response factors are not met, the system must be evaluated and corrective action must be taken before sample analysis begins. Some possible problems are standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or chromatographic system. This check must be met before analysis begins.

SPCC Compounds:

N-nitroso-di-n-propylamine
Hexachlorocyclopentadiene
2,4-Dinitrophenol
4-Nitrophenol

10.4.6 Calibration Check Compounds (CCCs): The %RSD of the response factors for each CCC in the initial calibration must be less than 30% for the initial calibration to be considered valid. This criterion must be met before sample analysis begins. Problems similar to those listed under SPCCs could affect this criterion.

10.4.6.1 If none of the CCCs are required analytes, project specific calibration specifications must be agreed with the client.

10.4.6.2 CCC Compounds:

Phenol
Acenaphthene
1,4-Dichlorobenzene
N-nitrosodiphenylamine
2-Nitrophenol
Pentachlorophenol
2,4-Dichlorophenol

Fluoranthene
Hexachlorobutadiene
Di-n-octylphthalate
4-Chloro-3-methylphenol
Benzo(a)pyrene
2,4,6-Trichlorophenol

10.4.7 If the average of all %RSDs in the initial calibration is ≤ 15%, then all analytes may use average response factor for calibration.

10.4.7.1 If the software in use is capable of routinely reporting curve coefficients for data validation purposes, and the necessary calibration reports can be generated, then the analyst should evaluate analytes with %RSD > 15% for calibration on a curve. If it appears that substantially better accuracy would be obtained using quantitation from a curve then the appropriate curve should be used for quantitation.

10.4.7.2 If the average of all the %RSDs in the initial calibration is > 15%, then calibration on a curve must be used for those analytes with %RSD > 15%. Linear or quadratic curve fits may be used. Use of 1/Concentration^2 weighting is recommended to improve the accuracy of quantitation at the low end of the curve. The analyst should consider instrument maintenance to improve the linearity of response. If Relative Standard Error (RSE) is used to evaluate the curve it must be better than 15%. Otherwise the correlation coefficient (coefficient of determination for non-linear curves) must be ≥ 0.990.

10.4.8 Weighting of data points

In a linear or quadratic calibration fit, the points at the lower end of the calibration curve have less weight in determining the curve generated than points at the high concentration end of the curve. However, in environmental analysis, accuracy at the low end of the curve is very important. For this reason it is preferable to increase the weighting of the lower concentration points. 1/Concentration^2 weighting (often called $1/X^2$ weighting) will improve accuracy at the low end of the curve and should be used if the data system has this capability.

10.4.9 If time remains in the 12 hour period initiated by the DFTPP injection before the initial calibration, samples may be analyzed. Otherwise, proceed to continuing calibration.

10.4.10 Quantitation is performed using the calibration curve or average response factor form the initial curve, not the continuing calibration.

10.5 Continuing Calibration

10.5.1 At the start of each 12-hour period, the GC/MS tuning standard must be analyzed. A 50 ng injection of DFTPP must result in a mass spectrum for DFTPP which meets the criteria given in Table 6.

10.5.2 Following a successful DFTPP analysis the continuing calibration standard(s) are analyzed. The standards must contain all semivolatile analytes, including all required surrogates. A mid level calibration standard is used for the continuing calibration.

10.5.3 The following criteria must be met for the continuing calibration to be acceptable:

- The SPCC compounds must have a response factor of ≥ 0.05.
- The percent difference or drift of the CCC compounds from the initial calibration must be $\leq 20\%$. (see section 12 for calculations) In addition, the percent difference or drift of all analytes must be $\leq 50\%$, with allowance being made for up to six target compounds to have percent drift greater than 50%.
- The internal standard response must be within 50-200% of the response in the mid level of the initial calibration.
- The internal standard retention times must be within 30 seconds of the retention times in the mid-level of the initial calibration.

10.5.3.1 If none of the CCCs are required analytes, project specific calibration specifications must be agreed with the client.

10.5.4 Once the above criteria have been met, sample analysis may begin. Initial calibration average RFs (or the calibration curve) will be used for sample quantitation, not the continuing calibration RFs. Analysis may proceed until 12 hours from the injection of the DFTPP have passed. (A sample *injected* less than 12 hours after the DFTPP is acceptable.)

11 PROCEDURE

11.1 Sample Preparation

Samples are prepared following SOP CORP-OP-0001.

11.2 Sample Analysis Procedure

11.2.1 Calibrate the instrument as described in section 10. Depending on the target compounds required by the client, it may be necessary to use more than one calibration standard.

11.2.2 All samples must be analyzed using the same instrument conditions as the preceeding continuing calibration standard.

11.2.3 Add internal standard to the extract to result in 40 ng injected on column (for example, 50 µL internal standard solution in 0.5 mL of extract for a 1 µL injection). Mix thoroughly before injection into the instrument.

11.2.4 Inject the sample extract into the GC/MS system using the same injection technique as used for the standards.

11.2.5 The data system will determine the concentration of each analyte in the extract using calculations equivalent to those in section 12. Quantitation is based on the initial calibration, not the continuing calibration.

11.2.6 Identified compounds are reviewed for proper integration. Manual integrations are performed if necessary and are documented by the analyst or automatically by the data system.

11.2.7 Target compounds identified by the data system are evaluated using the criteria listed in section 12.1.

11.2.8 Library searches of peaks present in the chromatogram that are not target compounds (Tentatively Identified Compounds, TIC) may be performed if required by the client. They are evaluated using the criteria in section 12.3.

11.3 Dilutions

If the response for any compound exceeds the working range of the GC/MS system, a dilution of the extract is prepared and analyzed. An appropriate dilution should be in the upper half of the calibration range. Samples may be screened to determine the appropriate dilution for the initial run. If the initial diluted run has no hits or hits below 20% of the calibration range and the matrix allows for analysis at a lesser dilution, the sample must be reanalyzed at a dilution targeted to bring the largest hit above 50% of the calibration range.

11.3.1 Guidance for Dilutions Due to Matrix

If the sample is initially run at a dilution and the baseline rise is less than the

height of the internal standards, or if individual non-target peaks are less than two times the height of the internal standards, the sample should be reanalyzed at a more concentrated dilution. This requirement is approximate and subject to analyst judgement. For example, samples containing organic acids may need to be analyzed at a higher dilution to avoid destroying the column.

11.3.2 Reporting Dilutions

The most concentrated dilution with no target compounds above the calibration range will be reported. Other dilutions will only be reported at client request.

11.4 Perform all qualitative and quantitative measurements. When the extracts are not being used for analyses, refrigerate them at 4 ± 2°C, protected from light in screw cap vials equipped with unpierced Teflon lined septa.

11.5 Retention time criteria for samples

If the retention time for any internal standard changes by more than 0.5 minutes from the last continuing calibration standard, the chromatographic system must be inspected for malfunctions and corrected. Reanalysis of samples analyzed while the system was malfunctioning is required.

11.5.1 If the retention time of any internal standard in any sample varies by more than 0.1 minute from the preceeding continuing calibration standard, the data must be carefully evaluated to ensure that no analytes have shifted outside their retention time windows.

11.6 Percent Moisture

Analytical results may be reported as dry or wet weight, as required by the client. Percent moisture must be determined if results will be reported as dry weight. Refer to the facility specific SOP for determination of percent moisture.

11.7 Procedural Variations

11.7.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file. Any unauthorized deviations from this procedure must also be documented as a non-conformance, with a cause and corrective action described.

11.8 Troubleshooting Guide

11.8.1 Daily Instrument Maintenance

In addition to the checks listed in the instrument maintenance schedule in the Quanterra QAMP, the following daily maintenance should be performed.

- Clip Column as necessary.
- Install new or cleaned injection port liner as necessary.
- Install new septum as necessary.
- Perform mass calibration as necessary.

11.8.2 Major Maintenance

A new initial calibration is necessary following major maintenance. Major maintenance includes changing the column, cleaning the ion volume or repeller, cleaning the source, and replacing the multiplier. Refer to the manufacturer's manual for specific guidance.

12 DATA ANALYSIS AND CALCULATIONS

12.1 Qualitative identification

An analyte is identified by retention time and by comparison of the sample mass spectrum with the mass spectrum of a standard of the suspected compound (standard reference spectrum). Mass spectra for standard reference may be obtained on the user's GC/MS by analysis of the calibration standards or from the NBS library. Two criteria must be satisfied to verify identification: (1) elution of sample component at the same GC retention time as the standard component; and (2) correspondence of the sample component and the standard component characteristic ions. (Note: Care must be taken to ensure that spectral distortion due to co-elution is evaluated.)

- The sample component retention time must compare to within ± 0.2 min. of the retention time of the standard component. For reference, the standard must be run within the same twelve hours as the sample.
- All ions present in the standard mass spectra at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) should be present in the sample spectrum.
- The characteristic ions of a compound must maximize in the same scan or

within one scan of each other.

- The relative intensities of ions should agree to within ±30% between the standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample abundance must be between 20% and 80%.)

12.1.1 If a compound cannot be verified by all the above criteria, but in the technical judgment of the analyst the identification is correct, the analyst shall report that identification and proceed with quantitation.

12.2 Mass chromatogram searches.

Certain compounds are unstable in the calibration standard and cannot be calibrated in the normal way. In particular, the compound hexachlorophene (CAS 70-30-4) falls into this category, and is required for Appendix IX analysis. For this analyte a mass chromatogram search is made.

12.2.1 Hexachlorophene

Display the mass chromatograms for mass 196 and mass 198 for the region of the chromatogram from at least 2 minutes before chrysene-d12 to at least 4 minutes after chrysene-d12. If peaks for both ions coincide then the analyst evaluates the spectrum for the presence of hexachlorophene. No quantitation is possible.

12.3 For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the type of analyses being conducted. Computer generated library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. Only after visual comparison of sample spectra with the nearest library searches shall the mass spectral interpretation specialist assign a tentative identification. Guidelines for making tentative identification are:

- Relative intensities of major ions in the reference spectrum (ions >10% of the most abundant ion) should be present in the sample spectrum.

- The relative intensities of the major ions should agree within ±20%. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance should be between 30%and 70%.)

- Molecular ions present in the reference spectrum should be present in the sample spectrum.

- Ions present in the sample spectrum, but not in the reference spectrum, should be

reviewed for possible background contamination or presence of coeluting compounds.

- Ions present in the reference spectrum, but not in the sample spectrum, should be reviewed for possible subtraction from the sample spectrum because of background contamination or coeluting peaks. Data system library reduction programs can sometimes create these discrepancies.

- Automatic background subtraction can severely distort spectra from samples with unresolved hydrocarbons.

12.4 Anyone evaluating data is trained to know how to handle isomers with identical mass spectra and close elution times. These include:

Dichlorobenzenes
Methylphenols
Trichlorophenols
Phenanthrene, anthracene
Fluoranthene, pyrene
Benzo(b) and (k)fluoranthene
Chrysene, benzo(a)anthracene

Extra precautions concerning these compounds are to more closely scrutinize retention time vs. the calibration standard and also to check that all isomers have distinct retention times.

A second category of problem compounds would be the poor responders or compounds that chromatograph poorly. Included in this category would be:

Benzoic acid
Chloroanilines
Nitroanilines
2,4-Dinitrophenol
4-Nitrophenol
Pentachlorophenol
3,3'-Dichlorobenzidine
Benzyl alcohol
4,6-Dinitro-2-methylphenol

Manually checking the integrations would be appropriate for these compounds.

12.5 Calculations

12.5.1 Percent Relative Standard Deviation for Initial Calibration

$$\%RSD = \frac{SD}{\overline{RF}} \times 100$$

RF = Mean of RFs from intial caibration for a compound

SD = Standard deviation of RFs from initial calibration for a compound,

$$= \sqrt{\sum_{i=1}^{N} \frac{\left(RFi - \overline{RF}\right)^2}{N-1}}$$

RFi = RF for each of the calibration levels

N = Number of RF values

12.5.2 Continuing calibration percent drift

$$\%Drift = \frac{C_{actual} - C_{found}}{C_{actual}} \times 100\%$$

C_{actual} = Known concentration in standard

C_{found} = Measured concentration using selected quantitation method

12.5.3 Concentration in the extract

The concentration of each identified analyte and surrogate in the extract is calculated from the linear or quadratic curve fitted to the initial calibration points, or from the average RF of the initial calibration.

12.5.3.1 Average response factor

If the average of all the %RSDs of the response factors in the initial calibration is ≤ 15%, the average response factor from the initial calibration may be used for quantitation.

$$C_{ex} = \frac{R_x C_{is}}{R_{is} \overline{RF}}$$

12.5.3.2 Linear fit

$$C_{ex} = A + B\frac{(R_x C_{is})}{R_{is}}$$

C_{ex}= Concentration in extract, µg/mL

R_x= Response for analyte

R_{is}= Response for internal standard

C_{is}= Concentration of internal standard

A= Intercept

B= Slope

12.5.3.3 Quadratic fit

$$C_{ex} = A + B\left(\frac{R_x C_{is}}{R_{is}}\right) + C\left(\frac{R_x C_{is}}{R_{is}}\right)$$

C= Curvature

12.5.4 The concentration in the sample is then calculated.

12.5.4.1 Aqueous Calculation

$$\text{Concentration, } \mu g / L = \frac{C_{ex}V_t}{V_o}$$

Where:

V_t = Volume of total extract, µL, taking into account dilutions (i.e., a 1-to-10 dilution of a 1 mL extract will mean V_t = 10,000 µL. If half of the base/neutral extract and half of the acid extract are combined, V_t = 2,000.)

V_o = Volume of water extracted (mL)

12.5.5 Sediment/Soil, Sludge (on a dry-weight basis) and Waste (normally on a wet-weight basis:

$$\text{Concentration, } \mu g / kg = \frac{C_{ex}V_t}{W_s D}$$

W_s = Weight of sample extracted or diluted in grams

D = (100 - % moisture in sample)/100, for a dry weight basis or 1 for a wet weight basis

12.6 MS/MSD percent recovery calculation.

$$\text{Matrix Spike Recovery} = \frac{S_{SR} - S_R}{S_A} \times 100\%$$

S_{SR} = Spike sample result

S_R=Sample result

S_A = Spike added

12.7 Relative % Difference calculation for the MS/MSD

$$RPD = \frac{MS_R - MSD_R}{1/2(MS_R + MSD_R)} \times 100$$

RPD = Relative percent difference

MS_R = Matrix spike result

MSD_R = Matrix spike duplicate result

12.8 Relative response factor calculation.

$$RF = \frac{A_x C_{is}}{A_{is} C_x}$$

A_x=Area of the characteristic ion for the compound being measured

A_{is}=Area of the characteristic ion for the specific internal standard

C_x=Concentration of the compound being measured (µg/L)

C_{is} =Concentration of the specific internal standard (µg/L)

12.9 Calculation of TICs: The calculation of TICs (tentatively identified compounds) is identical to the above calculations with the following exceptions:

A_x=Area of the total ion chromatogram for the compound being measured

A_{is}=Area of the total ion chromatogram for the nearest internal standard without interference

RF=1

12.10 Percent DDT breakdown

$$\% \text{ DDT breakdown} = \frac{\text{DDEarea} + \text{DDDarea}}{\text{DDTarea} + \text{DDEarea} + \text{DDarea}}$$

The total ion current areas are used for this calculation

13 METHOD PERFORMANCE

13.1 Method Detection Limit

Each laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in QA Policy #: QA-005.

13.2 Initial Demonstration

Each laboratory must make an initial demonstration of capability for each individual method. Demonstration of capability for both soil and water matrices is required. This requires analysis of QC check samples containing all of the standard analytes for the method. For some tests it may be necessary to use more than one QC check mix to cover all analytes of interest.

13.2.1 Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to the level 4 calibration standard.

13.2.2 Calculate the average recovery and standard deviation of the recovery for each analyte of interest. Compare these results with the acceptance criteria given in table 14.

13.2.3 If any analyte does not meet the acceptance criteria the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

13.3 Non-standard analytes

For non-standard analytes, an MDL study must be performed and calibration curve generated before analyzing any samples, unless lesser requirements are previously agreed to with the client. In any event, the minimum initial demonstration required is analysis of an extracted standard at the reporting limit and a single point calibration.

13.4 Training Qualification

The group/team leader has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

13.5 Data Quality Objectives (DQO). Refer to project-specific Quality Assurance plans for DQO information.

14 POLLUTION PREVENTION

14.1 This section is not applicable to this procedure.

15 WASTE MANAGEMENT

15.1 Waste generated during aliquotting and from used vials must be disposed of in accordance with the facility hazardous waste procedures. The Health and Safety Director should be contacted if additional information is required.

16 REFERENCES

16.1 SW846, Test Methods for Evaluating Solid Waste, Third Edition, Update II, October 1994, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS): Capillary Column Technique, Method 8270B.

16.2 J. W. Eichelberger, L. E. Harris, and W. L. Budde, "Reference Compound to Calibrate Ion Abundance Measurement in Gas Chromatography/Mass Spectrometry," Analytical Chemistry, 47, 995 (1975)

17 MISCELLANEOUS

17.1 Modifications from Reference Method

17.1.1 A retention time window of 0.2 minutes is used for all components, since some data systems do not have the capability of using the relative retention time units specified in the reference method.

17.1.2 The quantitation and qualifier ions for some compounds have been changed from those recommended in SW-846 in order to improve the reliability of qualitative identification.

17.2 Modifications from Previous Revision

17.2.1 This SOP has been substantially revised to meet the requirements of method 8270C.

17.2.2 Directions for analysis be method 625 have been added as an attachment.

17.3 Facility Specific SOPs

Each facility shall attach a list of facility specific SOPs or approved attachments (if

applicable) which are required to implement this SOP or which are used in conjunction with this SOP. If no facility specific SOPs or amendments are to be attached, a statement must be attached specifying that there are none.

17.4

Tables

Table 1

Quanterra Primary Standard[1] and Standard Reporting Limits

Analytes	CAS Number	Standard Reporting Limits	
		Aqueous µg/L	Low Soil/Sediment µg/kg
Pyridine	110-86-1	20	660
N-nitrosodimethylamine	62-75-9	10	330
Aniline	62-53-3	10	330
Phenol	108-95-2	10	330
Bis(2-chloroethyl)ether	111-44-4	10	330
2-Chlorophenol	95-57-8	10	330
1,3-Dichlorobenzene	541-73-1	10	330
1,4-Dichlorobenzene	106-46-7	10	330
Benzyl alcohol	100-51-6	10	330
1,2-Dichlorobenzene	95-50-1	10	330
2-Methylphenol	95-48-7	10	330
2,2'-oxybis(1-chloropropane)[2]	108-60-1	10	330
4-Methylphenol	106-44-5	10	330
N-Nitroso-di-n-propylamine	621-64-7	10	330
Hexachloroethane	67-72-1	10	330
Nitrobenzene	98-95-3	10	330
Isophorone	78-59-1	10	330
2-Nitrophenol	88-75-5	10	330
2,4-Dimethylphenol	105-67-9	10	330
Benzoic acid	65-85-0	50	1600
Bis(2-chloroethoxy)methane	111-91-1	10	330
2,4-Dichlorophenol	120-83-2	10	330
1,2,4-Trichlorobenzene	120-82-1	10	330
Naphthalene	91-20-3	10	330
4-Chloroaniline	106-47-8	10	330
Hexachlorobutadiene	87-68-3	10	330
4-Chloro-3-methylphenol	59-50-7	10	330
2-Methylnaphthalene	91-57-6	10	330
Hexachlorocyclopentadiene	77-47-4	50	1600
2,4,6-Trichlorophenol	88-06-2	10	330
2,4,5-Trichlorophenol	95-95-4	10	330
2-Chloronaphthalene	91-58-7	10	330
2-Nitroaniline	88-74-4	50	1600
Dimethyl phthalate	131-11-3	10	330
Acenaphthylene	208-96-8	10	330
3-Nitroaniline	99-09-2	50	1600
Acenaphthene	83-32-9	10	330
2,4-Dinitrophenol	51-28-5	50	1600
4-Nitrophenol	100-02-7	50	1600
Dibenzofuran	132-64-9	10	330
2,4-Dinitrotoluene	121-14-2	10	330
2,6-Dinitrotoluene	606-20-2	10	330
Diethylphthalate	84-66-2	10	330

Table 1

Quanterra Primary Standard[1] and Standard Reporting Limits

Analytes	CAS Number	Standard Reporting Limits	
		Aqueous µg/L	Low Soil/Sediment µg/kg
4-Chlorophenyl phenyl ether	7005-72-3	10	330
Fluorene	86-73-7	10	330
4-Nitroaniline	100-01-6	50	1600
4,6-Dinitro-2-methylphenol	534-52-1	50	1600
N-Nitrosodiphenylamine	86-30-6	10	330
Azobenzene	103-33-3	10	330
4-Bromophenyl phenyl ether	101-55-3	10	330
Hexachlorobenzene	118-74-1	10	330
Pentachlorophenol	87-86-5	50	1600
Phenanthrene	85-01-8	10	330
Anthracene	120-12-7	10	330
Carbazole	86-74-8	10	330
Di-n-butyl phthalate	84-74-2	10	330
Fluoranthene	206-44-0	10	330
Benzidine	92-87-5	100	3300
Pyrene	129-00-0	10	330
Butyl benzyl phthalate	85-68-7	10	330
3,3'-Dichlorobenzidine	91-94-1	50	1600
Benzo(a)anthracene	56-55-3	10	330
Bis(2-ethylhexyl)phthalate	117-81-7	10	330
Chrysene	218-01-9	10	330
Di-n-octylphthalate	117-84-0	10	330
Benzo(b)fluoranthene	205-99-2	10	330
Benzo(k)fluoranthene	207-08-9	10	330
Benzo(a)pyrene	50-32-8	10	330
Indeno(1,2,3-cd)pyrene	193-39-5	10	330
Dibenz(a,h)anthracene	53-70-3	10	330
Benzo(g,h,i)perylene	191-24-2	10	330

[1] The Quanterra primary standard is the standard normally used at Quanterra. Additional standards, such as the Appendix IX standard may be necessary to include all target analytes required for some clients.

[2] 2,2'oxybis(1-chloropropane) was formally known as bis(2-chloroisopropyl)ether

Table 2

Quanterra Appendix IX[1] Standard Reporting Limits

Semivolatiles	CAS Number	Standard Reporting Limits	
		Aqueous µg/L	Low Soil/Sediment µg/kg
2-Picoline	109-06-8	20	660
N-Nitrosomethylethylamine	10595-95-6	10	330
Methyl methanesulfonate	66-27-3	10	330
N-Nitrosodiethylamine	55-18-5	10	330
Ethyl methanesulfonate	62-50-0	10	330
Pentachloroethane	76-01-7	50	1600
Acetophenone	98-86-2	10	330
N-Nitrosopyrrolidine	930-55-2	10	330
N-Nitrosomorpholine	59-89-2	10	330
o-Toluidine	95-53-4	20	660
3-Methylphenol	108-39-4	10	330
N-Nitrosopiperidine	100-75-4	10	330
o,o,o-Triethyl-Phosphorothioate[2]	126-68-1	50	1600
a,a-Dimethyl-phenethylamine	122-09-8	50	1600
2,6-Dichlorophenol	87-65-0	10	330
Hexachloropropene	1888-71-7	100	3300
p-Phenylenediamine	106-50-3	100	3300
n-Nitrosodi-n-butylamine	924-16-3	10	330
Safrole	94-59-7	20	660
1,2,4,5-Tetrachlorobenzene	95-94-3	10	330
Isosafrole	120-58-1	20	660
1,4-Dinitrobenzene	100-25-4	10	330
1,4-Naphthoquinone	130-15-4	50	1600
1,3-Dinitrobenzene	99-65-0	10	330
Pentachlorobenzene	608-93-5	10	330
1-Naphthylamine	134-32-7	10	330
2-Naphthylamine	91-59-8	10	330
2,3,4,6-Tetrachlorophenol	58-90-2	50	1600
5-Nitro-o-toluidine	99-55-8	20	660
Thionazin[2]	297-97-2	50	1600
1,3,5-Trinitrobenzene	99-35-4	50	1600
Sulfotepp[2]	3689-24-5	50	1600
Phorate[2]	298-02-2	50	1600
Phenacetin	62-44-2	20	660
Diallate[3]	2303-16-4	20	660
Dimethoate[2]	60-51-5	20	660
4-Aminobiphenyl	92-67-1	50	1600
Pentachloronitrobenzene	82-68-8	50	1600
Pronamide	23950-58-5	20	660
Disulfoton[2]	298-04-4	50	1600
2-secbutyl-4,6-dinitrophenol (Dinoseb)	88-85-7	20	660
Methyl Parathion[2]	298-00-0	50	1600
4-Nitroquinoline-1-oxide	56-57-5	100	3300
Parathion[2]	56-38-2	50	1600

Table 2

Quanterra Appendix IX[1] Standard Reporting Limits

Semivolatiles	CAS Number	Standard Reporting Limits	
		Aqueous µg/L	Low Soil/Sediment µg/kg
Methapyrilene	91-80-5	50	1600
Aramite	140-57-8	20	660
Isodrin[3]	465-73-6	10	330
Kepone[2]	143-50-0	100	3300
Famphur[3]	52-85-7	100	3300
p-(Dimethylamino)azobenzene	60-11-7	20	660
p-Chlorobenzilate[3]	510-15-6	10	330
3,3'-Dimethylbenzidine	119-93-7	50	1600
2-Acetylaminofluorene	53-96-3	100	3300
Dibenz(a,j)acridine	224-42-0	20	660
7,12-Dimethylbenz(a)anthracene	57-97-6	20	660
3-Methylcholanthrene	56-49-5	20	660

1 The Appendix IX standard contains additional analytes required for the Appendix IX list. The Quanterra primary standard must also be analyzed to include all of the Appendix IX list.

2 May also be analyzed by method 8140 or 8141, which can achieve lower reporting limits.

3 May also be analyzed by method 8080 or 8081, which can achieve lower reporting limits

Table 3

Reportable Analytes for Quanterra Standard Tests, Primary Standard

Analyte	CAS Number	Quanterra Standard List	TCLP	TCL	Appendix IX
Pyridine	110-86-1		X		X
N-nitrosodimethylamine	62-75-9				X
Aniline	62-53-3				X
Phenol	108-95-2	X		X	X
Bis(2-chloroethyl)ether	111-44-4	X		X	X
2-Chlorophenol	95-57-8	X		X	X
1,3-Dichlorobenzene	541-73-1	X		X	X
1,4-Dichlorobenzene	106-46-7	X	X	X	X
Benzyl alcohol	100-51-6				X
1,2-Dichlorobenzene	95-50-1	X		X	X
2-Methylphenol	95-48-7	X	X	X	X
2,2'-oxybis(1-chloropropane)[1]	180-60-1	X		X	X
4-Methylphenol	106-44-5	X	X	X	X
N-Nitroso-di-n-propylamine	621-64-7	X		X	X
Hexachloroethane	67-72-1	X	X	X	X
Nitrobenzene	98-95-3	X	X	X	X
Isophorone	78-59-1	X		X	X
2-Nitrophenol	88-75-5	X		X	X
2,4-Dimethylphenol	105-67-9	X		X	X
Benzoic acid	65-85-0				
Bis(2-chloroethoxy)methane	111-91-1	X		X	X
2,4-Dichlorophenol	120-83-2	X		X	X
1,2,4-Trichlorobenzene	120-82-1	X		X	X
Naphthalene	91-20-3	X		X	X
4-Chloroaniline	106-47-8	X		X	X
Hexachlorobutadiene	87-68-3	X	X	X	X
4-Chloro-3-methylphenol	59-50-7	X		X	X
2-Methylnaphthalene	91-57-6	X		X	X
Hexachlorocyclopentadiene	77-47-4	X		X	X
2,4,6-Trichlorophenol	88-06-2	X	X	X	X
2,4,5-Trichlorophenol	95-95-4	X	X	X	X
2-Chloronaphthalene	91-58-7	X		X	X
2-Nitroaniline	88-74-4	X		X	X
Dimethyl phthalate	131-11-3	X		X	X
Acenaphthylene	208-96-8	X		X	X
3-Nitroaniline	99-09-2	X		X	X
Acenaphthene	83-32-9	X		X	X
2,4-Dinitrophenol	51-28-5	X		X	X
4-Nitrophenol	100-02-7	X		X	X
Dibenzofuran	132-64-9	X		X	X
2,4-Dinitrotoluene	121-14-2	X	X	X	X
2,6-Dinitrotoluene	606-20-2	X		X	X
Diethylphthalate	84-66-2	X		X	X
4-Chlorophenyl phenyl ether	7005-72-3	X		X	X
Fluorene	86-73-7	X		X	X

Table 3

Reportable Analytes for Quanterra Standard Tests, Primary Standard

Analyte	CAS Number	Quanterra Standard List	TCLP	TCL	Appendix IX
4-Nitroaniline	100-01-6	X		X	X
4,6-Dinitro-2-methylphenol	534-52-1	X		X	X
N-Nitrosodiphenylamine	86-30-6	X		X	X
Azobenzene[4]	103-33-3				
4-Bromophenyl phenyl ether	101-55-3	X		X	X
Hexachlorobenzene	118-74-1	X	X	X	X
Pentachlorophenol	87-86-5	X	X	X	X
Phenanthrene	85-01-8	X		X	X
Anthracene	120-12-7	X		X	X
Carbazole	86-74-8	X		X	
Di-n-butyl phthalate	84-74-2	X		X	X
Fluoranthene	206-44-0	X		X	X
Benzidine	92-87-5				
Pyrene	129-00-0	X		X	X
Butyl benzyl phthalate	85-68-7	X		X	X
3,3'-Dichlorobenzidine	91-94-1	X		X	X
Benzo(a)anthracene	56-55-3	X		X	X
Bis(2-ethylhexyl)phthalate	117-81-7	X		X	X
Chrysene	218-01-9	X		X	X
Di-n-octylphthalate	117-84-0	X		X	X
Benzo(b)fluoranthene	205-99-2	X		X	X
Benzo(k)fluoranthene	207-08-9	X		X	X
Benzo(a)pyrene	50-32-8	X		X	X
Indeno(1,2,3-cd)pyrene	193-39-5	X		X	X
Dibenz(a,h)anthracene	53-70-3	X		X	X
Benzo(g,h,i)perylene	191-24-2	X		X	X

[1] 2,2'oxybis(1-chloropropane) was formally known as bis(2-chloroisopropyl)ether

[2] Azobenzene is formed by decomposition of 1,2-diphenlyhydrazine. If 1,2-diphenylhydrazine is requested, it will be analyzed as azobenzene.

Table 4

Reportable analytes for Quanterra Standard Tests, Appendix IX Standard

Semivolatiles	CAS Number	Quanterra Standard List	TCLP	TCL	Appendix IX
2-Picoline	109-06-8				X
N-Nitrosomethylethylamine	10595-95-6				X
Methyl methanesulfonate	66-27-3				X
N-Nitrosodiethylamine	55-18-5				X
Ethyl methanesulfonate	62-50-0				X
Pentachloroethane	76-01-7				X
Acetophenone	98-86-2				X
N-Nitrosopyrrolidine	930-55-2				X
N-Nitrosomorpholine	59-89-2				X
o-Toluidine	95-53-4				X
3-Methylphenol	108-39-4				X
N-Nitrosopiperidine	100-75-4				X
o,o,o-Triethyl-Phosphorothioate[2]	126-68-1				X
a,a-Dimethyl-phenethylamine	122-09-8				X
2,6-Dichlorophenol	87-65-0				X
Hexachloropropene	1888-71-7				X
p-Phenylenediamine	106-50-3				X
n-Nitrosodi-n-butylamine	924-16-3				X
Safrole	94-59-7				X
1,2,4,5-Tetrachlorobenzene	95-94-3				X
Isosafrole	120-58-1				X
1,4-Dinitrobenzene	100-25-4				
1,4-Naphthoquinone	130-15-4				X
1,3-Dinitrobenzene	99-65-0				X
Pentachlorobenzene	608-93-5				X
1-Naphthylamine	134-32-7				X
2-Naphthylamine	91-59-8				X
2,3,4,6-Tetrachlorophenol	58-90-2				X
5-Nitro-o-toluidine	99-55-8				X
Thionazin[2]	297-97-2				X
1,3,5-Trinitrobenzene	99-35-4				X
Sulfotepp[2]	3689-24-5				X
Phorate[2]	298-02-2				X
Phenacetin	62-44-2				X
Diallate	2303-16-4				X
Dimethoate[2]	60-51-5				X
4-Aminobiphenyl	92-67-1				X
Pentachloronitrobenzene	82-68-8				X
Pronamide	23950-58-5				X
Disulfoton[2]	298-04-4				X
2-secbutyl-4,6-dinitrophenol (Dinoseb)[2]	88-85-7				X
Methyl parathion[2]	298-00-0				X
4-Nitroquinoline-1-oxide	56-57-5				X
Parathion[2]	56-38-2				X

Table 4

Reportable analytes for Quanterra Standard Tests, Appendix IX Standard

Semivolatiles	CAS Number	Quanterra Standard List	TCLP	TCL	Appendix IX
Isodrin[3]	465-73-6				X
Kepone [2]	143-50-0				X
Famphur[2]	52-85-7				X
Methapyrilene	91-80-5				X
Aramite	140-57-8				X
p-(Dimethylamino)azobenzene	60-11-7				X
p-Chlorobenzilate[3]	510-15-6				X
3,3'-Dimethylbenzidine	119-93-7				X
2-Acetylaminofluorene	53-96-3				X
Dibenz(a,j)acridine	224-42-0				
7,12-Dimethylbenz(a)anthracene	57-97-6				X
3-Methylcholanthrene	56-49-5				X
Hexachlorophene[4]	70-30-4				X
Diphenylamine[5]	122-39-4				X

2 May also be analyzed by method 8140 or 8141, which can achieve lower reporting limits.

3 May also be analyzed by method 8080 or 8081, which can achieve lower reporting limits

4 Hexachlorophene is a required analyte for Appendix IX. This compound is not stable, and therefore not included in the calibration standard. The characteristic ions for hexachlorophene are searched for in the chromatogram. (See section 12.2.1)

5 Diphenylamine is a required compound for Appendix IX. N-nitrosodiphenylamine decomposes in the injection port to form diphenylamine. Therefore these two compounds cannot be distinguished. Diphenylamine is not included in the calibration standard.

Table 5

Suggested Instrumental Conditions

Mass Range	35-500 amu
Scan Time	≤1 second/scan
Initial Column Temperature/Hold Time	40°C for 2 minutes
Column Temperature Program	40 - 320°C at 11.5°C/min
Final Column Temperature/Hold Time	320°C (until at least one minute after benzo(g,h,i)perylene has eluted)
Injector Temperature	250 - 300°C
Transfer Line Temperature	250 - 300°C
Source Temperature	According to manufacturer's specifications
Injector	Grob-type, split / splitless
Sample Volume	1 or 2 µl
Carrier Gas	Helium at 30 cm/sec

Table 6

DFTPP Key Ions and Ion Abundance Criteria

Mass	Ion Abundance Criteria
51	30 - 60% of mass 198
68	<2% of mass 69
70	<2% of mass 69
127	40 - 60% of mass 198
197	<1% of mass 198
198	Base peak, 100% relative abundance
199	5 - 9% of mass 198
275	10 - 30% of mass 198
365	>1% of mass 198
441	Present, but less than mass 443
442	>40% of mass 198
443	17 - 23% of mass 442

Table 7

Analytes in Approximate Retention Time Order and Characteristic Ions, Primary Standard

Analyte	Primary	Secondary	Tertiary
N-nitrosodimethylamine	74	42	
Pyridine	79	52	
2-Fluorophenol (Surrogate Standard)	112	64	63
Phenol-d5 (Surrogate Standard)	99	42	71
Aniline	93	66	
Phenol	94	65	66
Bis(2-chloroethyl)ether	93	63	95
2-Chlorophenol	128	64	130
1,3-Dichlorobenzene	146	148	113
1,4-Dichlorobenzene-d4 (Internal Standard)	152	150	115
1,4-Dichlorobenzene	146	148	113
Benzyl Alcohol	108	79	77
1,2-Dichlorobenzene	146	148	113
2-Methylphenol	108	107	79
2,2'-oxybis(1-chloropropane)[1]	45	77	79
4-Methylphenol	108	107	79
N-Nitroso-di-n-propylamine	70	42	101,130
Hexachloroethane	117	201	199
Nitrobenzene-d5 (Surrogate Standard)	82	128	54
Nitrobenzene	77	123	65
Isophorone	82	95	138
2-Nitrophenol	139	65	109
2,4-Dimethylphenol	107	121	122
Benzoic Acid	122	105	77
Bis(2-chloroethoxy)methane	93	95	123
2,4-Dichlorophenol	162	164	98
1,2,4-Trichlorobenzene	180	182	145
Naphthalene-d8 (Internal Standard)	136	68	54
Naphthalene	128	129	127
4-Chloroaniline	127	129	65
Hexachlorobutadiene	225	223	227
4-Chloro-3-methylphenol	107	144	142
2-Methylnaphthalene	142	141	115
Hexachlorocyclopentadiene	237	235	272
2,4,6-Trichlorophenol	196	198	200
2,4,5-Trichlorophenol	196	198	200
2-Fluorobiphenyl (Surrogate Standard)	172	171	170
2-Chloronaphthalene	162	164	127
2-Nitroaniline	65	92	138
Dimethylphthalate	163	194	164
Acenaphthylene	152	151	153
2,6-Dinitrotoluene	165	63	89

Table 7

Analytes in Approximate Retention Time Order and Characteristic Ions, Primary Standard

Analyte	Primary	Secondary	Tertiary
Acenaphthene-d10 (Internal Standard)	164	162	160
3-Nitroaniline	138	108	92
Acenaphthene	153	152	154
2,4-Dinitrophenol	184	63	154
Dibenzofuran	168	139	84
4-Nitrophenol	109	139	65
2,4-Dinitrotoluene	165	63	89
Diethylphthalate	149	177	150
Fluorene	166	165	167
4-Chlorophenylphenylether	204	206	141
4-Nitroaniline	138	92	108
4,6-Dinitro-2-methylphenol	198	182	77
N-Nitrosodiphenylamine	169	168	167
2,4,6-Tribromophenol (Surrogate Standard)	330	332	141
Azobenzene	77	182	105
4-Bromophenylphenylether	248	250	141
Hexachlorobenzene	284	142	249
Pentachlorophenol	266	264	268
Phenanthrene-d10 (Internal Standard)	188	94	80
Phenanthrene	178	179	176
Anthracene	178	179	176
Carbazole	167	166	139
Di-n-butylphthalate	149	150	104
Fluoranthene	202	101	100
Benzidine	184	92	185
Pyrene	202	101	100
Terphenyl-d14 (Surrogate Standard)	244	122	212
Butylbenzylphthalate	149	91	206
Benzo(a)Anthracene	228	229	226
Chrysene-d12 (Internal Standard)	240	120	236
3,3'-Dichlorobenzidine	252	254	126
Chrysene	228	226	229
Bis(2-ethylhexyl)phthalate	149	167	279
Di-n-octylphthalate	149	167	43
Benzo(b)fluoranthene	252	253	125
Benzo(k)fluoranthene	252	253	125
Benzo(a)pyrene	252	253	125
Perylene-d12 (Internal Standard)	264	260	265
Indeno(1,2,3-cd)pyrene	276	138	277
Dibenz(a,h)anthracene	278	139	279
Benzo(g,h,i)perylene	276	138	277

Table 8

Analytes in Approximate Retention Time Order and Characteristic Ions, Appendix IX Standard

Analyte	Primary	Secondary	Tertiary
2-Picoline	93	66	92
N-Nitrosomethylethylamine	88	42	43
Methyl methanesulfonate	80	79	65
N-Nitrosodiethylamine	102	44	57
Ethyl methanesulfonate	79	109	97
Pentachloroethane	117	119	167
Acetophenone	105	77	120
N-Nitrosopyrrolidine	100	41	42
N-Nitrosomorpholine	116	56	86
o-Toluidine	106	107	
3-Methylphenol	108	107	77
N-Nitrosopiperidine	114	42	55
o,o,o-Triethyl-Phosphorothioate	198	121	93
a,a-Dimethyl-phenethylamine	58	91	
2,6-Dichlorophenol	162	164	63
Hexachloropropene	213	215	211
p-Phenylenediamine	108	80	
n-Nitrosodi-n-butylamine	84	57	41
Safrole	162	104	77
1,2,4,5-Tetrachlorobenzene	216	214	218
Isosafrole 1	162	104	131
Isosafrole 2	162	104	131
1,4-Dinitrobenzene	168	75	122
1,4-Naphthoquinone	158	104	102
1,3-Dinitrobenzene	168	75	76
Pentachlorobenzene	250	248	252
1-Naphthylamine	143	115	
2-Naphthylamine	143	115	
2,3,4,6-Tetrachlorophenol	232	230	131
5-Nitro-o-toluidine	152	77	106
Thionazin	97	96	143
1,3,5-Trinitrobenzene	213	75	120
Sulfotepp	97	322	202
Phorate	75	97	121
Phenacetin	108	179	109
Diallate	86	234	
Dimethoate	87	93	125
4-Aminobiphenyl	169		
Pentachloronitrobenzene	237	142	214
Pronamide	173	175	255
Disulfoton	88	97	89
2-secbutyl-4,6-dinitrophenol (Dinoseb)	211	163	147
Methyl parathion	109	125	263
4-Nitroquinoline-1-oxide	190	128	160
Parathion	109	97	291
Isodrin	193	66	195

Table 8

Analytes in Approximate Retention Time Order and Characteristic Ions, Appendix IX Standard

Analyte	Primary	Secondary	Tertiary
Kepone	272	274	237
Famphur	218	125	93
Methapyrilene	97	58	
Aramite 1	185	319	
Aramite 2	185	319	
p-(Dimethylamino)azobenzene	120	225	77
p-Chlorobenzilate	251	139	253
3,3'-Dimethylbenzidine	212	106	
2-Acetylaminofluorene	181	180	223
Dibenz(a,j)acridine	279	280	
7,12-Dimethylbenz(a)anthracene	256	241	120
3-Methylcholanthrene	268	252	253

Table 9

8270C LCS Compounds

LCS Compounds	Spiking Level, ng/µL in extract[1]
1,2,4-Trichlorobenzene	100
Acenaphthene	100
2,4-Dinitrotoluene	100
Pyrene	100
N-Nitroso-di-n-propylamine	100
1,4-Dichlorobenzene	100
Pentachlorophenol	150
Phenol	150
2-Chlorophenol	150
4-Chloro-3-methylphenol	150
4-Nitrophenol	150

[1] Levels are 50 and 75 ng/µL if 2 µL injection is used

Table 10

TCLP LCS Compounds

LCS Compounds	Spiking Level, ng/µL in extract[1]
1,4-Dichlorobenzene	100
2,4-Dinitrotoluene	100
Hexachlorobenzene	100
Hexachlorobutadiene	100
Hexachloroethane	100
2-Methylphenol	100
3-Methylphenol	100
4-Methylphenol	100
Nitrobenzene	100
Pentachlorophenol	100
Pyridine	100
2,4,5-Trichlorophenol	100
2,4,6-Trichlorophenol	100

[1] Levels are 50 ng/µL if 2 µL injection is used

Recovery limits for the LCS and for matrix spikes are generated from historical data and are maintained by the QA department.

Table 11

8270C Surrogate Compounds

Surrogate Compounds	Spiking Level, ng/µL in extract[2]
Nitrobenzene-d5	100
2-Fluorobiphenyl	100
Terphenyl-d14	100
1,2-Dichlorobenzene-d4[1]	100
Phenol-d5	150
2-Fluorophenol	150
2,4,6-Tribromophenol	150
2-Chlorophenol-d4[1]	150

[1] Included in standard mix, but not routinely evaluated for method 8270B

[2] Levels are 50 and 75 ng/µL if 2 µL injection is used

Recovery limits for surrogates are generated from historical data and are maintained by the QA department.

Table 12

Calibration Levels, Primary Standard, µg/mL

Analyte	Level 1	Level 2	Level 3	Level 4	Level 5
Pyridine	10	20	50	100	160
N-nitrosodimethylamine	10	20	50	100	160
Aniline	10	20	50	100	160
Phenol	10	20	50	100	160
Bis(2-chloroethyl)ether	10	20	50	100	160
2-Chlorophenol	10	20	50	100	160
1,3-Dichlorobenzene	10	20	50	100	160
1,4-Dichlorobenzene	10	20	50	100	160
Benzyl alcohol	10	20	50	100	160
1,2-Dichlorobenzene	10	20	50	100	160
2-Methylphenol	10	20	50	100	160
2,2'-oxybis(1-chloropropane)[1]	10	20	50	100	160
4-Methylphenol	10	20	50	100	160
N-Nitroso-di-n-propylamine	10	20	50	100	160
Hexachloroethane	10	20	50	100	160
Nitrobenzene	10	20	50	100	160
Isophorone	10	20	50	100	160
2-Nitrophenol	10	20	50	100	160
2,4-Dimethylphenol	10	20	50	100	160
Benzoic acid	20	50	100	200	320
Bis(2-chloroethoxy)methane	10	20	50	100	160
2,4-Dichlorophenol	10	20	50	100	160
1,2,4-Trichlorobenzene	10	20	50	100	160
Naphthalene	10	20	50	100	160
4-Chloroaniline	10	20	50	100	160
Hexachlorobutadiene	10	20	50	100	160
4-Chloro-3-methylphenol	10	20	50	100	160
2-Methylnaphthalene	10	20	50	100	160
Hexachlorocyclopentadiene	10	20	50	100	160
2,4,6-Trichlorophenol	10	20	50	100	160
2,4,5-Trichlorophenol	10	20	50	100	160
2-Chloronaphthalene	10	20	50	100	160
2-Nitroaniline	20	50	100	200	320
Dimethyl phthalate	10	20	50	100	160
Acenaphthylene	10	20	50	100	160
3-Nitroaniline	20	50	100	200	320
Acenaphthene	10	20	50	100	160
2,4-Dinitrophenol	20	50	100	200	320
4-Nitrophenol	20	50	100	200	320
Dibenzofuran	10	20	50	100	160
2,4-Dinitrotoluene	10	20	50	100	160
2,6-Dinitrotoluene	10	20	50	100	160
Diethylphthalate	10	20	50	100	160
4-Chlorophenyl phenyl ether	10	20	50	100	160
Fluorene	10	20	50	100	160
4-Nitroaniline	10	20	50	100	160

Table 12

Calibration Levels, Primary Standard, µg/mL

Analyte	Level 1	Level 2	Level 3	Level 4	Level 5
4,6-Dinitro-2-methylphenol	20	50	100	200	320
N-Nitrosodiphenylamine	10	20	50	100	160
Azobenzene[2]	10	20	50	100	160
4-Bromophenyl phenyl ether	10	20	50	100	160
Hexachlorobenzene	10	20	50	100	160
Pentachlorophenol	20	50	100	200	320
Phenanthrene	10	20	50	100	160
Anthracene	10	20	50	100	160
Carbazole	10	20	50	100	160
Di-n-butyl phthalate	10	20	50	100	160
Fluoranthene	10	20	50	100	160
Benzidine	20	50	100	200	320
Pyrene	10	20	50	100	160
Butyl benzyl phthalate	10	20	50	100	160
3,3'-Dichlorobenzidine	20	50	100	200	320
Benzo(a)anthracene	10	20	50	100	160
Bis(2-ethylhexyl)phthalate	10	20	50	100	160
Chrysene	10	20	50	100	160
Di-n-octylphthalate	10	20	50	100	160
Benzo(b)fluoranthene	10	20	50	100	160
Benzo(k)fluoranthene	10	20	50	100	160
Benzo(a)pyrene	10	20	50	100	160
Indeno(1,2,3-cd)pyrene	10	20	50	100	160
Dibenz(a,h)anthracene	10	20	50	100	160
Benzo(g,h,i)perylene	10	20	50	100	160

[1] 2,2'oxybis(1-chloropropane) was formally known as bis(2-chloroisopropyl)ether

[2]Azobenzene is formed by decomposition of 1,2-diphenlyhydrazine. If 1,2-diphenylhydrazine is requested, it will be analyzed as azobenzene.

Table 13

Calibration Levels, Appendix IX Standard, µg/mL

Semivolatiles	Level 1	Level 2	Level 3	Level 4	Level 5
2-Picoline	10	20	50	100	160
N-Nitrosomethylethylamine	10	20	50	100	160
Methyl methanesulfonate	10	20	50	100	160
N-Nitrosodiethylamine	10	20	50	100	160
Ethyl methanesulfonate	10	20	50	100	160
Pentachloroethane	10	20	50	100	160
Acetophenone	10	20	50	100	160
N-Nitrosopyrrolidine	10	20	50	100	160
N-Nitrosomorpholine	10	20	50	100	160
o-Toluidine	10	20	50	100	160
3-Methylphenol	10	20	50	100	160
N-Nitrosopiperidine	10	20	50	100	160
o,o,o-Triethyl-Phosphorothioate	20	50	100	200	320
a,a-Dimethyl-phenethylamine	10	20	50	100	160
2,6-Dichlorophenol	10	20	50	100	160
Hexachloropropene	20	50	100	200	320
p-Phenylenediamine	10	20	50	100	160
n-Nitrosodi-n-butylamine	10	20	50	100	160
Safrole	10	20	50	100	160
1,2,4,5-Tetrachlorobenzene	10	20	50	100	160
Isosafrole 1 + 2	20	50	100	200	320
1,4-Dinitrobenzene	10	20	50	100	160
1,4-Naphthoquinone	10	20	50	100	160
1,3-Dinitrobenzene	10	20	50	100	160
Pentachlorobenzene	10	20	50	100	160
1-Naphthylamine	10	20	50	100	160
2-Naphthylamine	10	20	50	100	160
2,3,4,6-Tetrachlorophenol	10	20	50	100	160
5-Nitro-o-toluidine	10	20	50	100	160
Thionazin	10	20	50	100	160
1,3,5-Trinitrobenzene	20	50	100	200	320
Sulfotepp	10	20	50	100	160
Phorate	10	20	50	100	160
Phenacetin	10	20	50	100	160
Diallate 1 + 2	20	50	100	200	320
Dimethoate	10	20	50	100	160
4-Aminobiphenyl	10	20	50	100	160
Pentachloronitrobenzene	20	50	100	200	320
Pronamide	10	20	50	100	160
Disulfoton	10	20	50	100	160
2-secbutyl-4,6-dinitrophenol (Dinoseb)	20	50	100	200	320
Methyl parathion	10	20	50	100	160
4-Nitroquinoline-1-oxide	20	40	100	200	320
Parathion	10	20	50	100	160
Isodrin	10	20	50	100	160
Kepone	20	50	100	200	320

Table 13

Calibration Levels, Appendix IX Standard, µg/mL

Semivolatiles	Level 1	Level 2	Level 3	Level 4	Level 5
Famphur	20	50	100	200	320
Methapyrilene	10	20	50	100	160
Aramite 1 and 2	20	50	100	200	320
p-(Dimethylamino)azobenzene	10	20	50	100	160
p-Chlorobenzilate	10	20	50	100	160
3,3'-Dimethylbenzidine	10	20	50	100	160
2-Acetylaminofluorene	10	20	50	100	160
Dibenz (a,j)acridine	10	20	50	100	160
7,12-Dimethylbenz(a)anthracene	10	20	50	100	160
3-Methylcholanthrene	10	20	50	100	160

Table 14
Initial demonstration recovery and precision limits

Compound	Spiking concentration µg/L	Limit for Relative Standard Deviation	Limit for average recovery, %
Acenaphthene	60	27.6	60.1-132.3
Acenaphthylene	60	40.2	53.5-126.0
Aldrin[1]	60	39.0	7.2-152.2
Anthracene	60	32.0	43.4-118.0
Benz(a)anthracene	60	27.6	41.8-133.0
Benzo(b)fluoranthene	60	38.8	42.0-140.4
Benzo(k)fluoranthene	60	32.3	25.2-145.7
Benzo(a)pyrene	60	39.0	31.7-148.0
Benzo(ghi)perylene	60	58.9	D-195.0
Benzylbutyl phthalate	60	23.4	D-139.9
B-BHC[1]	60	31.5	41.5-130.6
d-BHC[1]	60	21.6	D-100.0
Bis(2-chloroethyl) ether	60	55.0	42.9-126.0
Bis(2-chloroethoxy)methane	60	34.5	49.2-164.7
Bis(2-chloroisopropyl) ether	60	46.3	62.8-138.6
Bis(2-ethylhexyl) phthalate	60	41.1	28.9-136.8
4-Bromophenyl phenyl ether	60	23.0	64.9-114.4
2-Chloronaphthalene	60	13.0	64.5-113.5
4-Chlorophenyl phenyl ether	60	33.4	38.4-144.7
Chrysene	60	48.3	44.1-139.9
4,4'-DDD[1]	60	31.0	D-134.5
4,4'-DDE[1]	60	32.0	19.2-119.7
4,4'-DDT[1]	60	61.6	D-170.6
Dibenzo(a,h)anthracene	60	70.0	D-199.7
Di-n-butyl phthalate	60	16.7	8.4-111.0
1,2-Dichlorobenzene	60	30.9	48.6-112.0
1,3-Dichlorobenzene	60	41.7	16.7-153.9
1,4-Dichlorobenzene	60	32.1	37.3-105.7

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Table 14
Initial demonstration recovery and precision limits

Compound	Spiking concentration µg/L	Limit for Relative Standard Deviation	Limit for average recovery, %
3,3'-Dichlorobenzidine	60	71.4	8.2-212.5
Dieldrin[1]	60	30.7	44.3-119.3
Diethyl phthalate	60	26.5	D-100.0
Dimethyl phthalate	60	23.2	D-100.0
2,4-Dinitrotoluene	60	21.8	47.5-126.9
2,6-Dinitrotoluene	60	29.6	68.1-136.7
Di-n-octylphthalate	60	31.4	18.6-131.8
Endosulfan sulfate[1]	60	16.7	D-103.5
Endrin aldehyde	60	32.5	D-188.8
Fluoranthene	60	32.8	42.9-121.3
Fluorene	60	20.7	71.6-108.4
Heptachlor[1]	60	37.2	D-172.2
Heptachlor epoxide[1]	60	54.7	70.9-109.4
Hexachlorobenzene	60	24.9	7.8-141.5
Hexachlorobutadiene	60	26.3	37.8-102.2
Hexachloroethane	60	24.5	55.2-100.0
Indeno(1,2,3-cd)pyrene	60	44.6	D-150.9
Isophorone	60	63.3	46.6-180.2
Naphthalene	60	30.1	35.6-119.6
Nitrobenzene	60	39.3	54.3-157.6
N-Nitrosodi-n-propylamine	60	55.4	13.6-197.9
PCB-1260[1]	60	54.2	19.3-121.0
Phenanthrene	60	20.6	65.2-108.7
Pyrene	60	25.2	69.6-100.0
1,2,4-Trichlorobenzene	60	28.1	57.3-129.2
4-Chloro-3-methylphenol	60	37.2	40.8-127.9
2-Chlorophenol	60	28.7	36.2-120.4
2,4-Chlorophenol	60	26.4	52.5-121.7
2,4-Dimethylphenol	60	26.1	41.8-109.0
2,4-Dinitrophenol	60	49.8	D-172.9
2-Methyl-4,6-dinitrophenol	60	93.2	53.0-100.0
2-Nitrophenol	60	35.2	45.0-166.7
4-Nitrophenol	60	47.2	13.0-106.5
Pentachlorophenol	60	48.9	38.1-151.8
Phenol	60	22.6	16.6-100.0
2,4,6-Trichlorophenol	60	31.7	52.4-129.2

[1]Since the organochlorine pesticides and PCBs are normally determined by method 8080 at Quanterra, they will not be included in the initial demonstration of capability for method 8270B.

ATTACHMENT A

MODIFICATIONS REQUIRED FOR ANALYSIS OF WASTEWATER FOLLOWING METHOD 625

18

REQUIREMENTS FOR METHOD 625

18.1 Method 625 is required for demonstration of compliance with NPDES wastewater discharge permits. The standard analyte list and reporting limits are listed in Table A-1.

18.2 This method can be applied only to aqueous matrices.

18.3 The tune period for this method is defined as 24 hours.

18.4 Initial calibration curve requirements:

18.4.1 The initial calibration curve for this method requires at least three points.

18.4.2 Target compounds must have RSD ≤ 35%.

18.4.3 If this requirement can not be met, a regression curve must be constructed for the non-compliant compounds.

18.5 Continuing calibration verification requirements: All target compounds must have %D ≤ 20%.

18.6 Matrix Spike and LCS requirements:

18.6.1 A full analyte spike is required for method 625. The spiking levels are given in Table A-2.

Table A-1. Quanterra Method 625 standard reporting list and reporting limits.

Analytes	CAS Number	Aqueous µg/L
Phenol	108-95-2	10
Bis(2-chloroethyl)ether	111-44-4	10
2-Chlorophenol	95-57-8	10
1,3-Dichlorobenzene	541-73-1	10
1,4-Dichlorobenzene	106-46-7	10
1,2-Dichlorobenzene	95-50-1	10
2,2'-oxybis(1-chloropropane)	108-60-1	10
N-Nitroso-di-n-propylamine	621-64-7	10
Hexachloroethane	67-72-1	10
Nitrobenzene	98-95-3	10
Isophorone	78-59-1	10
2-Nitrophenol	88-75-5	10
2,4-Dimethylphenol	105-67-9	10
Bis(2-chloroethoxy)methane	111-91-1	10
2,4-Dichlorophenol	120-83-2	10
1,2,4-Trichlorobenzene	120-82-1	10
Naphthalene	91-20-3	10
Hexachlorobutadiene	87-68-3	10
4-Chloro-3-methylphenol	59-50-7	10
Hexachlorocyclopentadiene	77-47-4	50
2,4,6-Trichlorophenol	88-06-2	10
2-Chloronaphthalene	91-58-7	10
Dimethyl phthalate	131-11-3	10
Acenaphthylene	208-96-8	10
Acenaphthene	83-32-9	10
2,4-Dinitrophenol	51-28-5	50
4-Nitrophenol	100-02-7	50
2,4-Dinitrotoluene	121-14-2	10
2,6-Dinitrotoluene	606-20-2	10
Diethylphthalate	84-66-2	10
4-Chlorophenyl phenyl ether	7005-72-3	10
Fluorene	86-73-7	10
4,6-Dinitro-2-methylphenol	534-52-1	50
N-Nitrosodiphenylamine	86-30-6	10
4-Bromophenyl phenyl ether	101-55-3	10
Hexachlorobenzene	118-74-1	10
Pentachlorophenol	87-86-5	50
Phenanthrene	85-01-8	10
Anthracene	120-12-7	10
Di-n-butyl phthalate	84-74-2	10
Fluoranthene	206-44-0	10
Benzidine	92-87-5	100
Pyrene	129-00-0	10
Butyl benzyl phthalate	85-68-7	10
3,3'-Dichlorobenzidine	91-94-1	50

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Analytes	CAS Number	Aqueous µg/L
Benzo(a)anthracene	56-55-3	10
Bis(2-ethylhexyl)phthalate	117-81-7	10
Chrysene	218-01-9	10
Di-n-octylphthalate	117-84-0	10
Benzo(b)fluoranthene	205-99-2	10
Benzo(k)fluoranthene	207-08-9	10
Benzo(a)pyrene	50-32-8	10
Indeno(1,2,3-cd)pyrene	193-39-5	10
Dibenz(a,h)anthracene	53-70-3	10
Benzo(g,h,i)perylene	191-24-2	10

Table A-2. Method 625 LCS and MS compounds and spike concentrations.

LCS Compounds	Spiking Level, ng/µL in extract[1]
Phenol	100
Bis(2-chloroethyl)ether	100
2-Chlorophenol	100
1,3-Dichlorobenzene	100
1,4-Dichlorobenzene	100
1,2-Dichlorobenzene	100
2,2'-oxybis(1-chloropropane)	100
N-Nitroso-di-n-propylamine	100
Hexachloroethane	100
Nitrobenzene	100
Isophorone	100
2-Nitrophenol	100
2,4-Dimethylphenol	100
Bis(2-chloroethoxy)methane	100
2,4-Dichlorophenol	100
1,2,4-Trichlorobenzene	100
Naphthalene	100
Hexachlorobutadiene	100
4-Chloro-3-methylphenol	100
Hexachlorocyclopentadiene	100
2,4,6-Trichlorophenol	100
2-Chloronaphthalene	100
Dimethyl phthalate	100
Acenaphthylene	100
Acenaphthene	100
2,4-Dinitrophenol	100
4-Nitrophenol	100
2,4-Dinitrotoluene	100
2,6-Dinitrotoluene	100
Diethylphthalate	100
4-Chlorophenyl phenyl ether	100
Fluorene	100
4,6-Dinitro-2-methylphenol	100
N-Nitrosodiphenylamine	100
4-Bromophenyl phenyl ether	100
Hexachlorobenzene	100
Pentachlorophenol	100
Phenanthrene	100
Anthracene	100
Di-n-butyl phthalate	100
Fluoranthene	100
Benzidine	100
Pyrene	100
Butyl benzyl phthalate	100
3,3'-Dichlorobenzidine	100
Benzo(a)anthracene	100
Bis(2-ethylhexyl)phthalate	100

LCS Compounds	Spiking Level, ng/µL in extract[1]
Chrysene	100
Di-n-octylphthalate	100
Benzo(b)fluoranthene	100
Benzo(k)fluoranthene	100
Benzo(a)pyrene	100
Indeno(1,2,3-cd)pyrene	100
Dibenz(a,h)anthracene	100
Benzo(g,h,i)perylene	100

[1] **Levels are 50 and 75 ng/µL if 2 µL injection is used**

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QUANTERRA STANDARD OPERATING PROCEDURE

TITLE: GAS CHROMATOGRAPHIC ANALYSIS BASED ON METHOD 8000B, 8021B, 8081A, 8082 and 8151A, SW-846

(SUPERSEDES: Revision 3)

Prepared by: Richard Burrows

Reviewed by: _____
Technology Standardization Committee

Approved by: _____
Director, Quality Assurance

Approved by: _____
Director, Health and Safety

Approved by: _____
Management

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1.

SCOPE AND APPLICATION

This SOP describes procedures for analysis of organic analytes by Gas Chromatography (GC). The procedures are based on SW-846 methodology and are applicable for measurements made to comply with the Resource Conservation and Recovery Act (RCRA). Individual analytes and methods are described in the appendices.

2. SUMMARY OF METHOD

In general, semivolatile analytes in aqueous samples are prepared for analysis using continuous or separatory funnel liquid / liquid extraction or solid phase extraction (SOP # CORP-OP-0001) Solid samples are prepared using sonication, Soxhlet or pressurized fluid extraction (SOP # CORP-OP-0001). Volatile analytes are prepared for analysis using purge and trap methodology (Appendix A).

After the initial preparation step, the sample is introduced to the GC and concentrations of target analytes are measured by the detector response within a defined retention time window, relative to the response to standard concentrations. Internal or external standardization procedures are used as specified in the method appendices.

3. DEFINITIONS

Definitions of terms used in this SOP may be found in the glossary of the Quality Assurance Management Plan (QAMP).

4. INTERFERENCES

Contamination by carryover can occur when a low concentration sample is analyzed after a high concentration sample. In addition, some purge and trap autosamplers are susceptible to port specific contamination. Co-elution of target analytes with non-targets can occur, resulting in false positives or biased high results. In particular, this is a problem with non-selective detectors such as the Flame Ionization Detector (FID). See the appendices for interferences specific to individual tests and suggested corrective actions.

5. SAFETY

5.1. Procedures shall be carried out in a manner that protects the health and safety of all Quanterra associates. The following requirements must be met:

Eye protection that satisfies ANSI Z87.1 (as per the Chemical Hygiene Plan), laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have become contaminated will be removed and discarded; other gloves will be cleaned immediately. Refer to the Quanterra Chemical Hygiene plan for a complete description of personal protection equipment.

The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the MSDS files maintained in the laboratory. Specific hazards are covered in the appendices.

5.1.1. Opened containers of neat standards will be handled in a fume hood.

6. EQUIPMENT AND SUPPLIES

An analytical system complete with a gas chromatograph is required. A data system capable of measuring peak area and/or height is required. Recommended equipment and supplies for individual methods are listed in each method appendix.

7. REAGENTS AND STANDARDS

7.1. Stock Standards

Stock standards are purchased as certified solutions or prepared from pure solutions. Stock standards for method 8021B are stored at -10 to -20°C. Other stock standard solutions are stored at ≤6°C. All stock standards must be protected from light. Stock standard solutions should be brought to room temperature before using.

Semivolatile stock standard solutions must be replaced after one year. Stock standards of gases must be replaced at t every week, unless the acceptability of the standard is demonstrated (Less than 20% drift from the initial calibration.). Other volatile stock standards must be replaced every 6 or sooner if comparison with check standards prepared from an independent source indicates a problem.

7.1.1. Expiration times for all standards are measured from the time the standard is prepared or from the time that the standard ampoule is opened, if the standard is supplied in a sealed ampoule. If a vendor supplied standard has an earlier expiration date then that date is used.

7.2. Calibration Standards

7.2.1. Volatile Calibration Standards

The procedure for preparation of volatile standards is given in Appendix A.

7.2.2. Semivolatile Calibration Standards

Semivolatile calibration standards are prepared as dilutions of the stock standards. Surrogates and internal standards are used as specified in the method appendices. Semivolatile calibration solutions must be refrigerated at ≤6°C and protected from light. The standards must be replaced at least every six months or sooner if comparison with check standards indicates a problem.

7.3. Gases for carrier and make-up: Hydrogen, Helium, Nitrogen, Argon/Methane.

7.4. Quality control (QC) Standards

QC standards (matrix spiking and LCS standards) are prepared and stored in the same way as calibration standards. They must be made from a stock independent from the calibration standards.

8. SAMPLE PRESERVATION AND STORAGE

Semivolatile extracts must be refrigerated at ≤6°C and analyzed within 40 days of the start of the extraction. Volatile sample storage conditions and holding times are given in Appendix A.

9. QUALITY CONTROL

9.1. Initial Demonstration of Capability

9.1.1. For the standard analyte list, the initial demonstration and method detection limit (MDL) studies described in section 13 must be acceptable before analysis of samples may begin.

9.1.2. For non-standard analytes, a MDL study must be performed and calibration curve generated before analyzing any samples, unless lesser requirements are previously agreed to with the client. In any event the minimum initial demonstration required is analysis of an extracted standard at the reporting limit and a single point calibration.

9.2. Batch Definition

Batches are defined at the sample preparation stage. Batches should be kept together through the whole analytical process as far as possible, but it is not mandatory to analyze prepared extracts on the same instrument or in the same sequence. Refer to the Quanterra QC Program document (QA-003) for further details of the batch definition.

9.2.1. Quality Control Batch

The batch is a set of up to 20 samples of the same matrix processed using the same procedures and reagents within the same time period. The Quality Control batch must contain a matrix spike / spike duplicate (MS/MSD), a Laboratory Control Sample (LCS), and a method blank. Laboratory generated QC samples (Blank, LCS, MS/MSD) do not count towards the maximum 20 samples in a batch. Field QC samples are included in the batch count. In some cases, at client request, the MS/MSD may be replaced with a matrix spike and sample duplicate. If insufficient sample is available for an MS/MSD a LCSD may be substituted.

9.3. Control Limits

In-house historical control limits must be determined for surrogates, matrix spikes, and laboratory control samples (LCS). These limits must be determined at least semi-annually. The recovery limits are mean recovery +/- 3 standard deviations, unless that limit is tighter than the calibration criteria, in which case limits may be widened. Refer to policy QA-003 for more details.

9.3.1. These limits do not apply to dilutions (except for tests without a separate extraction), but surrogate and matrix spike recoveries will be reported unless the dilution is more than 5X.

9.3.2. All surrogate, LCS, and MS recoveries (except for dilutions) must be entered into QuantIMS (when available) or other database so that accurate historical control limits can be generated. For tests without a separate extraction, surrogates and matrix spikes will be reported for all dilutions.

9.3.3. Refer to the QC Program document (QA-003) for further details of control limits.

9.4. Surrogates

All methods must use surrogates to the extent possible. Surrogate recoveries in samples and QC samples must be assessed to ensure that recoveries are within established limits. If any surrogates are outside limits, the following corrective actions must take place (except for dilutions):

- Check all calculations for error.
- Ensure that instrument performance is acceptable.
- Recalculate the data and/or reanalyze the extract if either of the above checks reveal a problem.
- Reprepare and reanalyze the sample or flag the data as "Estimated Concentration" if neither of the above resolves the problem. Repreparation is not necessary is there is obvious chromatographic interference.
- The decision to reanalyze or flag the data should be made in consultation with the client. It is only necessary to reprepare / reanalyze a sample once to demonstrate that poor surrogate recovery is due to matrix effect, unless the analyst believes that the repeated out of control results are not due to matrix effect.

9.4.1. If dual column analysis is used the choice of which result to report in made in the same way as for samples (Section 12.1.2) unless one column is out of control, in which case the in control result is reported.

9.4.2. If the surrogates are out of control for the sample, matrix spike, and matrix spike duplicate, then matrix effect has been demonstrated for that sample and repreparation is not necessary. If the sample is out of control and the MS and/or MSD is in control, then repreparation or flagging of the data is required.

9.4.3. Refer to the Quanterra QC Program document (QA-003) for further details of the corrective actions.

9.5. Method Blanks

For each batch of samples, analyze a method blank. The method blank consists of reagent water for aqueous semivolatiles samples, and sodium sulfate for semivolatiles soils tests (Refer to SOP No. CORP-OP-0001 for details). For low level volatiles, the method blank consists of reagent water. For medium level volatiles, the method blank consists of methanol as described in Appendix A. Surrogates are added and the method blank is carried through the entire analytical procedure. The method blank must not contain any analyte of interest at or above the reporting limit (except common laboratory contaminants, see below) or at or above 5% of the measured concentration of that analyte in the associated samples, whichever is higher.

If the analyte is a common laboratory contaminant (methylene chloride, acetone, 2-butanone, phthalate esters) the data may be reported with qualifiers if the concentration of the analyte is less than five times the reporting limit. Such action must be taken in consultation with the client.

Re-extraction and reanalysis of samples associated with an unacceptable method blank is required when reportable concentrations are determined in the samples.

If there is no target analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. Such action should be taken in consultation with the client.

9.5.1. Refer to the Quanterra QC Program document (QA-003) for further details of the corrective actions.

9.6. Instrument Blanks

9.6.1. An instrument blank must be analyzed during any 12 hour period of analysis that does not contain a method blank.

9.6.2. An instrument blank consists of the appropriate solvent with internal standards added. If internal standards are not used the surrogates should be added.

9.6.3. Control criteria are the same as for the method blank, except that only reanalysis of affected samples would be required, not re-extraction.

9.7. Laboratory Control Samples (LCS)

For each batch of samples, analyze a LCS. The LCS contains a representative subset of the analytes of interest, and must contain the same analytes as the matrix spike. The LCS may also contain the full set of analytes. If any analyte or surrogate is outside established control limits, the system is out of control and corrective action must occur. Corrective action will normally be repreparation and reanalysis of the batch; however, if the matrix spike and matrix spike duplicate are within limits, the batch may be acceptable.

9.7.1. Refer to the Quanterra QC Program document (QA-003) for further details of the corrective action.

9.7.2. If dual column analysis is used the choice of which result to report in made in the same way as for samples (Section 12.1.2) unless one column is out of control, in which case the in control result is reported.

9.7.3. LCS compound lists are included in the appendices.

9.7.4. If full analyte spike lists are used at client request, it will be necessary to allow a percentage of the components to be outside control limits as this would be expected statistically. These requirements should be negotiated with the client.

9.8. Matrix Spikes

For each QC batch, analyze a matrix spike and matrix spike duplicate. Spiking compounds and levels are given in the appendices. Compare the percent recovery and relative percent difference (RPD) to those in the laboratory specific historically generated limits.

- If any individual recovery or RPD falls outside the acceptable range, corrective action must occur. The initial corrective action will be to check the recovery of that analyte in the Laboratory Control Sample (LCS). Generally, if the recovery of the analyte in the LCS is within limits, then the laboratory operation is in control and analysis may proceed.
- If the recovery for any component is outside QC limits for both the Matrix spike / spike duplicate and the LCS, the laboratory is out of control and corrective action must be taken. Corrective action will normally include repreparation and reanalysis of the batch.
- If a MS/MSD is not possible due to limited sample, then a LCS duplicate should be analyzed.
- The matrix spike / duplicate must be analyzed at the same dilution as the unspiked sample, unless the matrix spike components would then be above the calibration range.

9.8.1. If dual column analysis is used the choice of which result to report in made in the same way as for samples (Section 12.1.2) unless one column is out of control, in which case the in control result is reported.

9.9. Quality Assurance Summaries

Certain clients may require specific project or program QC which may supersede these method requirements. Quality Assurance Summaries should be developed to address these requirements.

9.10. Quanterra QC Program

Further details of QC and corrective action guidelines are presented in the Quanterra QC Program document (QA-003). Refer to this document if in doubt regarding corrective actions.

10. CALIBRATION AND STANDARDIZATION

Internal or external calibration may be used. Internal calibration is recommended unless the sample matrix is likely to interfere with the quantitation of the internal standard. In either event prepare standards containing each analyte of interest at a minimum of five concentration levels. The low level standard should be at or below the reporting limit. The other standards define the working range of the detector. Recommended calibration levels are given in the appendices.

10.1. A new calibration curve must be generated after major changes to the system or when the continuing calibration criteria cannot be met. Major changes include new columns, changing PID lamps or FID jets or replacing the ECD detector. A new calibration is not required after clipping the column, replacing the septum or syringe, or other minor maintenance.

10.2. With the exception of 10.3 below, it is NOT acceptable to remove points from a calibration curve for the purpose of meeting criteria, unless the points are the highest or lowest on the curve AND the reporting limit and/or linear range is adjusted accordingly. In any event, at least 5 points must be included in the calibration curve. Quadratic (second order) calibrations require at least six points. Third order calibrations require at least seven points.

10.3. A level may be removed from the calibration if the reason can be clearly documented, for example a broken vial or no purge run. A minimum of five levels must remain in the calibration. The documentation must be retained with the initial calibration. Alternatively, if the analyst believes that a point on the curve is inaccurate, the point may be reanalyzed and the reanalysis used for the calibration. All initial calibration points must be analyzed without any changes to instrument conditions, and all points must be analyzed within 24 hours.

10.4. External standard calibration

Quantitation by the external standard method assumes a proportional relationship between the calibration run and the analyte in the sample. To use this approach introduce each calibration standard into the GC using the technique that will be used for samples. The ratio of the peak height or area response to the mass or concentration injected may be used to prepare a calibration curve.

$$Calibration\ Factor\ (CF) = \frac{Area\ or\ Height\ of\ Peak}{Mass\ Injected\ (ng)}$$

Some data systems may use the inverse of this formula. This is acceptable so long as the same formula is used for standards and samples. It is also possible to use the concentration of the standard rather than the mass injected. (This would require changes in the equations used to calculate the sample concentrations). Use of peak area or height must be consistent. However, if matrix interferences would make quantitation using peak area inaccurate for a particular sample, then peak height may be used as a substitute.

10.5. Internal standard calibration

10.5.1. The internal standard approach assumes that variations in instrument sensitivity, amount injected etc. can be corrected by determining the ratio of the response of the analyte to the response of an internal standard that has been added to the extract. To use this approach, select one or more internal standard(s) that are similar in analytical behavior to the compounds of interest. Recommended internal standards are given in the appendices. The analyst must demonstrate that the measurement of the internal standard is not affected by method or matrix interferences. If the sample matrix interferes with quantitation of the internal standard, then the external standard approach must be used instead. In this event use the response factors from the previous continuing calibration to quantitate the analytes in the sample with the interference (applies only to the sample with the interference).

10.5.2. Introduce each calibration standard into the GC using the technique that will be used for samples. Response factors (RF) for each compound are calculated as follows:

$$RF = \frac{A_s \times C_{is}}{A_{is} \times C_s}$$

Where:

A_s = Response for the analyte to be measured
A_{is} = Response for the internal standard
C_{is} = Concentration of internal standard
C_s = Concentration of the analyte to be determined in the standard

10.6. Calibration curve fits

Average response factor, linear regression, or quadratic curves may be used to fit the data. Average response factor may be used if the average % RSD of the response factors or calibration factors of all the analytes in the calibration standard taken together is ≤ 20%. The average %RSD is calculated by summing the RSD value for each analyte and dividing by the total number of analytes.

10.6.1. In general, for environmental analysis, average response factors are the most appropriate calibration model. Linear or curved regression fits should only be used if the analyst has reason to believe that the average RF model does not fit the normal concentration/response behavior of the detector.

10.6.2. Average response factor

The average response factor may be used if the average percent relative standard deviation (%RSD) of all the response factors taken together is ≤ 20%.

The equation for average response factor is:

$$Average\ response\ factor = \overline{RF} = \frac{\sum_{i=1}^{n} RF_i}{n}$$

Where: n = Number of calibration levels

$\sum_{i=1}^{n} RF_i$ = Sum of response factors for each calibration level

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10.6.3. Linear regression

The linear fit uses the following functions:

10.6.3.1. External Standard

$$y = ax + b$$

or

$$x = \frac{(y-b)}{a}$$

Where: y = Instrument response
x = Concentration
a = Slope
b = Intercept

10.6.3.2. Internal Standard

$$C_s = \frac{\left[\frac{A_s C_{is}}{A_{is}} - b\right]}{a}$$

Where: C_s = Concentration in the sample
A_s = Area of target peak in the sample
A_{is} = Area of internal standard in the sample
C_{is} = Concentration of the internal standard

10.6.4. Quadratic curve

The quadratic curve uses the following functions:

10.6.4.1. External standard

$$y = ax + cx^2 + b$$

Where c is the curvature

10.6.4.2. Internal Standard

$$y = a\left(\frac{A_s \times C_{is}}{A_{is}}\right) + c\left(\frac{A_s \times C_{is}}{A_{is}}\right)^2 + b$$

10.7. Evaluation of calibration curves

10.7.1. The percent relative standard error (%RSE) from the calibration curve is used to evaluate the initial calibration. This provides a measure of how much error is associated with using the calibration curve for quantitation.

10.7.2. The least squares regression line is calculated and used to calculate the predicted concentration for each level. The percent relative standard error is calculated as follows:

$$\% RSE = 100\% \times \sqrt{\frac{\sum_{i=1}^{N}\left[\frac{C_i - PC_i}{C_i}\right]^2}{(N-P)}}$$

Where:
N = Number of points in the curve
P = Number of parameters in the curve (= 1 for average response factor, 2 for linear, 3 for quadratic)
C_i = True concentration for level i
PC_i = Predicted concentration for level i

Note that when average response factors are used, %RSE is equivalent to %RSD.

10.8. The following requirements must be met for any calibration to be used:

- Response must increase with increasing concentration.
- If a curve is used, the intercept of the curve at zero response must be less than ± the reporting limit for the analyte.
- The average Relative Standard Error (RSD for average response factors) of the calibration points from the curve used must be ≤ 20%.
- Some data systems will not measure the %RSE from a linear or quadratic fit. For the linear case, the correlation coefficient may be used as an alternative to the %RSE, and must be greater than or equal to 0.990. For the quadratic case the Coefficient of Determination may be used, and must be greater or equal to 0.990.

Note: The Relative Standard Error (RSE) is superior to the Correlation Coefficient (r^2) and Coefficient of Determination (r) for testing the fit of a set of calibration points to a line. The lower points on a curve have little effect on r. As a result a curve may have a very good correlation coefficient (>0.995), while also having > 100% error at the low point. (Edgerley, "Improving Calibration Accuracy in the Environmental Laboratory", in press).

10.9. Weighting of data points

10.9.1. In a quadratic calibration fit, the points at the lower end of the calibration curve have less relative variance than points at the high concentration end of the curve. This can cause severe errors in quantitation at the low end of the calibration. However, in environmental analysis, accuracy at the low end of the curve is very important. For this reason it is preferable to increase the weighting of the lower concentration points. 1/Concentration^2 weighting (often called $1/X^2$ weighting) will improve accuracy at the low end of the curve and should be used if the data system has this capability. Use of $1/X^2$ weighting for linear fits is currently not permitted by method 8000B.

10.10. Non-standard analytes are sometimes requested. For these analytes, it may be acceptable to analyze a single standard at the reporting limit with each continuing calibration rather than a five point initial calibration. This action must be with client approval. If the analyte is detected in any of the samples, a five point initial calibration must be generated and the sample(s) reanalyzed for quantitation.

10.11. Calibration Verification

10.11.1. 12 hour Calibration

The working calibration curve or RF must be verified by the analysis of a mid point calibration standard at the beginning, after every 12 hours, and at the end of the analysis sequence. The center of each retention time window is updated with each 12 hour calibration.

10.11.2. Calibration Verification

It may be appropriate to analyze a mid point standard more frequently than every 12 hours. If these calibration verification standards are analyzed, requirements are the same as the 12 hour calibration with the exception that retention times are not updated.

10.11.3. Any analyte that is reportable as found must have a % difference of $\leq$ 15% in the preceding verification or 12 calibration, on the column used for quantitation. Refer to section 12.1.2 for which result to report.

10.11.4. Analytes not detected in client samples will ideally also have a %D of $\leq$ 15%. However, the analysis is acceptable if the average of the %D for all the analytes is $\leq$ 15%. This average is calculated by summing all the %D results in the calibration and dividing by the number of analytes.

10.11.5. Reportable as found is defined as any analyte that would be reported as anything other than a non-detect.

10.11.6. It is not necessary to run a calibration verification standard at the beginning of the sequence if samples are analyzed immediately after the completion of the initial calibration.

10.11.7. Samples quantitated by external standard methods must be bracketed by calibration verification standards that meet the criteria listed above. Bracketing is not necessary for internal standard methods.

10.11.8. % Difference calculation

% Difference for internal and external methods is calculated as follows

Internal Standard:

$$\%D = \frac{RF_c - \overline{RF}}{\overline{RF}} \times 100$$

External standard:

$$\%D = \frac{CF_c - \overline{CF}}{\overline{CF}} \times 100$$

Where RF_c and CF_c are the response and calibration factors from the continuing calibration

$\overline{RF}$ and $\overline{CF}$ are the average response and calibration factors from the initial calibration

10.11.9. % Drift calculation

% Drift is used for comparing the continuing calibration to a linear or quadratic curve. The criteria for % drift are the same as for % difference

$$\%\ Drift = \frac{Calculated\ Conc.\ -\ Theoretical\ Conc.}{Theoretical\ Conc.} \times 100\%$$

10.11.10. Corrective Actions for Continuing Calibration

If the overall average %D of all analytes is greater than ± 15% corrective action must be taken. This may include clipping the column, changing the liner or other minor instrument adjustments, followed by reanalyzing the standard. If the overall average %D still varies by more than ± 15%, a new calibration curve must be prepared.

10.11.11. Corrective Action for Samples

For internal standard methods, any samples injected after a standard not meeting the calibration criteria must be reinjected.

For external standard methods, any samples injected after the last good continuing calibration standard must be reinjected.

11. PROCEDURE

11.1. Extraction

Extraction procedures are referenced in the appendices.

11.2. Cleanup

Cleanup procedures are referenced in the appendices.

11.3. Gas Chromatography

Chromatographic conditions for individual methods are presented in the appendices.

11.4. Sample Introduction

In general, volatiles analytes are introduced using purge and trap as described in Appendix A. Semivolatile analytes are introduced by direct injection of the extract. Samples, standards, and QC must be introduced using the same procedure.

11.5. Analytical Sequence

An analytical sequence starts with an initial calibration or a daily calibration. Refer to the individual method appendices for method specific details of daily calibrations and analytical sequences.

11.5.1. The daily calibration includes analysis of standards containing all single response analytes and updating the retention time windows.

11.5.2. If there is a break in the analytical sequence of greater than 12 hours, a new analytical sequence must be started with a daily calibration.

11.6. Retention Time Windows

11.6.1. Retention time windows must be determined for all analytes. Make an injection of all analytes of interest each day over a three day period. Calculate the standard deviation of the three retention times for each analyte (relative retention times may also be used). For multiresponse analytes (e.g., Aroclors) use the retention time of major peaks. Plus or minus three times the standard deviation of the retention times of each analyte defines the retention time window.

11.6.2. The center of the retention time window is the retention time from the last of the three standards. The centers of the windows are updated with the mid point of the initial calibration and each 12 hour calibration. The widths of the windows will remain the same until new windows are generated following the installation of a new column.

11.6.3. If the retention time window as calculated above is less than +/- 0.05 minutes, use +/- 0.05 minutes as the retention time window. This allows for slight variations in retention times caused by sample matrix.

11.6.4. The laboratory must calculate new retention time windows each time a new column is installed. The new windows must be generated within one week of the installation of the new column. Until these standards have been run on the new column, the retention time windows from the old column may be used, updated with the retention times from the new initial calibration.

11.6.5. Corrective Action for Retention Times

The retention times of all compounds in each continuing calibration must be within the retention time windows established by the 12 hour calibration. If this condition is not met, all samples analyzed after the last compliant standard must be reanalyzed unless the following conditions are met for any compound that elutes outside the retention time window:

The retention time of that compound in the standard must be within a retention time range equal to twice the original window.

No peak that would be reportable may be present on the sample chromatogram within an elution time range equal to three times the original retention time window.

11.7. Daily Retention Time Windows

The center of the retention time windows determined in section 11.6 are adjusted to the retention time of each analyte as determined in the 12 hour calibration standards. (See the method 8081A and 8082 appendices for exceptions for multi-response components.) The retention time windows must be updated at the beginning of each analytical sequence and with each 12 hour calibration, but not for any other calibration verification standards.

11.8. Percent Moisture

Analytical results may be reported as dry or wet weight, as required by the client. Percent moisture must be determined if results will be reported as dry weight. Refer to SOP CORP-OP-0001 for determination of percent moisture.

11.9. Procedural Variations

Procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and approved by a supervisor and QA/QC manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file. The nonconformance is also addressed in the case narrative. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Qualitative Identification

12.1.1. Tentative identification occurs when a peak is found within the retention time window for an analyte, at a concentration above the reporting limit, or above the MDL if J flags are required. Normally confirmation is required on a second column, but if the detector is sufficiently specific or if the sample matrix is well enough defined, single column analysis may be adequate. In some cases GC/MS confirmation may be required. Client specific requirements may also define the need for second column confirmation and / or GC/MS confirmation. Refer to the appendices for test specific requirements for confirmation. Identification is confirmed if a peak is also present in the retention time window for that analyte on the confirmatory column, at a concentration greater than the reporting limit (MDL if J flag confirmation required).

12.1.2. Dual column quantitation

For confirmed results, two approaches are available to the analyst;
A) The primary column approach
Or
B) The better result approach
Both are acceptable to avoid the reporting of erroneous or unconfirmed data.

12.1.2.1. Primary column approach:

The result from the primary column is normally reported. The result from the secondary column is reported if any of the following three bulleted possibilities are true.

- There is obvious chromatographic interference on the primary column
- The result on the primary column is > 40% greater than the result on the secondary column
- Continuing or bracketing standard fails on the primary column but is acceptable on the secondary column. (If the primary column result is > 40% higher than the secondary, and the primary column calibration fails, then the sample must be evaluated for reanalysis.)

12.1.2.2. Better result approach

The lower of the two results is normally reported. The lower result is considered better because the higher result is generally higher because of chromatographic interference. The higher result is reported if any of the following two bulleted possibilities are true.

- There is obvious chromatographic interference on the column with the lower result
- The continuing or bracketing calibration on the column with the lower result fails. (If the higher result is > 40% higher and the calibration on the column with the lower result fails, then the sample must be evaluated for reanalysis.)

12.1.3. If the Relative percent difference (RPD) between the response on the two columns is greater than 40%, or if the opinion of an experienced analyst is that the complexity of the matrix is resulting in false positives, the confirmation is suspect and the results are qualified. RPD is calculated using the following formula:

$$RPD = \frac{R_1 - R_2}{\frac{1}{2}(R_1 + R_2)}$$

Where R=Result

12.1.4. Multi-response Analytes

For multi-response analytes, the analyst should use the retention time window, but should rely primarily on pattern recognition. The pattern of peaks will normally serve as confirmation.

12.1.5. The experience of the analyst should weigh heavily in the interpretation of the chromatogram. For example, sample matrix or laboratory temperature fluctuation may result in variation of retention times.

12.2. Calibration Range

If concentrations of any analytes exceed the working range as defined by the calibration standards, then the sample must be diluted and reanalyzed. Dilutions should target the most concentrated analyte in the upper half (over 50% of the high level standard) of the calibration range. It may be necessary to dilute samples due to matrix.

12.3. Dilutions

Samples may be screened to determine the appropriate dilution for the initial run. If the initial diluted run has no hits or hits below 20% of the calibration range and the matrix allows for analysis at a lesser dilution, then the sample must be reanalyzed at a dilution targeted to bring the largest hit above 50% of the calibration range.

12.3.1. Guidance for Dilutions Due to Matrix

If the sample is initially run at a dilution and only minor matrix peaks are, then the sample should be reanalyzed at a more concentrated dilution. Analyst judgement is required to determine the most concentrated dilution that will not result in instrument contamination.

12.3.2. Reporting Dilutions

The most concentrated dilution with no target compounds above the calibration range will be reported. Other dilutions will only be reported at client request.

12.4. Interferences

If peak detection is prevented by interferences, further cleanup should be attempted. If no further cleanup is reasonable, then elevation of reporting levels and/or lack of positive identification must be addressed in the case narrative.

12.5. Internal Standard Criteria for Continuing Calibration

If internal standard calibration is used, then the internal standard response in a continuing calibration standard must be within 50 to 150% of the response in the mid level of the initial calibration.

12.6. Calculations

Capabilities of individual data systems may require the use of different formulas than those presented here. When this is the case, the calculations used must be shown to be equivalent and must be documented in an appendix attached to this document.

12.6.1. External Standard Calculations

12.6.1.1. Aqueous samples

$$Concentration\ (mg\ /\ L) = \frac{(A_x \times V_t \times D_f)}{(CF \times V_i \times V_s)}$$

Where:

A_x = Response for the analyte in the sample
V_i = Volume of extract injected, µL
D_f = Dilution factor
V_t = Volume of total extract, µL
V_s = Volume of sample extracted or purged, mL
CF = Calibration factor, area or height/ng, Section 10.1

12.6.1.2. Non-aqueous Samples

$$Concentration\ (mg\ /\ kg) = \frac{(A_x \times V_t \times D_f)}{(CF \times V_i \times W \times D)}$$

Where:

W = Weight of sample extracted or purged, g

$$D = \frac{100 - \%\ Moisture}{100}$$ (D = 1 if wet weight is required)

12.6.2. Internal Standard Calculations

12.6.2.1. Aqueous Samples

$$Concentration\ (mg\ /\ L) = \frac{(A_x \times C_{is} \times D_f)}{(A_{is} \times RF \times V_s)}$$

Where:

C_{is} = Amount of internal standard added, ng
A_{is} = Response of the internal standard
RF = Response factor for analyte

12.6.2.2. Non-aqueous Samples

$$Concentration\ (mg\ /\ kg) = \frac{(A_x \times C_{is} \times D_f)}{(A_{is} \times RF \times W \times D)}$$

12.6.3. Surrogate Recovery

Concentrations of surrogate compounds are calculated using the same equations as for the target compounds. The response factor from the initial calibration is used. Surrogate recovery is calculated using the following equation:

$$\%\ \text{Recovery} = \frac{Concentration\ (or\ amount)\ found}{Concentration\ (or\ amount)\ spiked} \times 100$$

13. METHOD PERFORMANCE

13.1. Method Detection Limit

Each laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in QA Policy #: QA-005.

13.2. Initial Demonstration

Each laboratory must make a one time initial demonstration of capability for each individual method. Demonstration of capability for both soils and water matrices is required. This requires analysis of QC check samples containing all of the standard analytes for the method. For some tests it may be necessary to use more than one QC check mix to cover all analytes of interest.

13.2.1. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to a mid level calibration.

13.2.2. Calculate the average recovery and standard deviation of the recovery for each analyte of interest. Compare these results with the acceptance criteria given in each appendix.

13.2.3. If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

13.3. Training Qualification

The group/team leader has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

14. POLLUTION PREVENTION

This method does not contain any specific modifications that serve to minimize or prevent pollution.

15. WASTE MANAGEMENT

Waste generated in this procedure will be segregated and disposed according to the facility hazardous waste procedures. The Environmental Health and Safety Director should be contacted if additional information is required.

16. REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition, Final Update III, December 1996, Section 8000B

17. MISCELLANEOUS

17.1. Modifications from Reference Method

17.1.1. Chapter 1 of SW-846 states that the method blank should not contain any analyte of interest at or above the Method Detection Limit. This SOP states that the Method Blank must not contain any analyte of interest at or above the reporting limit. Common lab contaminants are allowed to be up to 5 times the reporting limit in the blank following consultation with the client.

17.2. Modifications from Previous Revision

This SOP has been substantially revised to reflect changes to Method 8000 contained in Update III to SW-846.

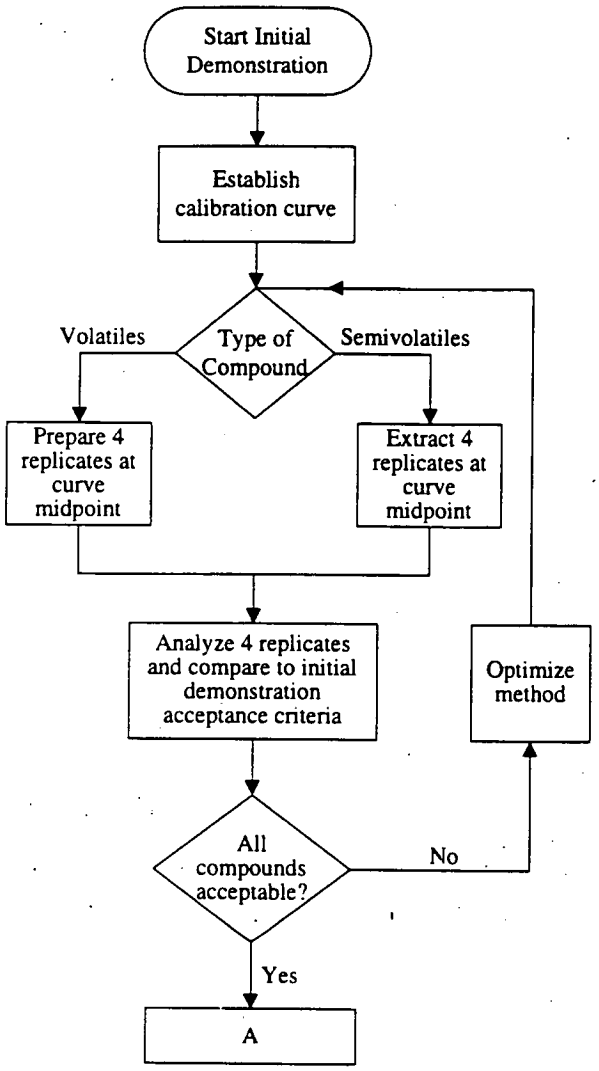
17.3. Facility Specific SOPs

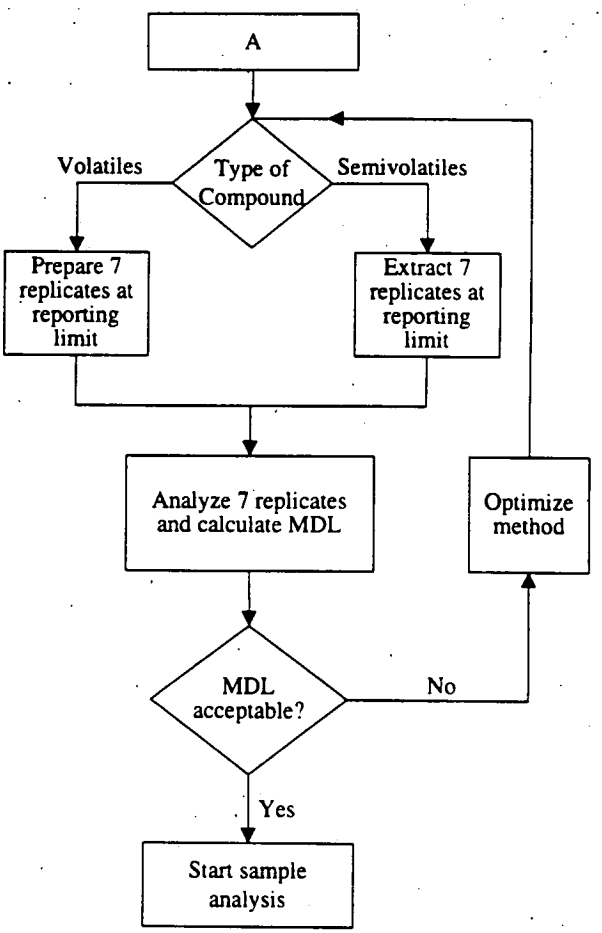
Each facility shall attach a list of facility specific SOPs or approved attachments (if applicable) which are required to implement this SOP or which are used in conjunction with this SOP. If no facility specific SOPs or amendments are to be attached, a statement must be attached specifying that there are none.

17.4.

Flow Diagrams

17.4.1. Initial demonstration and MDL[1]

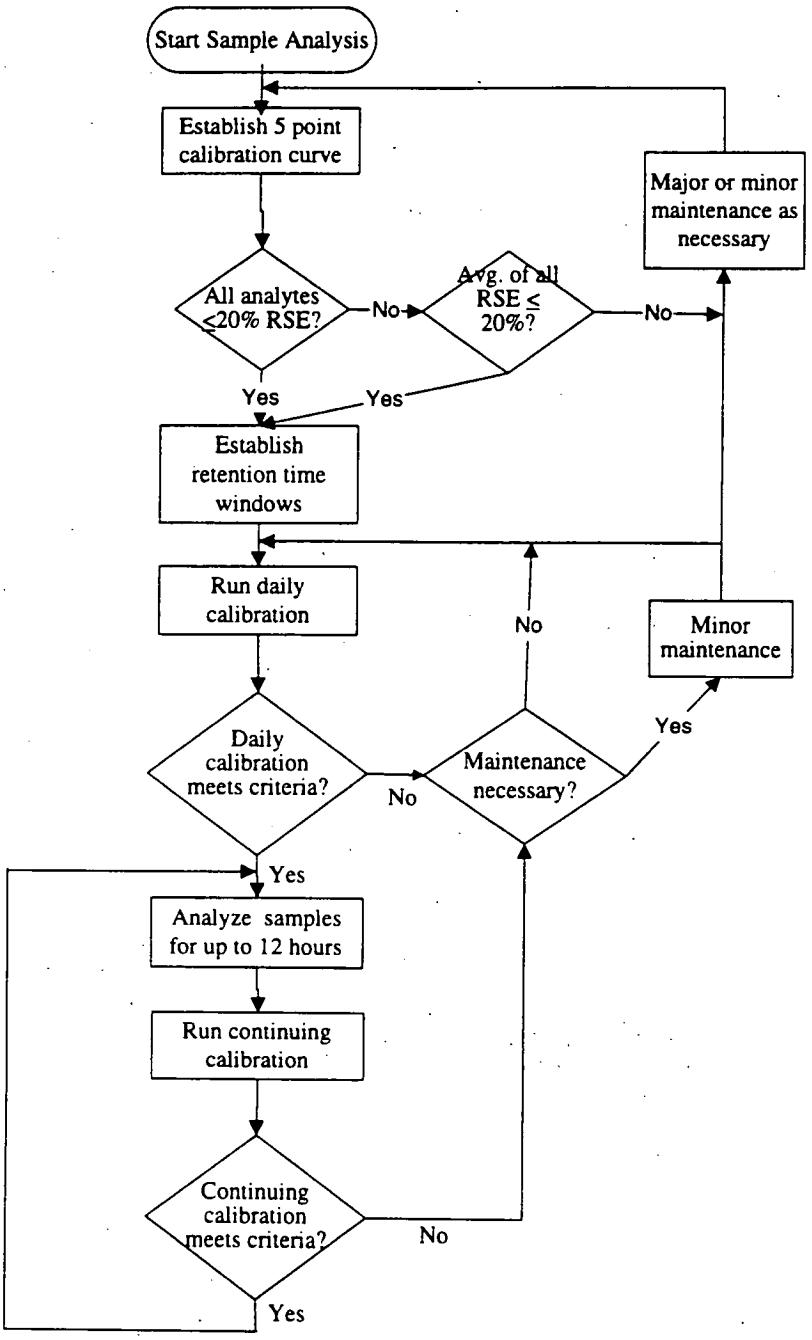




17.4.2.

[1] This flow diagram is for guidance and cannot cover all eventualities. Consult the SOP text and a supervisor if in doubt.

Sample Analysis[1]



[1] This flow diagram is for guidance and cannot cover all eventualities. Consult the SOP text and a supervisor if in doubt.

1. SCOPE AND APPLICATION

1.1. This method describes sample preparation and extraction for the analysis of volatile organics by a purge and trap procedure, following method 8021B. However, where required by a client QAPP this section may also be used to analyze aromatic volatiles by discontinued methods 8020A and 8010B. All requirements of the 8000B section of this SOP must be met except when superseded by this Appendix. Refer to Table A-1 for the individual analytes normally determined by these procedures.

1.2. Compounds within the scope of this method have boiling points below 200°C and are soluble or slightly soluble in water. Classes of compounds best suited to purge-and-trap analysis include low molecular weight halogenated hydrocarbons, aromatics, ketones, nitriles, acetates, acrylates, ethers, and sulfides.

1.3. Water samples and soils samples with low levels of contamination may be analyzed directly by purge-and-trap extraction and gas chromatography. Higher concentrations of these analytes in soil may be determined by the medium level methanol extraction procedure.

1.4. This method also describes the preparation of water-miscible liquids, non-water-miscible liquids, solids, wastes, and soils/sediments for analysis by the purge-and-trap procedure.

2. SUMMARY OF METHOD

2.1. An inert gas is bubbled through the sample at ambient temperature or at 40°C (40°C required for low level soils), and the volatile components are transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column where the volatile components are adsorbed. After purging is completed, the sorbent column is heated and backflushed with inert gas to desorb the components onto a gas chromatographic column. Analytes are detected using a photoionization Detector, an electrolytic conductivity detector or a combination of both.

2.2. For soil samples, a portion of the sample is dispersed in methanol to dissolve the volatile organic constituents. A portion of the methanolic solution is combined with water. It is then analyzed by purge-and-trap GC following the normal water method. If very low detection limits are needed for soil samples then direct purge using sodium bisulfate preservation may be necessary.

3. DEFINITIONS

Refer to the QAMP for definitions of terms used in this SOP.

4. INTERFERENCES

4.1. Refer to section 4 of the method 8000B part of this SOP for general information on chromatographic interferences.

4.2. Impurities in the purge gas, and from organic compounds out-gassing from the plumbing ahead of the trap, account for the majority of contamination problems. The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running laboratory reagent blanks. The use of non-TFE plastic tubing, non-TFE thread sealants, or flow controllers with rubber components in the purging device should be avoided.

4.3. Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) through the septum seal of the sample vial during shipment and storage. A trip blank prepared from organic-free reagent water and carried through sampling and handling protocols serves as a check on such contamination.

4.4. Contamination by carryover can occur whenever high-concentration and low-concentration samples are analyzed sequentially. Whenever an unusually concentrated sample is analyzed, it should be followed

by an analysis of organic-free reagent water to check for cross-contamination. The trap and other parts of the system are subject to contamination. Therefore, frequent bake-out and purging of the system may be required.

4.5. When utilizing an autosampler system which has multiple ports for sample analysis, it is likely that only a single stage or port may be contaminated by a highly concentrated sample. If a port is suspect, a water blank should be analyzed to verify lack of contamination. If the water blank and subsequent blanks on that port show contamination consistent with the concentrated sample, further maintenance is required. This may include replacing or cleaning the multi-port valve, transfer lines, etc.

4.6. A holding blank is kept in the sample refrigerator. This is analyzed and replaced every 14 days. If the holding blank does not meet the method blank criteria, the source of contamination must be found and corrected. Evaluation of all samples analyzed in the 14 day period prior to the analysis of the contaminated holding blank is required.

4.7. Acidification of samples may result in hydrolysis of 2-chloroethyl vinyl ether.

5. SAFETY

5.1. Refer to section 5 of the Method 8000B section of this SOP for general safety requirements.

5.2. Often, purge vessels on purge-and-trap instrumentation are pressurized by the time analysis is completed. Therefore, vent the pressure prior to removal of these vessels to prevent the contents from spraying out.

5.3. The toxicity or carcinogenicity of each chemical used in this procedure has not been fully defined. Additional health and safety information can be obtained from the MSDS files maintained in the laboratory. The following specific hazards are known:

Methanol -- Flammable and toxic

6. EQUIPMENT AND SUPPLIES

6.1. Microsyringes -- 10µL, 25µL, 100µL, 250µL, 500µL, and 1000µL. These should be equipped with a 20 gauge (0.006" ID) needle. These will be used to measure and dispense methanolic solutions and aqueous samples.

6.2. Gas tight syringes -- 5 mL and 25 mL. Used for measuring sample volumes.

6.3. Purge and Trap Apparatus -- A device capable of extracting volatile compounds, trapping on a sorbent trap, and introducing onto a gas chromatograph.

6.4. Purge and Trap Autosampler -- In order to maintain high sample throughput, an autosampler is highly recommended.

6.5. Trap -- The trap used is dependent on the class of compound to be analyzed. Refer to Table A-2 for suggested traps for specific tests.

6.6. Purge Vessels -- These are dependent on the purge and trap unit/autosampler used. Both disposable culture tubes (needle sparge units) and specially designed vessels with fritted bottoms may be used. Follow the manufacturer's suggestions for configuration.

6.7. Columns - Refer to Table A-2 for details of columns.

6.8. Volumetric flasks, Class A: 5 mL to 250 mL

6.9. pH paper

6.10. Balance capable of weighing to 0.01g for samples.

7. REAGENTS AND SUPPLIES

7.1. Refer to the method 8000B section of this SOP for general requirements for reagents and supplies.

7.2. Organic Free Water

Organic free water is defined as water in which an interferent is not observed at the reporting limit of the compounds of interest. Suggested methods for generating organic free water include:

- Filtration through a carbon bed.
- Continuously sparging water with helium or nitrogen.
- Use of commercial water purification systems.

Other methods may be used, so long as the requirement that the water not show any interferences is met. The procedure used should be documented in a lab specific attachment.

7.3. Sodium Bisulfate

7.4. Methanol -- Purge and Trap Grade

7.5. Standards

Refer to tables A-5 and A-6 for details of surrogate, matrix spiking and internal standards. Calibration standard levels are not specified, since they may depend on the sensitivity and linear range of specific detectors. However, the low level standard must be equivalent to the reporting limits specified in Table A-1.

7.5.1. Volatile standards are prepared by injecting a measured volume of the stock standard into a syringe containing the appropriate volume of organic free water. The calibration standard is then loaded into the purge device.

8. SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1. Holding times for all volatile analysis are 14 days from sample collection.

8.2. Water samples are normally preserved at pH < 2 with 1:1 hydrochloric acid. If residual chlorine is present, 2 drops of 10% sodium thiosulfate are added.

8.3. Solid samples are field preserved with sodium bisulfate solution for low level analysis, or with methanol for medium level analysis. Soil samples can also be taken using the EnCore™ sampler and preserved in the lab within 48 hours of sampling. At specific client request, unpreserved soil samples may be accepted.

8.4. There are several methods of sampling soil. The recommended method, which provides the minimum of field difficulties, is to take a EnCore sample. (The 5 g or 25 g sampler can be used, depending on client preference). Following shipment back to the lab the soil is preserved in methanol. This is the medium level procedure. If very low detection limits are needed (< 50 µg/kg for most analytes) then it will be necessary to use two additional 5 g EnCore samplers or to use field preservation.

8.5. Sample collection for medium level analysis using EnCore samplers.

8.5.1. Ship one 5 g (or 25 g) EnCore sampler per field sample position.

8.5.2. An additional bottle must be shipped for percent moisture determination.

8.5.3. When the samples are returned to the lab, extrude the (nominal) 5g (or 25 g) sample into a tared VOA vial containing 5 mL methanol (25 mL methanol for the 25 g sampler). Obtain the weight of the soil added to the vial and note on the label.

8.5.4. Add the correct amount of surrogate spiking mixture. (Add 100 µL of 250 µg/mL solution for a nominal 25 g sample, 20µL for a nominal 5 g sample.)

8.5.5. Add the correct amount of matrix spiking solution to the matrix spike and matrix spike duplicate samples. (Add 100 µL of 250 µg/mL solution for a nominal 25 g sample, 20µL for a nominal 5 g sample.) The addition of spike introduces a slight error, (0.4%) which can be neglected, into the calculations.

8.5.6. Prepare an LCS for each batch by adding the correct amount of matrix spiking solution to clean methanol. (100 µL of spike to 25 mL methanol or 20 µL spike to 5 mL methanol).

8.5.7. Shake the samples for two minutes to distribute the methanol throughout the soil.

8.5.8. Allow to settle, then remove a portion of methanol and store in a clean Teflon capped vial at 4+2°C until analysis.

8.6. Sample collection for medium level analysis using field methanol preservation

8.6.1. Prepare a VOA vial by adding 5 mL purge and trap grade methanol. (If a 25 g sample is to be used, add 25 mL methanol to the VOA vial).

8.6.2. Seal the bottle and attach a label.

8.6.3. Weigh the bottle to the nearest 0.01g and note the weight on the label.

8.6.4. Ship with appropriate sampling instructions.

8.6.5. Each sample will require an additional bottle with no preservative for percent moisture determination.

8.6.6. At client request, the methanol addition and weighing may also be performed in the field.

8.6.7. When the samples are returned to the lab, obtain the weight of the soil added to the vial and note on the label.

8.6.8. Add the correct amount of surrogate spiking mixture. (Add 100 µL of 250 µg/mL solution for a nominal 25 g sample, 20µL for a nominal 5 g sample.)

8.6.9. Add the correct amount of matrix spiking solution to the matrix spike and matrix spike duplicate samples. (Add 100 µL of 250 µg/mL solution for a nominal 25 g sample, 20µL for a nominal 5 g sample.) The addition of spike introduces a slight error, (0.4%) which can be neglected, into the calculations.

8.6.10. Prepare an LCS for each batch by adding the correct amount of matrix spiking solution to clean methanol. (100 µL of spike to 25 mL methanol or 20 µL spike to 5 mL methanol).

8.6.11. Shake the samples for two minutes to distribute the methanol throughout the soil.

8.6.12. Allow to settle, then remove a portion of methanol and store in a clean Teflon capped vial at 4+2°C until analysis.

8.7. Low level procedure

8.7.1. If low detection limits are required (typically < 50 µg/kg) sodium bisulfate preservation must be used. However, it is also necessary to take a sample for the medium level (methanol preserved) procedure, in case the concentration of analytes in the soil is above the calibration range of the low-level procedure.

8.7.2. A purge and trap autosampler capable of sampling from a sealed vial is required for analysis of samples collected using this method. (Varian Archon or O.I. 4552).

8.7.3. The soil sample is taken using a 5g EnCore sampling device and returned to the lab. It is recommended that two EnCore samplers be used for each field sample position, to allow for any reruns than may be necessary. A separate sample for % moisture determination is also necessary.

8.7.4. Prepare VOA vials by adding a magnetic stir bar, approximately 1 g of sodium bisulfate and 5 mL of reagent water.

8.7.5. Seal the vial and attach a label. The label must not cover the neck of the vial or the autosampler will malfunction.

8.7.6. Weigh the vial to the nearest 0.01g and note the weight on the label.

8.7.7. Extrude the soil sample from the EnCore sampler into the prepared VOA vial. Reweigh the vial to obtain the weight of soil and note on the label.

8.7.8. **Note:** Soils containing carbonates may effervesce when added to the sodium bisulfate solution. If this is the case at a specific site, add 5 mL of water instead, and freeze at ≥ -10°C until analysis.

8.7.9. Alternatively the sodium bisulfate preservation may be performed in the field. Ship at least two vials per sample. The field samplers must determine the weight of soil sampled. Each sample will require an additional bottle with no preservative for percent moisture determination, and an additional bottle preserved with methanol for the medium level procedure.

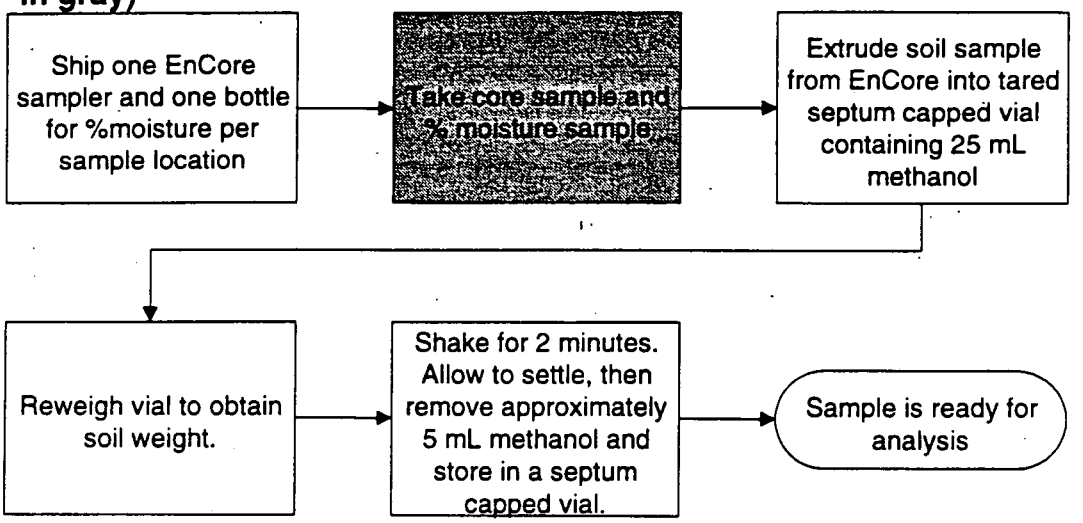
8.8. Aqueous samples are stored in glass containers with Teflon lined septa at 4°C +/- 2°C, with minimum headspace.

8.9. Medium level solid extracts are aliquoted into 2 - 5 mL glass vials with Teflon lined caps and stored at 4°C +/- 2°C. The extracts are stored with minimum headspace.

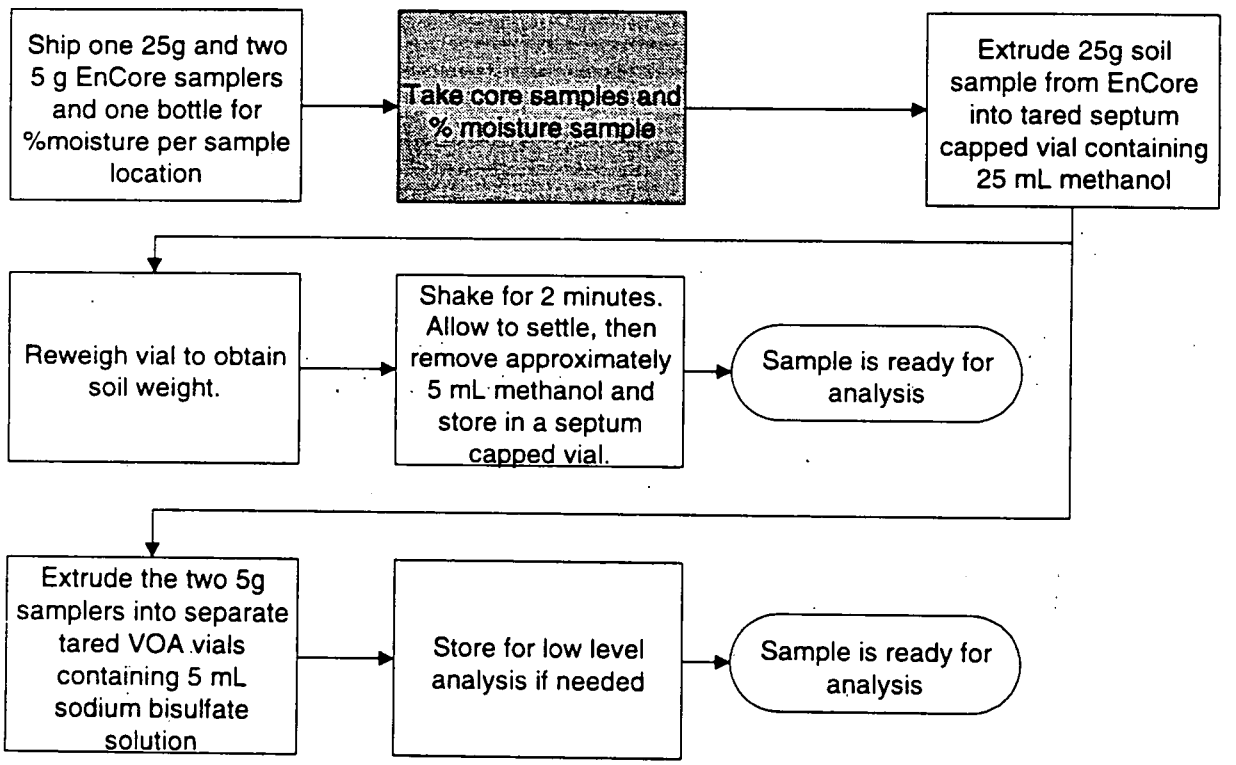
8.10. The maximum holding time is 14 days from sampling until the sample is analyzed. (Samples that are found to be unpreserved still have a 14 day holding time. However they should be analyzed as soon as possible. The lack of preservation should be addressed in the case narrative). Maximum holding time for the EnCore sampler (before the sample is added to methanol or sodium bisulfate) is 48 hours.

8.11. A holding blank is stored with the samples. This is analyzed and replaced if any of the trip blanks show any contamination. Otherwise it is replaced every 14 days.

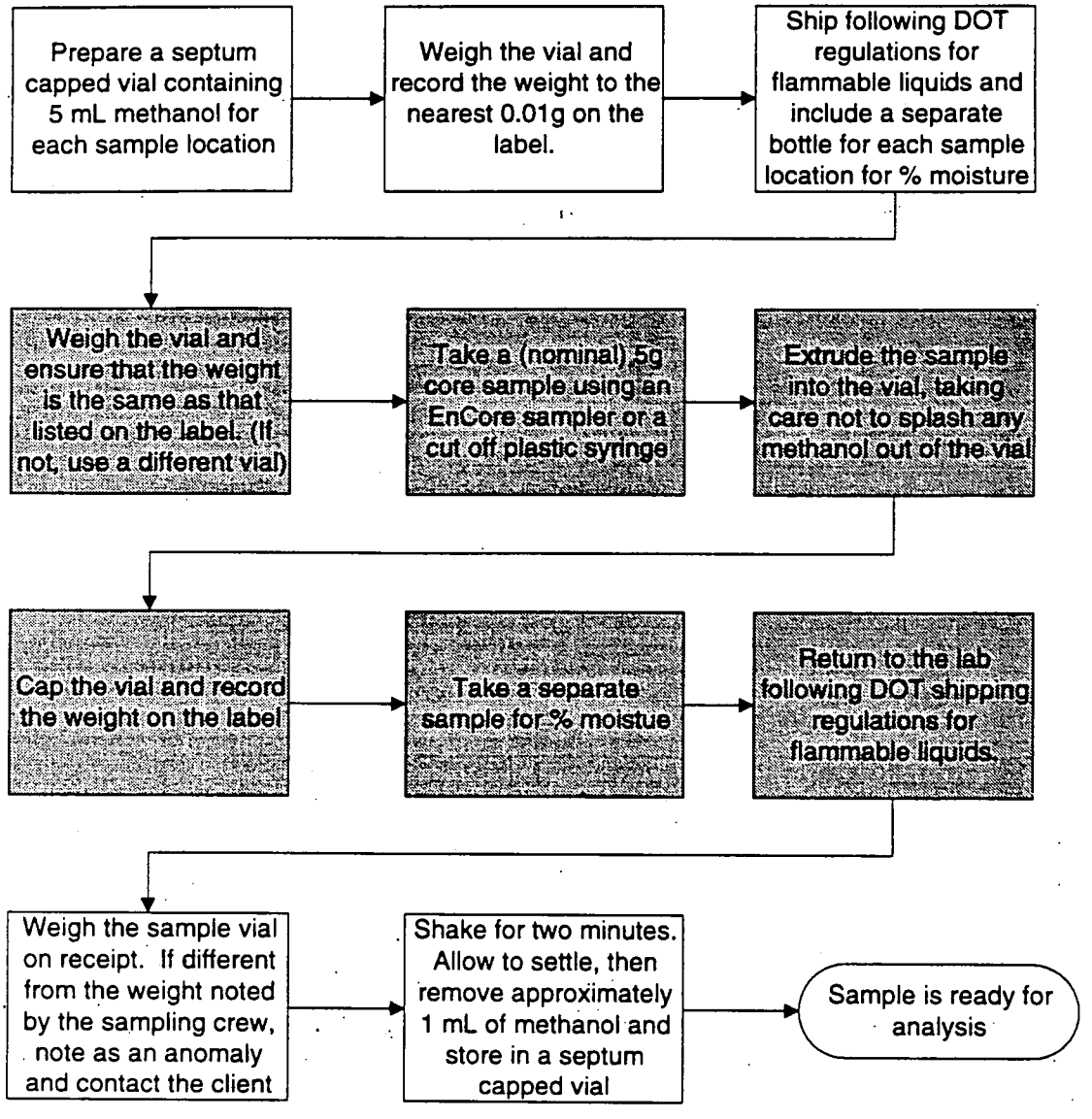
EnCore procedure when low level is not required (field steps in gray)



EnCore procedure when low level is required

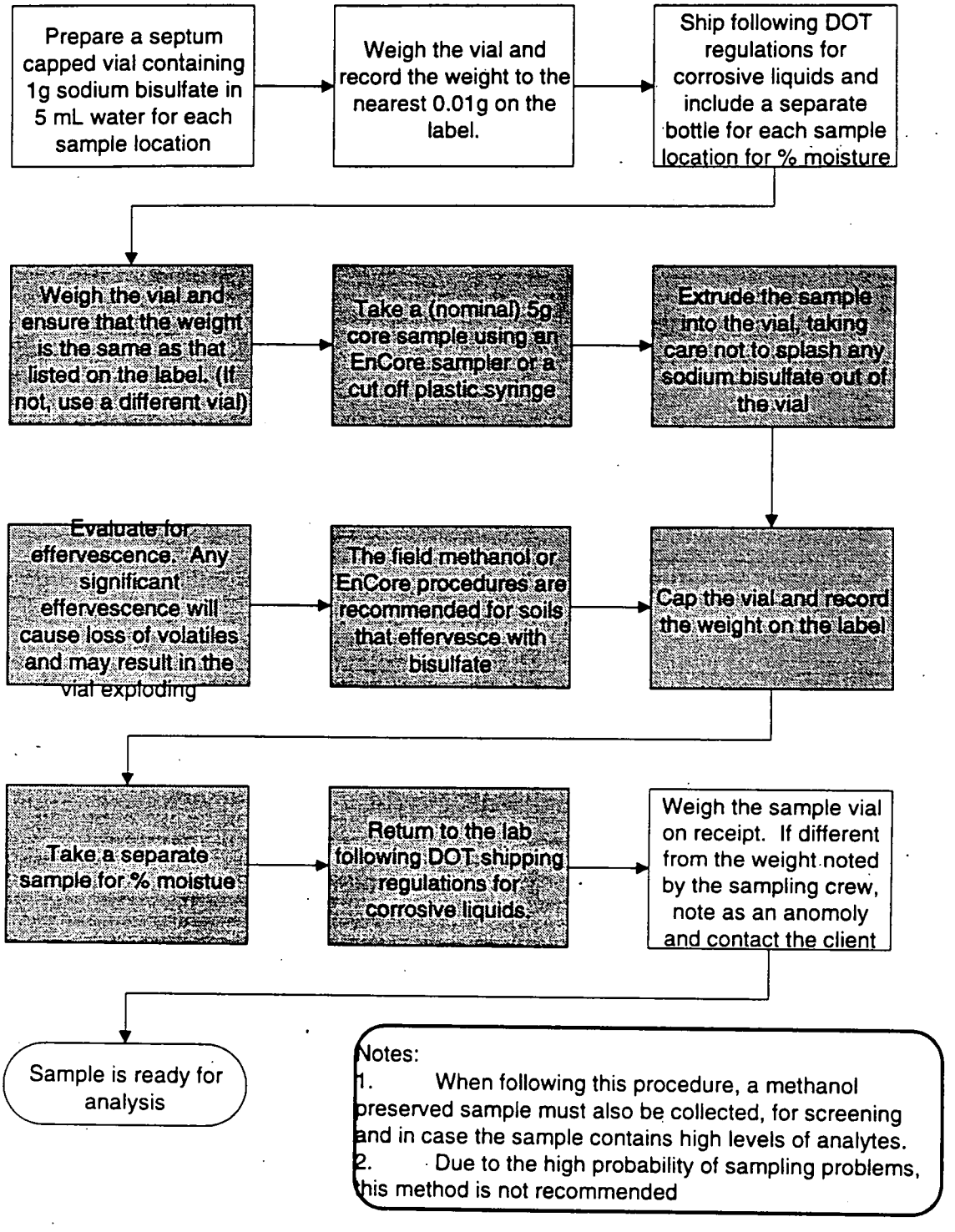


Field methanol extraction procedure (field steps in gray)



9.

Field bisulfate preservation procedure (field steps in gray)



Notes:
1. When following this procedure, a methanol preserved sample must also be collected, for screening and in case the sample contains high levels of analytes.
2. Due to the high probability of sampling problems, this method is not recommended

9. QUALITY CONTROL

9.1. Refer to the method 8000B section of this SOP, section 9, for general quality control procedures, including batch definition, requirements for method blanks, LCS, matrix spikes, surrogates, and control limits.

10. CALIBRATION AND STANDARDIZATION

10.1. Refer to the method 8000B section of this SOP, section 10, for general calibration procedures.

10.2. Gas Chromatograph Operating Conditions

Various column configurations are possible. If dual column confirmation is necessary, the sample may be split using a Y splitter at the injector end to direct the sample to two columns and two detectors. For simultaneous determination of aromatic and halogenated volatiles, a single column is used and the PID and ELCD detectors are connected in series.

10.2.1. Refer to Table A-2, A-3 and A-4 for GC operating conditions.

10.3. Initial Calibration

10.3.1. Refer to Section 10 of the 8000B section of this SOP for details of initial calibration criteria.

10.3.2. Low level soil samples must be purged at 40°C, therefore the calibration curve must also be purged at 40°C. In addition, the low level soil calibration solutions should contain approximately the same amount of sodium bisulfate as the samples.

10.3.3. The low level calibration must be at the reporting limit or below. The remaining standards encompass the working range of the detector.

10.3.4. Calibrate the instrument using the same volume that will be used during sample analysis.

10.4. Calibration Verification

10.4.1. A mid level calibration standard is used for the calibration verification. The gases have a 20%D criteria rather than the 15% used for other analytes.

10.4.2. A calibration verification run is performed after every 10 samples for this method.

10.4.3. Bracketing of samples with calibration verification runs is only necessary for external standard analysis.

11. PROCEDURE

11.1. Refer to the method 8000B section of this SOP for general procedural requirements.

11.2. Analytical Sequence

The analytical sequence starts with an initial calibration of at least five points, or a 12 hour calibration that meets % difference criteria from an existing initial calibration.

11.3. Confirmation

The PID and ELCD detectors are sufficiently selective that second column confirmation is not always necessary. Requirements for second column confirmation should be decided in consultation with the client. If the PID and ELCD are used in series confirmatory information for many analytes can be gained by comparing the relative response from the two detectors.

11.4. Aqueous Sample Analysis (Purge and Trap units using sparge vessels)

11.4.1. Depending on the sensitivity of the instrument and capabilities of the purge and trap device, 5, 10, 20, or 25 mL sample volumes may be analyzed. A 5 mL sample volume is recommended.

11.4.2. Rinse a 5 mL (or 25 mL for larger sample volumes) gas-tight syringe with organic free water. Fill the syringe with the sample to be analyzed, and compress to volume.

11.4.3. Check and document the pH of the sample remaining in the VOA vial after loading the syringe.

11.4.4. This procedure invalidates the contents of the VOA vial for further analysis, unless an aliquot is transferred to a smaller VOA vial with no headspace (e.g., 20 mL) at the same time the analysis aliquot is removed.

11.4.5. Spike with the appropriate volume of surrogate/internal standard solution and spike solution (if required) through the barrel of the syringe. The method blank is spiked with surrogates only, the LCS and matrix spikes with the surrogate and matrix spiking solutions. Refer to Tables A-5 and A-6 for volumes and concentrations of spiking solutions.

11.4.6. Load onto the purge and trap device and start the run.

11.4.7. If the initial analysis of a sample or a dilution of the sample has a concentration of analytes that exceeds the initial calibration range, the sample must be reanalyzed at a higher dilution. When a sample has a high response for a compound, analysis should be followed by an organic free water blank. It is recognized that during automated unattended analysis, this may not occur. If any potential carryover hits are present in samples following highly contaminated samples, the sample must be reanalyzed to determine if any of these hits are a result of carryover or are actually present in the sample.

11.4.8. Dilutions may be made in gas tight syringes unless the volume of sample used is less than 5 µL, in which case dilution in volumetric flasks will be necessary.

11.4.8.1. Spike with the same volume of surrogate/internal standard solution as used for undiluted samples prior to loading onto the purge and trap device.

11.4.8.2. For Matrix spike / matrix spike duplicates where the sample requires dilution, the sample is spiked <u>after</u> the dilution is performed.

11.5. Aqueous and Soil Sample Analysis (Purge and Trap units that sample directly from the VOA vial)

11.5.1. Units which sample from the VOA vial should be equipped with a module which automatically adds surrogate and internal standard solution to the sample prior to purging the sample.

11.5.2. If the autosampler uses automatic IS/SS injection, no further preparation of the VOA vial is needed. Otherwise the internal and surrogate standards must be added to the vial. *Note:* Aqueous samples with high amounts of sediment present in the vial may not be suitable for analysis on this instrumentation, or they may need to analyzed as soils.

11.5.3. Sample remaining in the vial after sampling with one of these mechanisms is no longer valid for further analysis. A fresh VOA vial must be used for further sample analysis.

11.5.4. Check the pH of the sample remaining in the VOA vial after analysis is completed.

11.6. Low-Level Solids Analysis using discrete autosamplers

Note: This technique may seriously underestimate analyte concentration and must not be used except at specific client request for the purpose of comparability with previous data. It is no longer part of SW-846.

This method is based on purging a heated sediment/soil sample mixed with reagent water containing the surrogate and, if applicable, internal and matrix spiking standards. Analyze all reagent blanks and standards under the same conditions as the samples (e.g., heated). The calibration curve is also heated during analysis. Purge temperature is 40°C.

11.6.1. Do not discard any supernatant liquids. Mix the contents of the container with a narrow metal spatula.

11.6.2. Weigh out 5 g (or other appropriate aliquot) of sample into a disposable culture tube or other purge vessel. Record the weight to the nearest 0.1 g. If method sensitivity is demonstrated, a smaller aliquot may be used. Do not use aliquots less than 1.0 g. If the sample is contaminated with analytes such that a purge amount less than 1.0 g is appropriate, use the medium level method described in section 11.7.

11.6.3. Connect the purge vessel to the purge and trap device.

11.6.4. Rinse a 5 mL gas-tight syringe with organic free water, and fill. Compress to 5 mL. Add surrogate/internal standard (and matrix spike solutions if required.) (See Tables A-5, A-6, A-7 and A-8.) Add directly to the sample from 11.6.2.

11.6.5. The above steps should be performed rapidly and without interruption to avoid loss of volatile organics.

11.6.6. Add the heater jacket or other heating device and start the purge and trap unit.

11.6.7. Soil samples that have low IS recovery when analyzed (<50%) should be reanalyzed once to confirm matrix effect. If external standard calibration is used, samples with surrogate recovery below the control limit should be reanalyzed once to confirm matrix effect.

11.7. Methanol Extract Soils

11.7.1. Rinse a gas-tight syringe with organic free water. Fill the syringe with the same volume of organic free water as used in the calibrations. Add no more than 2% (v/v) (100 µL for a 5 mL purge) methanolic extract (from Section 8.5 or 8.6) to the syringe. Add internal standard (if used). Load the sample onto the purge and trap device and analyze as for aqueous samples. If less than 5µL of methanolic extract is to be added to the water, dilute the methanolic extract such that a volume greater than 5µL will be added to the water in the syringe.

12. DATA ANALYSIS AND CALCULATIONS

Refer to section 12 of the 8000B section of this SOP.

13. METHOD PERFORMANCE

13.1. Performance limits for the four replicate initial demonstration of capability required under Section 13.1 of the 8000B section of this SOP are 70-130%, with %RSD ≤ 25% The spiking level should be 20 µg/L. These limits are for guidance, and certain "difficult" analytes may fall outside this range. Any outliers must be discussed with Quanterra's Director of Quality Assurance and/or Director of Technology before proceeding with sample analysis.

14. POLLUTION PREVENTION

This method does not contain any specific modifications that serve to minimize or prevent pollution.

15. WASTE MANAGEMENT

15.1. Waste generated in this procedure will be segregated and disposed according to the facility hazardous waste procedures. The Environmental Health and Safety Director should be contacted if additional information is required.

16. REFERENCES

16.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition, Final Update III, December 1996, Sections 5000, 5030B, 5035 and 8021B

17. MISCELLANEOUS

17.1. Modifications from Reference Method

17.2. Modifications from previous revision

17.2.1. Substantial changes to this method were made to reflect changes in Update III of SW-846.

17.3. Facility Specific SOPs

Each facility shall attach a list of facility specific SOPs or approved attachments (if applicable) which are required to implement this SOP or which are used in conjunction with this SOP. If no facility specific SOPs or amendments are to be attached, a statement must be attached specifying that there are none.

17.4.

Tables

Table A-1

Standard Analyte List

Test	Compound	CAS number	Reporting Limit, µg/L or µg/kg		
			Aqueous	Low Soil	Medium Soil
Halogenated volatiles by 8021B	Bromodichloromethane	75-27-4	1.0	1.0	50
	Bromoform	75-25-2	1.0	1.0	50
	Bromomethane	74-83-9	1.0	1.0	50
	Carbon Tetrachloride	56-23-5	1.0	1.0	50
	Chlorobenzene	108-90-7	1.0	1.0	50
	Chloroethane	70-00-3	1.0	1.0	50
	2-Chloroethyl vinyl ether	110-75-8	5.0	5.0	250
	Chloroform	67-66-3	1.0	1.0	50
	Chloromethane	74-87-3	1.0	1.0	50
	Dibromochloromethane	124-48-1	1.0	1.0	50
	1,2-Dichlorobenzene	95-50-1	1.0	1.0	50
	1,3-Dichlorobenzene	541-73-1	1.0	1.0	50
	1,4-Dichlorobenzene	106-46-7	1.0	1.0	50
	Dichlorodifluoromethane	75-71-8	1.0	1.0	50
	1,1-Dichloroethane	75-34-3	1.0	1.0	50
	1,2-Dichloroethane	107-06-2	1.0	1.0	50
	1,1-Dichloroethene	75-45-4	1.0	1.0	50
	cis-1,2 Dichloroethene	156-59-4	1.0	1.0	50
	trans-1,2-Dichloroethene	156-60-5	1.0	1.0	50
	Dichloromethane(DCM)	75-09-2	5.0	5.0	250
	1,2-Dichloropropane	78-87-5	1.0	1.0	50
	cis-1,3-Dichloropropene	10061-01-5	1.0	1.0	50
	trans-1,3-Dichloropropene	10061-02-6	1.0	1.0	50
	1,1,2,2-Tetrachloroethane	79-34-5	1.0	1.0	50
	Tetrachloroethene	127-18-4	1.0	1.0	50
	1,1,1-Trichloroethane	71-55-6	1.0	1.0	50
	1,1,2-Trichloroethane	79-00-5	1.0	1.0	50
	Trichloroethene	79-01-6	1.0	1.0	50
	Trichlorofluoromethane	75-69-4	1.0	1.0	50
	Vinyl Chloride	75-01-4	1.0	1.0	50
Additional halogenated volatiles	Benzyl Chloride	100-44-7	5.0	5.0	250
	Bromobenzene	108-86-1	1.0	1.0	50
	Dibromomethane	74-95-3	1.0	1.0	50
	1,1,1,2-Tetrachloroethane	630-20-6	1.0	1.0	50
	1,2,3-Trichloropropane	96-18-4	1.0	1.0	50
BTEX by 8021B	Benzene	71-43-2	1.0	1.0	50
	Ethyl Benzene	100-41-4	1.0	1.0	50
	Toluene	108-88-3	1.0	1.0	50
	Xylenes (total)	1330-20-7	1.0	1.0	50
Aromatic volatiles by 8021B	Benzene	71-43-2	1.0	1.0	50
	Chlorobenzene	108-90-7	1.0	1.0	50
	1,2-Dichlorobenzene	75-34-3	1.0	1.0	50
	1,3-Dichlorobenzene	107-06-2	1.0	1.0	50

Table A-1

Standard Analyte List

Test	Compound	CAS number	Reporting Limit, µg/L or µg/kg		
			Aqueous	Low Soil	Medium Soil
	1,4-Dichlorobenzene	75-45-4	1.0	1.0	50
	Ethyl Benzene	100-41-4	1.0	1.0	50
	Toluene	108-88-3	1.0	1.0	50
	Xylenes (total)	1330-20-7	1.0	1.0	50
Additional aromatic and unsaturated volatiles	1,2,4 Trimethylbenzene	95-63-6	1.0	1.0	50
	1,3,5 Trimethylbenzene	108-67-8	1.0	1.0	50
	Acetone	67-64-1	10	10	500
	MEK (2-butanone)	78-93-3	5.0	5.0	250
	MIBK (4-methyl-2-pentanone)	108-10-1	5.0	5.0	250
	Naphthalene	91-20-3	2.0	2.0	250
	Styrene	100-42-5	1.0	1.0	50
	Methyl tert-butyl ether (MTBE)	1634-04-4	1.0	1.0	50
Combined halogenated and aromatic volatiles by 8021B	Benzene	71-43-2	1.0	1.0	50
	Bromobenzene	108-86-1	1.0	1.0	50
	Bromochloromethane	74-97-5	1.0	1.0	50
	Bromodichloromethane	75-27-4	1.0	1.0	50
	Bromoform	75-25-2	1.0	1.0	50
	Bromomethane	74-83-9	1.0	1.0	50
	n-butylbenzene	104-51-8	1.0	1.0	50
	sec-Butylbenzene	135-98-8	1.0	1.0	50
	tert-Butylbenzene	98-06-6	1.0	1.0	50
	Carbon Tetrachloride	56-23-5	1.0	1.0	50
	Chlorobenzene	108-90-7	1.0	1.0	50
	Chlorodibromomethane	124-48-1	1.0	1.0	50
	Chloroethane	75-00-3	1.0	1.0	50
	Chloroform	67-66-3	1.0	1.0	50
	Chloromethane	74-87-3	1.0	1.0	50
	2-Chlorotoluene	95-49-8	1.0	1.0	50
	4-Chlorotoluene	106-43-4	1.0	1.0	50
	1,2-Dibromo-3-Chloropropane(DBCP)	96-12-8	1.0	1.0	50
	1,2-Dibromoethane(EDB)	106-93-4	1.0	1.0	50
	Dibromomethane	74-95-3	1.0	1.0	50
	1,2-Dichlorobenzene	95-50-1	1.0	1.0	50
	1,3-Dichlorobenzene	541-73-1	1.0	1.0	50
	1,4-Dichlorobenzene	106-46-7	1.0	1.0	50
	Dichlorodifluoromethane	75-71-8	1.0	1.0	50
	1,1-Dichloroethane	75-34-3	1.0	1.0	50
	1,2-Dichloroethane	107-06-2	1.0	1.0	50
	1,1-Dichloroethene	75-35-4	1.0	1.0	50
	cis-1,2-Dichloroethene	156-59-4	1.0	1.0	50
	trans-1,2-Dichloroethene	156-60-5	1.0	1.0	50
	1,2-Dichloropropane	78-87-5	1.0	1.0	50
	1,3-Dichloropropane	142-28-9	1.0	1.0	50
	2,2-Dichloropropane	590-20-7	1.0	1.0	50
	1,1-Dichloropropene	563-58-6	1.0	1.0	50

Table A-1
Standard Analyte List

Test	Compound	CAS number	Reporting Limit, µg/L or µg/kg		
			Aqueous	Low Soil	Medium Soil
	cis-1,3-Dichloropropene	10061-01-5	1.0	1.0	50
	trans-1,3-Dichloropropene	10061-02-6	1.0	1.0	50
	Ethylbenzene	100-41-4	1.0	1.0	50
	Hexachlorobutadiene	87-68-3	1.0	1.0	50
	Isopropylbenzene	98-82-8	1.0	1.0	50
	p-Isopropyltoluene	99-87-6	1.0	1.0	50
	Methylene Chloride	75-09-2	5.0	5.0	250
	Naphthalene	91-20-3	2.0	2.0	250
	n-Propylbenzene	10306501	1.0	1.0	50
	Styrene	100-42-5	1.0	1.0	50
	1,1,1,2-Tetrachloroethane	630-20-6	1.0	1.0	50
	1,1,2,2-Tetrachloroethane	79-34-5	1.0	1.0	50
	Tetrachloroethene	127-18-4	1.0	1.0	50
	Toluene	108-88-3	1.0	1.0	50
	1,2,3-Trichlorobenzene	87-61-6	1.0	1.0	50
	1,2,4-Trichlorobenzene	120-82-1	1.0	1.0	50
	1,1,1-Trichloroethane	71-55-6	1.0	1.0	50
	1,1,2-Trichloroethane	79-00-5	1.0	1.0	50
	Trichloroethene	79-01-6	1.0	1.0	50
	Trichlorofluoromethane	75-69-4	1.0	1.0	50
	1,2,3-Trichloropropane	96-18-4	1.0	1.0	50
	1,2,4-Trimethylbenzene	95-63-6	1.0	1.0	50
	1,3,5-Trimethylbenzene	108-67-8	1.0	1.0	50
	Vinyl Chloride	75-01-4	1.0	1.0	50
	Xylenes (total)	1330-20-7	1.0	1.0	50

Table A-2
Recommended Conditions for Aromatic Volatiles

Parameter	Recommended Conditions
Temperature program	50°C, 1min, 10°C/min to 200°C,1min
Column 1	Rtx-502.2 or DB-502.2 60m x 0.53mm 3.0um
Column 2	Rtx-1 or DB-1 60m x 0.53mm 3.0 um
Carrier gas	Helium or hydrogen
Purge Flow / time	40 mL/min, 11 minutes
Desorb Temp / time	180°C, 2 minutes (220°C for Vocarb 3000)
Bake Time / temp	200°C, 12 minutes (230°C for Vocarb 3000)
Transfer line / valve temp	115°C

Table A-3
Recommended Conditions for Method Halogenated Volatiles

Parameter	Recommended Conditions
Temperature program	35°C, 12 min, then 4°C/min to 200°C, hold for 5 min
Column 1	DB-VRX or RTX-502.2 105m x 0.53 mm id df = 3.0um
Column 2	DB-1 or RTX-1 105m x 0.53 mm ID df = 3.0um
Column 3	Rtx - Volatiles 120m x 0.53mm ID df=2.0um
Carrier gas	Helium
Purge Flow / time	40 mL/min, 11 minutes
Desorb Temp / time	180°C, 2 minutes (220°C for Vocarb 3000)
Bake Time / temp	200°C, 12 minutes (230°C for Vocarb 3000)
Transfer line / valve temp	115°C

Table A-4
Recommended Conditions for Method Combined Aromatic and Halogenated Volatiles

Parameter	Recommended Conditions
Temperature program	35°C, 12 min, then 4°C/min to 200°C, hold for 5 min
Column 1	DB-VRX or RTX-502.2 105m x 0.53 mm id df = 3.0um
Column 2	DB-1 or RTX-1 105m x 0.53 mm ID df = 3.0um
Column 3	Rtx - Volatiles 120m x 0.53mm ID df=2.0um
Carrier gas	Helium
Purge Flow / time	40 mL/min, 11 minutes
Desorb Temp / time	180°C, 2 minutes (220°C for Vocarb 3000)
Bake Time / temp	200°C, 12 minutes (230°C for Vocarb 3000)
Transfer line / valve temp	115°C

Table A-5
Surrogate and Internal Standard Concentrations

Standard	Components	Working Solution µg/mL	Spike amount µL (for 5 mL purge)	Final concentration µg/L (µg/kg)
Aromatic volatiles IS/SS	4-Chlorotoluene (SS) 1-Chloro-4-fluorobenzene (IS)	20 40	5	20 40
Halogenated volatiles IS/SS	4-chlorotoluene (SS) 1-Chloro-4-fluorobenzene (IS)	20 40	5	20 40
Combined Aromatic and halogenated volatiles IS/SS	Fluorobenzene (SS)	20	5	20
	1,4-Dichlorobutane (SS)	20	5	20
	1-Chloro-4-fluorobenzene (IS)	40	10	40

It may be necessary to select different surrogates in order to minimize sample interferences. 1-chloro-4-fluorobenzene and 4-chlorotoluene are fairly well resolved from analytes listed in this SOP. However 4-chlorotoluene may sometimes be requested as a target analyte. Other surrogates that may be considered, and issues associated with their use are:

Bromochloromethane: Elutes very close to chloroform and cis-1,2-dichloroethene on the 502.2 column. May be a target analyte.

1,2-Bromochloroethane:

1-Chloro-2-fluorobenzene: Elutes close to ethylbenzene on DB-1 or Rtx-1 and close to m,p-xylene on 502.2

a,a,a-Trifluorotoluene: Good for aromatic volatiles, coelutes or very close to trichloroethene

Bromofluorobenzene: Close to 1,1,2,2-trichloroethane and 1,2,3-trichloropropane on the 502.2 column. Good on DB-1 or Rtx-1.

2-Bromo-1-chloropropane: May coelute with 1,1,2-trichloroethane

Table A-6
Concentrations for LCS and MS/MSD compounds

Standard	Components	Working Solution µg/mL	Spike amount µL (5 mL purge)	Final concentration µg/L (µg/kg)
Aromatic	Benzene	20	5	20
	Toluene	20		20
	Chlorobenzene	20		20
Halogenated	Chlorobenzene	20	5	20
	1,1-Dichloroethene	20		20
	Trichloroethene	20		20
Combination aromatic / halogenated	Benzene	20	5	20
	Toluene	20		20
	Chlorobenzene	20		20
	1,1-Dichloroethene	20		20
	Trichloroethene	20		20

1. SCOPE AND APPLICATION

This SOP Appendix describes procedures to be used when SW-846 Method 8081A is applied to the analysis of organochlorine pesticides by GC/ECD. This Appendix may also to be applied when discontinued SW-846 Method 8080A is requested, and is applicable to extracts derived from any matrix which are prepared according to the appropriate Quanterra sample extraction SOPs. (CORP-OP-0001)

Table B-1 lists compounds which are routinely determined by this method and gives the Reporting Limits (RL) for each matrix. RLs given are based on the low level standard and the sample preparation concentration factors. Matrix interferences may result in higher RLs than those listed.

At client request, this method may also be used for the analysis of PCBs (Arochlors) in combination with pesticides, although these are normally analyzed following method 8082, as described in Appendix C of this SOP. In any event, if samples for PCB analysis do not need the acid clean up procedure, then the same injection may be used for method 8081B and 8082, assuming all calibration and QC requirements for both methods are met. Extracts that have been acid cleaned may not be analyzed for pesticides, since several of the pesticides will be degraded.

2. SUMMARY OF METHOD

This method presents conditions for the analysis of prepared extracts of organochlorine pesticides. The pesticides are injected onto the column and separated and detected by electron capture detection. Quantitation may be by internal or external standard methods.

3. DEFINITIONS

Refer to the QAMP for definitions of terms used in this document.

4. INTERFERENCES

4.1. Refer to the method 8000B section of this SOP for information regarding chromatographic interferences.

4.2. Interferences in the GC analysis arise from many compounds amenable to gas chromatography that give a measurable response on the electron capture detector. Phthalate esters, which are common plasticizers, can pose a major problem in the determinations. Interferences from phthalates are minimized by avoiding contact with any plastic materials.

4.3. Sulfur will interfere and can be removed using procedures described in SOP CORP-OP-0001.

4.4. Interferences co-extracted from samples will vary considerably from source to source. The presence of interferences may raise quantitation limits for individual samples. Specific cleanups may be performed on the sample extracts, including florisil cleanup (Method 3620), Gel Permeation Chromatography (Method 3640), and Sulfur cleanup (Method 3660). These cleanup procedures are included in SOP # CORP-OP-0001. Use or hexane / acetone as the extraction solvent (rather than hexane / methylene chloride) will reduce the amount of interferences extracted.

5. SAFETY

5.1. Refer to section 5 of the Method 8000B SOP for general safety requirements.

5.2. Aroclors have been classified as a potential carcinogen under OSHA. Concentrated solutions of Aroclors must be handled with extreme care to avoid excess exposure. Contaminated gloves and clothing must be removed immediately. Contaminated skin surfaces must be washed thoroughly.

6. EQUIPMENT AND SUPPLIES

6.1. Refer to Section 6 of the 8000B section of this SOP. A ^{63}Ni electron capture detector is required.

6.2. Refer to Table B-2 for analytical columns.

6.3. Microsyringes, various sizes, for standards preparation, sample injection, and extract dilution.

7. REAGENTS AND STANDARDS

7.1. Refer to the method 8000B section of this SOP for general requirements for reagents and supplies.

7.2. Refer to Table B-3 for details of calibration standards.

7.3. Surrogate Standards

Tetrachloro-m-xylene and decachlorobiphenyl are the surrogate standards. Refer to tables B-5 and B-6 for details of surrogate standards.

7.4. Column Degradation Evaluation Mix

A mid-level standard containing 4,4'-DDT and Endrin and not containing any of their breakdown products must be prepared for evaluation of degradation of these compounds by the GC column and injection port. This mix must be replaced after one year, or whenever corrective action to columns fails to eliminate the breakdown of the compounds, whichever is shorter. This solution also contains the surrogates. Refer to Table B-4 for details of the column degradation evaluation mix.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

Refer to Section 8 of the 8000B section of this SOP.

9. QUALITY CONTROL

Refer to Section 9 of the 8000B section of this SOP.

10. CALIBRATION AND STANDARDIZATION

10.1. Refer to Section 10 of the 8000B section of this SOP for general calibration requirements.

10.2. Refer to Table B-2 for details of GC operating conditions. The conditions listed should result in resolution of all analytes listed in Table B-1 on both columns. Closely eluting pairs are DDE and Dieldrin on the Rtx-5 or DB-5 column and Endosulfan II and DDD on the 1701 column.

10.3. Column Degradation Evaluation

Before any calibration runs, either initial or 12 hour, The column evaluation mix must be injected before each initial or daily calibration. The degradation of DDT and endrin must be calculated (see equations 9 and 10) and each shown to be less than 15% before calibration can proceed. This is only necessary if the target compound list includes DDT, Endrin, or any of their degradation products.

If the breakdown of DDT and/or endrin exceeds the limits given above, corrective action must be taken. This action may include:

- Replacement of the injection port liner or the glass wool.
- Cutting off a portion of the injection end of a capillary column.
- Replacing the GC column.

10.4. Initial Calibration

Refer to Section 10 of the 8000B section of this SOP for details of calibration procedures.

10.4.1. Refer to Table B-8 for the initial calibration analytical sequence.

10.4.2. The response for each single-peak analyte will be calculated by the procedures described in the general method for GC analysis.

10.4.3. The surrogate calibration curve is calculated from the Individual AB mix. Surrogates in the other calibration standards are used only as retention time markers. If there are resolution problems, then the A and B mixes may be analyzed separately.

10.4.4. For multi-component pesticides:

Single point calibration is used for multicomponent pesticides (typically toxaphene and technical chlordane). Two options are possible; the same quantitation option must be used for standards and samples. Refer to section 12.3 for guidance on which option to use.

10.4.5. For multicomponent analytes, the mid level standard must be analyzed as part of the initial calibration. This single point calibration is used to quantitate multicomponent analytes.

10.4.6. The analyst may include a full 5 point calibration for any of the multicomponent analytes with the initial calibration.

10.5. 12 hour Calibration Verification

The 12 hour calibration verification sequence must be analyzed within 12 hours of the start of the initial calibration and at least once every 12 hours thereafter if samples are being analyzed. If more than 12 hours have elapsed since the injection of the last sample in the analytical sequence, a new analytical sequence must be started with a 12 hour calibration. A mid level calibration standard is used for the 12 hour calibration. Refer to the 8000B section of this SOP for acceptance criteria.

10.5.1. At a minimum, the 12 hour calibration includes analysis of the breakdown mix followed by mid level standards of any single and multicomponent analytes.

10.5.2. The retention time windows for any analytes included in the 12 hour calibration are updated.

10.6. Continuing Calibration

The AB calibration mix is analyzed as the continuing calibration standard. At a minimum, this is analyzed after every 20 samples, including matrix spikes, LCS, and method blanks. If 12 hours elapse analyze the 12 hour standard sequence instead. The continuing calibration standard need not include multicomponent analytes. If instrument drift is expected due to sample matrix or other factors, it may be advisable to analyze the continuing calibration standard more frequently.

10.6.1. A mid level calibration standard is used for the continuing calibration.

11. PROCEDURE

11.1. Refer to the method 8000B section of this SOP for general procedural requirements.

11.2. Extraction

The extraction procedure is described in SOP No. CORP-OP-0001.

11.3. Cleanup

Cleanup procedures are described in SOP No. CORP-OP-0001.

11.4. Suggested gas chromatographic conditions are given in Table B-2.

11.5. Allow extracts to warm to ambient temperature before injection.

11.6. The suggested analytical sequence is given in Table B-8.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Refer to the 8000B section of this SOP for identification and quantitation of single component analytes.

12.2. Identification of Multicomponent Analytes

Retention time windows are also used for identification of multi-component analytes, but the "fingerprint" produced by major peaks of those compounds in the standard is used in tandem with the retention times to identify the compounds. The ratios of the areas of the major peaks are also taken into consideration. Identification of these compounds may be made even if the retention times of the peaks in the sample fall outside of the retention time windows of the standard, if in the analyst's judgment the fingerprint (retention time and peak ratios) resembles the standard chromatogram.

12.3. Quantitation of Multicomponent Analytes

Use 3-10 major peaks or total area for quantitation as described in section 10.4.4, initial calibration of multicomponent analytes.

12.3.1. If there are no interfering peaks within the envelope of the multicomponent analyte, the total area of the standards and samples may be used for quantitation. Any surrogate or extraneous peaks within the envelope must be subtracted from the total area.

12.3.1.1. Multiple peak option

This option is particularly valuable if toxaphene is identified but interferences make quantitation based on total area difficult. Select 3-10 major peaks in the analyte pattern. Calculate the response using the total area or total height of these peaks. Alternatively, find the response of each of the 3-10 peaks per multi-peak pesticide, and use these responses independently, averaging the resultant concentrations found in samples for a final concentration result. When using this option, it is appropriate to remove peaks that appear to be coeluting with contaminant peaks from the quantitation. (i.e. peaks which are significantly larger than would be expected from the rest of the pattern.)

Chlordane may be quantitated either using the multiple peak option (12.3.1.1) total area option (12.3.1.2.) or by quantitation of the major components, α-chlordane, γ-chlordane and heptachlor.

12.3.1.2. Total area option

The total area of the standards and samples may be used for quantitation of multicomponent analytes. Any surrogate or extraneous peaks within the envelope must be subtracted from the total area. This option should not be used if there are significant interference peaks within the multicomponent pattern in the samples. The retention time window for total area measurement must contain at least 90% of the area of the analyte.

12.4. Second column confirmation multi-component analytes will only be performed when requested by the client, because the appearance of the multiple peaks in the sample usually serves as a confirmation of analyte presence.

12.5. Surrogate recovery results are calculated and reported for decachlorobiphenyl (DCB) unless it is determined that sample interference has adversely affected the quantitation of that surrogate. Tetrachloro-m-xylene (TCMX) recovery is reported in lieu of DCB in samples having the interference,

and also may be used in samples in which DCB recovery is low. Corrective action is only necessary if DCB and TCMX are both outside of acceptance limits.

12.6. Calculation of Column Degradation/% Breakdown (%B)

$$DDT\ \%B = \frac{A_{DDD} + A_{DDE}}{A_{DDD} + A_{DDE} + A_{DDT}} x\ 100$$

where:
A_{DDD}, A_{DDE}, and A_{DDT} = the response of the peaks for 4,4'-DDD, 4,4'-DDE, and 4,4'-DDT in the column degradation evaluation mix.

$$Endrin\ \%B = \frac{A_{EK} + A_{EA}}{A_{EK} + A_{EA} + A_{E}} x\ 100$$

where:
A_{EK}, A_{EA}, and A_{E} = the response of endrin ketone, endrin aldehyde, and endrin in the column degradation evaluation mix.

13. METHOD PERFORMANCE

13.1. Performance limits for the four replicate initial demonstration of capability required under Section 13.1 of the main body of this SOP are presented in Table B-7. The spiking level should be equivalent to a mid level calibration.

14. POLLUTION PREVENTION

Refer to section 14 of the 8000B section of this SOP.

15. WASTE MANAGEMENT

15.1. Waste generated in this procedure will be segregated and disposed according to the facility hazardous waste procedures. The Environmental Health and Safety Director should be contacted if additional information is required.

16. REFERENCES

SW846, Update III, December 1996, Method 8081A

17. MISCELLANEOUS

17.1. Modifications from Reference Method

None

17.2. Modifications from Previous Revisions

17.3. Substantial revision were made to reflect the changes to method 8081 in Update III to SW-846.

17.4. Tables

Table B-1
Standard Analyte list and Reporting Limits

Compound	Reporting Limit, µg/L or µg/kg		
	water	soil	waste
Aldrin	0.05	1.7	50
α-BHC	0.05	1.7	50
β-BHC	0.05	1.7	50
δ-BHC	0.05	1.7	50
γ-BHC (Lindane)	0.05	1.7	50
α-Chlordane	0.05	1.7	50
γ-Chlordane	0.05	1.7	50
Chlordane (technical)	0.5	17	500
4,4'-DDD	0.05	1.7	50
4,4'-DDE	0.05	1.7	50
4,4'-DDT	0.05	1.7	50
Dieldrin	0.05	1.7	50
Endosulfan I	0.05	1.7	50
Endosulfan II	0.05	1.7	50
Endosulfan Sulfate	0.05	1.7	50
Endrin	0.05	1.7	50
Endrin Aldehyde	0.05	1.7	50
Heptachlor	0.05	1.7	50
Heptachlor Epoxide	0.05	1.7	50
Methoxychlor	0.1	3.3	100
Toxaphene	2.0	67	2000
APPENDIX IX ADD ONs			
Diallate	1.0	33	1000
Isodrin	0.1	3.3	100
Chlorobenzillate	0.1	3.3	100
Kepone[1]	1.0	33	1000

[1] Kepone is sometimes requested for analysis by method 8081A. However kepone may produce peaks with broad tails that elute later than the standard by up to a minute (presumably due to hemi-acetal formation). As a result kepone analysis by 8081A is unreliable and not recommended. Analysis by method 8270C is a possible alternative. Note: alpha chlordane, gamma chlordane, and endrin ketone are not required for some projects.

The following concentration factors are assumed in calculating the Reporting Limits:

	Extraction Vol.	Final Vol.
Ground water	1000 mL	10 mL
Low-level Soil	30 g	10 mL
High-level soil / waste	1 g	10 mL

Table B-2	
Parameter	**Recommended Conditions**
Injection port temp	220°C
Detector temp	325°C
Temperature program	120°C for 1 min, 8.5°C/min to 285°C, , 6 min hold
Column 1	Rtx-CLPesticides 30m x 0.32mm id, 0.5µm
Column 2	Rtx-35 30m x 0.32 mm id, 0.5µm
Column 3	DB-608, 30m X 0.32 mm, 0.25µm
Injection	2µL
Carrier gas	Helium or Hydrogen
Make up gas	Nitrogen
Y splitter	Restek or J&W or Supelco glass tee

Table B-3						
Calibration Levels ng/mL						
	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6[2]
Individual Mix AB[1]						
Aldrin	5	10	25	50	100	200
g-BHC (Lindane)	5	10	25	50	100	200
Heptachlor	5	10	25	50	100	200
Methoxychlor	10	20	50	100	200	400
Dieldrin	5	10	25	50	100	200
Endosulfan I	5	10	25	50	100	200
Endosulfan II	5	10	25	50	100	200
4,4'-DDT	5	10	25	50	100	200
Endrin Aldehyde	5	10	25	50	100	200
Endrin Ketone	5	10	25	50	100	200
β-BHC	5	10	25	50	100	200
δ-BHC	5	10	25	50	100	200
α-BHC	5	10	25	50	100	200
4,4'-DDD	5	10	25	50	100	200
4,4'-DDE	5	10	25	50	100	200
Endosulfan Sulfate	5	10	25	50	100	200
Endrin	5	10	25	50	100	200
α-Chlordane [3]	5	10	25	50	100	200
γ-Chlordane [3]	5	10	25	50	100	200
Multicomponent Standards						
Chlordane (Technical)			250[4]			
Toxaphene			1000[5]			
Surrogates are included with all the calibration mixes at the following levels:						
Tetrachloro-m-xylene	5	10	25	50	100	200
Decachlorobiphenyl	5	10	25	50	100	200

[1] Standards may be split into an A and B mix if resolution of all compounds on both columns is not obtained.
[2] Level 6 is optional and should only be used if linearity can be maintained on the instrument to this level.
[3] Compounds may be used in lieu of running a daily technical Chlordane standard for samples that are non-detect for technical Chlordane.
[4] This standard may be used for quantitation of technical chlordane between 50 and 1000 ng/mL. If the chlordane is more concentrated, the extract must be diluted and reanalyzed.
[5] This standard may be used for quantitation of toxaphene between 200 and 4000 ng/mL. If the toxaphene is more concentrated, the extract must be diluted and reanalyzed.

Table B-4	
Column Degradation Evaluation Mix ng/mL	
Component	Concentration
4,4'-DDT	25
Endrin	25
Tetrachloro-m-xylene (Surrogate)	20
Decachlorobiphenyl (Surrogate)	20

Table B-5			
LCS/Matrix Spike and Surrogate Spike levels µg/L or µg/kg			
	Aqueous	Soil	Waste
gamma BHC (Lindane)	0.20	6.67	200
Aldrin	0.20	6.67	200
Heptachlor	0.20	6.67	200
Dieldrin	0.50	16.7	500
Endrin	0.50	16.7	500
4,4'DDT	0.50	16.7	500
Tetrachloro-m-xylene (Surrogate)	0.20	6.67	200
Decachlorobiphenyl (Surrogate)	0.20	6.67	200

Table B-6		
LCS/Matrix Spike and Surrogate Spike levels for TCLP µg/L or µg/kg		
	Aqueous	Waste
Heptachlor	5	500
Heptachlor epoxide	5	500
Lindane	5	500
Endrin	5	500
Methoxychlor	10	1000

Table B-7
Suggested Analytical Sequence

Initial Calibration

Solvent blank (optional)	
Breakdown Mix	
Individual mix AB	All levels
Technical Chlordane	Level 3[1]
Toxaphene	Level 3[1]
Solvent blank	
Up to 20 samples unless 12 hours comes first)	
Solvent blank (optional)	
Individual mix AB	Mid level (Continuing calibration)
Samples	
After 12 hours:	
Breakdown mix	
Individual mix AB	
Any other single component analytes	
Any multicomponent analytes	

[1] A five point curve for any of the multicomponent analytes may be included
If Arochlors are included, a 5 point calibration for Arochlor 1016/1260 should be included with the initial calibration and a single point for the other Arochlors. The mid point 1016/1260 mix is included with the daily calibration (every 12 hours).

12 hour Calibration

At least every 12 hours, counting from the start of the initial calibration, or from the start of the last daily calibration, the retention time windows must be updated using the Individual mix AB, and the breakdown mix must be run before the continuing calibration.

Table B-8		
Performance limits, four replicate initial demonstration of capability		
Compound	Initial demonstration, mean recovery limits	Initial demonstration, RSD limits
Aldrin	46-112	21
alpha-BHC	51-122	24
beta-BHC	61-120	32
delta-BHC	49.5-118.5	36
gamma-BHC	57-116	23
Chlordane	44.8-108.6	20
4,4'-DDD	52-126	28
4,4'-DDE	46-120	27.5
4,4'-DDT	54-137	36
Dieldrin	42.5-124.5	38
Endosulfan I	43-141	24.5
Endosulfan II	78-171	61
Endosulfan Sulfate	62-132	27
Endrin	49-126	37
Heptachlor	57-100	20
Heptachlor Epoxide	43.5-131.5	25.4
Toxaphene	44.4-111.2	20

1. SCOPE AND APPLICATION

1.1. This SOP Appendix describes procedures to be used when SW-846 Method 8000B is applied to the analysis of polychlorinated biphenyls (PCB) by GC/ECD. This Appendix is to be applied when SW-846 Method 8082 is requested, and is applicable to extracts derived from any matrix which are prepared according to the appropriate Quanterra sample extraction SOPs. (CORP-OP-0001). The PCBs are determined and quantitated as Arochlor mixes.

Table C-1 lists compounds which are routinely determined by this method and gives the Reporting Limits (RL) for each matrix. RLs given are based on the low level standard and the sample preparation concentration factors. Matrix interferences may result in higher RLs than those listed.

Note: SW-846 method 8082 provides incomplete guidance for determination of individual PCB congeners. This SOP does not include directions for congener specific analysis.

2. SUMMARY OF METHOD

This method presents conditions for the analysis of prepared extracts of PCBs. The PCBs are injected onto the column and separated and detected by electron capture detection. Quantitation is by the external standard method.

3. DEFINITIONS

Refer to the QAMP for definitions of terms used in this document.

4. INTERFERENCES

4.1. Refer to the method 8000B section of this SOP for information regarding chromatographic interferences.

4.2. Interferences in the GC analysis arise from many compounds amenable to gas chromatography that give a measurable response on the electron capture detector. Phthalate esters, which are common plasticizers, can pose a major problem in the determinations. Interferences from phthalates are minimized by avoiding contact with any plastic materials.

4.3. Sulfur will interfere and can be removed using procedures described in SOP CORP-OP-0001.

4.4. Interferences co-extracted from samples will vary considerably from source to source. The presence of interferences may raise quantitation limits for individual samples. Specific cleanups may be performed on the sample extracts, including florisil cleanup (Method 3620), Gel Permeation Chromatography (Method 3640), and Sulfur cleanup (Method 3660). These cleanup procedures are included in SOP # CORP-OP-0001.

5. SAFETY

5.1. Refer to section 5 of the Method 8000B SOP for general safety requirements.

5.2. Aroclors have been classified as a potential carcinogen under OSHA. Concentrated solutions of Aroclors must be handled with extreme care to avoid excess exposure. Contaminated gloves and clothing must be removed immediately. Contaminated skin surfaces must be washed thoroughly.

6. EQUIPMENT AND SUPPLIES

6.1. Refer to Section 6 of the 8000B section of this SOP. A ^{63}Ni electron capture detector is required.

6.2. Refer to Table C-2 for analytical columns.

6.3. Microsyringes, various sizes, for standards preparation, sample injection, and extract dilution.

7. REAGENTS AND STANDARDS

7.1. Refer to the method 8000B section of this SOP for general requirements for reagents and supplies. All standards for this method must be replaced

7.2. Refer to Table C-3 for details of calibration standards.

7.3. Surrogate Standards

Tetrachloro-m-xylene and decachlorobiphenyl are the surrogate standards. Other surrogates may be used at client request. Refer to Table C-4 for details of surrogate standards.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

Refer to Section 8 of the 8000B section of this SOP.

9. QUALITY CONTROL

Refer to Section 9 of the 8000B section of this SOP.

10. CALIBRATION AND STANDARDIZATION

10.1. Refer to Section 10 of the 8000B section of this SOP for general calibration requirements.

10.2. Initial Calibration

10.2.1. Refer to Table C-5 for the initial calibration analytical sequence.

10.2.2. The response for each Arochlor will be calculated by the procedures described in the general method for GC analysis, with the following modifications.

10.2.3. A five point calibration of the Arochlor 1016/1260 mix is generated with at least mid level single points for the other Aroclor mixes. The average response factor is used to quantitate Arochlors 1260 and 1016, other Arochlors are quantitated from the mid level single point.

10.2.4. The analyst may include a full 5 point calibration for any of the Arochlors with the initial calibration.

10.2.5. The high and low standards for the initial 5 point calibration of 1016 / 1260 define the acceptable quantitation range for the other Arochlors. If any Arochlor is determined above this concentration the extract must be diluted and reanalyzed.

10.2.6. If the analyst knows that a specific Arochlor is of interest for a particular project, that Arochlor may be used for the five point calibration rather than the 1016 / 1260 mix.

10.2.7. The surrogate calibration curve is calculated from the Aroclor 1016/1260 mix. Surrogates in the other calibration standards are used only as retention time markers.

10.2.8. Two options are possible for quantitation of Aroclors. The same quantitation option must be used for standards and samples.

10.2.8.1. Multiple peak option

Select 3-10 major peaks in the analyte pattern. Calculate the response using the total area or total height of these peaks. Alternatively, find the response of each of the 3-10 peaks per Aroclor, and use these responses

independently, averaging the resultant concentrations found in samples for a final concentration result. When using this option, it is appropriate to remove peaks that appear to be coeluting with contaminant peaks from the quantitation. (i.e. peaks which are significantly larger than would be expected from the rest of the pattern.)

10.2.8.2. Total area option

The total area of the standards and samples may be used for quantitation of multicomponent analytes. Any surrogate or extraneous peaks within the envelope must be subtracted from the total area. This option should not be used if there are significant interference peaks within the multicomponent pattern in the samples. The retention time window for total area measurement must contain at least 90% of the area of the analyte.

10.3. 12 hour Calibration

The 12 hour calibration verification must be analyzed within 12 hours of the start of the initial calibration and at least once every 12 hours thereafter if samples are being analyzed. If there is a break in the analytical sequence of greater than 12 hours, then a new continuing calibration run must be analyzed before proceeding with the sequence. If more than 12 hours have elapsed since the injection of the last sample in the analytical sequence, a new analytical sequence must be started with a 12 hour calibration.

10.3.1. At a minimum, the 12 hour calibration includes analysis of the Arochlor 1260 / 1016 mix.

10.3.2. It is adequate to verify calibration with a mixture of Aroclors 1016 and 1260. If a specific Aroclor is expected, it should be included in the daily calibration check.

10.3.3. The retention time windows for any analytes included in the daily calibration are updated.

10.3.4. For this method samples must be bracketed with successful calibration verification runs.

10.4. Calibration verification

The Arochlor 1260/1016 calibration mix is analyzed as the calibration verification standard. This is analyzed after every 20 samples, including matrix spikes, LCS, and method blanks. (Depending on the type of samples, it may be advisable to analyze verifications more frequently in order to minimize reruns.).

10.4.1. A mid level standard is used for the calibration verification.

11. PROCEDURE

11.1. Refer to the method 8000B section of this SOP for general procedural requirements.

11.2. Extraction

The extraction procedure is described in SOP No. CORP-OP-0001.

11.3. Cleanup

Cleanup procedures are described in SOP No. CORP-OP-0001.

11.4. Suggested gas chromatographic conditions are given in Table C-2.

11.5. Allow extracts to warm to ambient temperature before injection.

11.6. The suggested analytical sequence is given in Table C-5.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Identification of Arochlors

Retention time windows are used for identification of Arochlors, but the "fingerprint" produced by major peaks of those analytes in the standard is used in tandem with the retention times for identification. The ratios of the areas of the major peaks are also taken into consideration. Identification may be made even if the retention times of the peaks in the sample fall outside of the retention time windows of the standard, if in the analyst's judgment the fingerprint (retention time and peak ratios) resembles the standard chromatogram.

A clearly identifiable Arochlor pattern serves as confirmation of single column GC analysis. However, if the pattern is not clear, or if no historical data for the site is available, then second column confirmation must be performed.

12.2. Quantitation of Arochlors

Use 3-10 major peaks or total area for quantitation

If the analyst believes that a combination of Aroclor 1254 and 1260, or a combination of 1242, 1248 and 1232 is present, then only the predominant Arochlor is quantitated and reported, but the suspicion of multiple Aroclors is discussed in the narrative. If well separated Aroclor patterns are present, then both Aroclors are quantitated and reported.

12.3. If there are no interfering peaks within the envelope of the Arochlor, the total area of the standards and samples may be used for quantitation. Any surrogate or extraneous peaks within the envelope must be subtracted from the total area.

12.4. Second column confirmation of Aroclors will only be performed when requested by the client, or if the pattern is not clear or there is no historical data leading to a suspicion that Arochlors may be present. The appearance of the multiple peaks in the sample usually serves as a confirmation of Aroclor presence.

12.5. Surrogate recovery results are calculated and reported for decachlorobiphenyl (DCB) unless it is determined that sample interference has adversely affected the quantitation of that surrogate. Tetrachloro-m-xylene (TCMX) recovery is reported in lieu of DCB in samples having the interference, and also may be used in samples in which DCB recovery is low. Corrective action is only necessary if DCB and TCMX are both outside of acceptance limits.

13. METHOD PERFORMANCE

13.1. Performance limits for the four replicate initial demonstration of capability required under Section 13.1 of the main body of this SOP are recovery of 70-130%. The spiking level should be equivalent to a mid level calibration.

13.2. Method detection limits (MDL) are determined for Arochlor 1016 and 1260. Arochlor 1016 represents an Arochlor consisting primarily of low chlorinated congeners while Arochlor 1260 represents an Arochlor consisting primarily of high chlorinated congeners. The same reporting limit is applied to all Arochlors, and must be supported by both MDLs.

14. POLLUTION PREVENTION

Refer to section 14 of the 8000B section of this SOP.

15. WASTE MANAGEMENT

15.1. Waste generated in this procedure will be segregated and disposed according to the facility hazardous waste procedures. The Environmental Health and Safety Director should be contacted if additional information is required.

16. REFERENCES

SW846, Update III, December 1996, Method 8082

17. MISCELLANEOUS

17.1. Modifications from Reference Method

17.1.1. Method 8082 includes limited direction for congener specific quantitation. This is outside the scope of this SOP.

17.2. Modifications from Previous Revisions

The 8082 procedure was added to this revision.

17.3.

Tables

Table C-1
Standard Analyte list and Reporting Limits

Compound	Reporting Limit, µg/L or µg/kg		
	water	soil	waste
Aroclor-1016	1.0	33	1000
Aroclor-1221	1.0	33	1000
Aroclor-1232	1.0	33	1000
Aroclor 1242	1.0	33	1000
Aroclor-1248	1.0	33	1000
Aroclor-1254	1.0	33	1000
Aroclor-1260	1.0	33	1000

The following concentration factors are assumed in calculating the Reporting Limits:

	Extraction Vol.	Final Vol.
Ground water	1000 mL	10 mL
Low-level Soil	30 g	10 mL
High-level soil / waste	1 g	10 mL

Table C-2

Parameter	Recommended Conditions
Injection port temp	220°C
Detector temp	325°C
Temperature program	70°C for 0.5min, 30°C/min to 190°C, 2.5°C/min to 225, 18°C/min to 280°C, 3 min hold
Column 1	DB-5 or Rtx-5 30m x 0.32mm id, 0.5µm
Column 2	DB-1701 or Rtx 1701 30m x 0.32 mm id, 0.25µm
Column 3	DB-608, 30m X 0.32 mm, 0.25µm
Injection	1-2µL
Carrier gas	Helium or Hydrogen
Make up gas	Nitrogen
Y splitter	Restek or J&W or Supelco glass tee

Table C-3						
Calibration Levels ng/mL						
	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6[1]
Aroclor 1016/1260	100	200	500	1000	2000	4000
Aroclor 1242[2]			500			
Aroclor 1221 +1254[2]			500			
Aroclor 1232[2]			500			
Aroclor 1248[2]			500			
Surrogates are included with all the calibration mixes at the following levels:						
Tetrachloro-m-xylene	5	10	25	50	100	200
Decachlorobiphenyl	5	10	25	50	100	200

[1] Level 6 is optional and should only be used if linearity can be maintained on the instrument to this level.
[2] Arochlors may be quantitated within the range 100 to 2000 ng/mL (4000ng/mL if the level 6 1016/1260 standard is included). If the Arochlor is more concentrated, it must be reanalyzed at a dilution.

Table C-4			
LCS/Matrix Spike and Surrogate Spike levels for Aroclor analysis with Acid Cleanup µg/L or µg/kg			
	Aqueous	Soil	Waste
Aroclor 1016/1260	10	333	10,000
Tetrachloro-m-xylene (Surrogate)	0.20	6.67	200
Decachlorobiphenyl (Surrogate)	0.20	6.67	200

Table C-5
Suggested Analytical Sequence

Initial Calibration

Injection #		
1	Solvent blank (optional)	
2	Aroclor 1016/1260	Level 1
3	Aroclor 1016/1260	Level 2
4	Aroclor 1016/1260	Level 3
5	Aroclor 1016/1260	Level 4
6	Aroclor 1016/1260	Level 5
7	Aroclor 1232	Level 3
8	Aroclor 1242	Level 3
9	Aroclor 1248	Level 3
10	Aroclor 1221/1254	Level 3
11	Solvent blank	
12-31	Sample 1-20 (or as many samples as can be analyzed in 12 hours	
	Solvent blank (optional)	
32	Aroclor 1016/1260	Level 3

etc

12 hour Calibration

At least every 12 hours, counting from the start of the initial calibration, or from the start of the last daily calibration, the retention time windows must be updated using the Arochlor 1260 / 1016 mix. Mid level standards of any other Arochlors expected to be present in the samples are also injected.

1. SCOPE AND APPLICATION

This method is applicable to the gas chromatographic determination of Chlorinated phenoxy acid herbicides in extracts prepared by SOP CORP-OP-0001. The herbicides listed in Table D1 are routinely analyzed. Other chlorinated acids may be analyzed by this method if the quality control criteria in section 9 and the initial demonstration of method performance in section 13 are met.

2. SUMMARY OF METHOD

This method presents conditions for the analysis of prepared extracts of phenoxy acid herbicides by gas chromatography. The herbicides, as their methyl esters, are injected onto the column, separated, and detected by electron capture detectors. Quantitation is by the external standard method.

3. DEFINITIONS

Refer to the QAMP for definitions of terms used in this document.

4. INTERFERENCES

4.1. Refer to the method 8000B section of this SOP for general information regarding chromatographic interferences.

4.2. Chlorinated acids and phenols cause the most direct interference with this method.

4.3. Sulfur may interfere and may be removed by the procedure described in SOP#CORP-OP-0001.

5. SAFETY

5.1. Refer to section 5 of the Method 8000B SOP for general safety requirements.

6. EQUIPMENT AND SUPPLIES

6.1. Refer to Section 6 of the 8000B section of this SOP. A Ni_{63} electron capture detector is required.

6.2. Refer to Table D2 for analytical columns.

6.3. Microsyringes, various sizes, for standards preparation, sample injection, and extract dilution.

7. REAGENTS AND STANDARDS

7.1. Refer to section 7 of the 8000B section of this SOP for general information on reagents and standards.

7.2. Refer to Table D-3 and D-4 for details of calibration and other standards.

8. SAMPLE PREPARATION, PRESERVATION AND STORAGE

Refer to section 8 of the 8000B section of this SOP.

9. QUALITY CONTROL

9.1. Refer to Section 9 of the 8000B section of this SOP for quality control requirements, including the initial demonstration of capability, definition of a batch, surrogate limits, method blanks, laboratory control samples (LCS), and matrix spikes (MS).

9.2. Refer to Table D-5 for minimum performance criteria for the initial demonstration of capability.

9.3. Refer to Table D-4 for the components and levels of the LCS and MS mixes.

10. CALIBRATION AND STANDARDIZATION

10.1. Refer to Section 10 of the 8000B section of this SOP for general calibration requirements.

10.2. External standard calibration is used for this method.

10.3. A mid level standard is used for the continuing calibration.

10.4. Refer to Table D-2, for details of GC operating conditions.

11. PROCEDURE

11.1. Refer to the method 8000B section of this SOP for procedural requirements.

11.2. Extraction

The extraction procedure is described in SOP #CORP-OP-0001.

11.3. Cleanup

The alkaline hydrolysis and subsequent extraction of the basic solution described in the extraction procedure provides an effective cleanup.

11.4. Analytical Sequence

The analytical sequence starts with an initial calibration of at least five points, or a daily calibration that meets % difference criteria from an existing initial calibration.

11.4.1. The daily calibration must be analyzed at least once every 24 hours when samples are being analyzed. If there is a break in the analytical sequence of greater than 12 hours, then a new continuing calibration run must be analyzed before proceeding with the sequence. If more than 24 hours have elapsed since the injection of the last sample in the analytical sequence, a new analytical sequence must be started with a daily calibration.

11.4.2. The daily calibration consists of mid level standards of all analytes of interest. Retention time windows must be updated with the daily calibration.

11.4.3. After every 12 hours a continuing calibration is analyzed. The continuing calibration consists of mid level standards of all analytes of interest. Retention time windows are updated with continuing calibrations.

11.5. Gas Chromatography

Chromatographic conditions are listed in Table D-2.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Refer to the 8000B section of this SOP for identification and quantitation of single component analytes.

12.2. The herbicides are analyzed as their methyl esters, but reported as the free acid. For this reason it is necessary to correct the results for the molecular weight of the ester versus the free acid. This is achieved through the concentrations of the calibration standards. For example the 20µg/L calibration standard for 2,4-D contains 21.3 µg/L of the methyl ester. No further correction is necessary.

13. METHOD PERFORMANCE

13.1. Multiple laboratory performance data has not been published by the EPA for this method. Table D-5 lists minimum performance standards required by Quanterra for the four replicate initial demonstration or capability (required by Section 13.2 of the 8000B part of this SOP) for this method. The spiking level should be equivalent to a mid level calibration.

14. POLLUTION PREVENTION

This method does not contain any specific modifications that serve to minimize or prevent pollution.

15. WASTE MANAGEMENT

Waste generated in this procedure will be segregated and disposed according to the facility hazardous waste procedures. The Environmental Health and Safety Director should be contacted if additional information is required.

16. REFERENCES

Method 8151A, SW-846, Update III, December 1996

17. MISCELLANEOUS

17.1. Modifications from Reference Method

Refer to the method 8000B section of this SOP for modifications from the reference method.

17.2. Modifications from Previous Revision

Modifications to the calibration directions were made in order to make the method consistent with method 8000B.

17.3. Tables

Table D-1				
Standard Analyte list				
Compound	CAS Number	Reporting Limit, µg/L or µg/kg		
		Aqueous	Soil	Waste
2,4-D	94-75-7	4	80	4000
2,4-DB	94-82-6	4	80	4000
2,4,5-TP (Silvex)	93-72-1	1	20	1000
2,4,5-T	93-76-5	1	20	1000
Dalapon	75-99-0	2	40	2000
Dicamba	1918-00-9	2	40	2000
Dichloroprop	120-36-5	4	80	4000
Dinoseb	88-85-7	0.6	12	600
MCPA	94-74-6	400	8000	400,000
MCPP	93-65-2	400	8000	400,000

The following concentration factors are assumed in calculating the Reporting Limits:

	Extraction Vol.	Final Vol.	Dilution Factor
Ground water	1000 mL	10 mL	20
Low-level Soil without GPC	50 g	10 mL	20
High-level soil / waste	1 g	10 mL	20

Specific reporting limits are highly matrix dependent. The reporting limits listed above are provided for guidance only and may not always be achievable. For special projects, the extracts may be analyzed without any dilution, resulting in reporting limits 20 times lower than those in Table D-1.

Table D-2	
Instrumental Conditions	
PARAMETER	Recommended conditions
Injection port temp	220°C
Detector temp	325°C
Temperature program	80,2/30/170,0/1/180,1
Column 1	DB-5MS or RTX 5 30x0.32, 0.5um
Column 2	DB-1701 or Rtx-1701
Injection	1-2µL
Carrier gas	Helium / Hydrogen
Make up gas	Nitrogen

Recommended conditions should result in resolution of all analytes listed in Table D-1.

Table D-3						
Calibration Standards, free acid equivalent (methyl ester)[1] ng/mL						
Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6[2]
2,4-D	20 (21.3)	40 (42.5)	80 (85.1)	160 (170)	320 (340)	640 (681)
2,4-DB	20 (21.1)	40 (42.2)	80 (84.5)	160 (169)	320 (338)	640 (676)
2,4,5-TP [Silvex]	5 (5.25)	10 (10.5)	20 (21.1)	40 (42.1)	80 (84)	160 (168)
2,4,5-T	5 (5.27)	10 (10.5)	20 (21.1)	40 (42.2)	80 (84.4)	160 (169)
Dalapon	10 (11.0)	20 (22.0)	40 (43.9)	80 (87.8)	160 (176)	320 (351)
Dicamba	10 (10.6)	20 (21.3)	40 (42.5)	80 (85.1)	160 (170)	320 (340)
Dichloroprop	20 (21.2)	40 (42.4)	80 (84.8)	160 (170)	320 (339)	640 (678)
Dinoseb	3 (3.17)	6 (6.35)	12 (12.7)	24 (25.4)	48 (50.8)	96 (102)
MCPA	2,000 (2,140)	4,000 (4,280)	8,000 (8,560)	16,000 (17,100)	32,000 (34000)	64,000 (68,000)
MCPP	2,000 (2,120)	4,000 (4,260)	8,000 (8,520)	16,000 (17,000)	32,000 (34,100)	64,000 (68,200)
2,4-DCAA [Surrogate]	20	40	80	160	320	640

[1]The calibration standard solutions contain the herbicides as their methyl esters at the levels in parentheses above. These levels are equivalent to the free acid levels also listed above. The herbicides are reported as the free acids.

[2] Level 6 is optional

The reporting limits listed in Table D-1 will be achieved with these calibration levels and a 20 fold dilution of the sample extract. Lower reporting limits can be achieved with lesser dilutions of the sample extract.

Table D-4			
LCS/Matrix Spike and Surrogate Spike levels µg/L or µg/kg[1]			
	Aqueous	Soil	Waste
2,4-D	16	800	16000
Silvex	4	200	4000
2,4,5-T	4	200	4000
2,4-DB	16	800	16000
Dalapon	8	400	8000
DCAA (surrogate)	16	800	16000

[1] LCS, MS and SS spikes are as the free acid.

Table D-5		
Performance limits, four replicate initial demonstration of capability		
Compound	Initial demonstration, mean recovery limits	Initial demonstration, RSD limits
2,4-D	50-150	25
2,4-DB	50-150	25
2,4,5-TP (Silvex)	50-150	25
2,4,5-T	50-150	25
Dalapon	50-150	25
Dicamba	50-150	25
Dichloroprop	50-150	25
Dinoseb	25-120	40
MCPA	50-150	25
MCPP	50-150	25

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OPERATION-SPECIFIC STANDARD OPERATING PROCEDURE

TITLE: AMMONIA AS N (LACHAT AUTOANALYZER)

(SUPERSEDES: LM-CAL-1105, REVISION 2.0, MARCH 6, 1990)

Prepared by: Hamid Foolad

Reviewed by: _____
Technical Specialist, Josefina P. Jones

Approved by: _____
Quality Assurance Manager, Gail Cho Celaschi

Approved by: _____
Environmental Health and Safety Coordinator, Cheryl Ann Stith

Approved by: _____
Laboratory Manager, Robert Dovi

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CU-561

1. SCOPE AND APPLICATION

1.1. This method is applicable to the analysis of Ammonia using a LACHAT QUIKCHEM AE autoanalyzer system by EPA Method 350.1.

1.2. CAS Number: 7664-41-7.

1.3. The detection limit is 0.1 mg/L as N.

1.4. This method is applicable to drinking, surface and saline water, domestic and industrial wastes. Non-aqueous samples may also be analyzed if first extracted with deionized water.

1.5. Instrument response is linear to at least 20 mg/L under the conditions stated in the method. However, the instrument is usually only calibrated over the range 0.1 to 5 mg/L in order to improve sensitivity and avoid a large number of calibration points.

1.6. Approximate analytical time is 2 minutes per sample.

2. SUMMARY OF METHOD

2.1. Samples are analyzed on the LACHAT QUIKCHEM AE system. The method is based on the Berthelot reaction: ammonia reacts with alkaline phenol and sodium hypochlorite to form indophenol blue. The blue color is intensified using sodium nitroprusside. The LACHAT measures absorbance at 630 nm. According to Beer's Law, the absorbance of the reaction product is directly proportional to ammonia concentration.

3. DEFINITIONS

3.1. Deionized water

3.1.1. Distilled water pumped through the Millipore system to ensure that it is ammonia-free water.

3.2. Definitions of terms used in this SOP may be found in the glossary of the Quality Assurance Management Plan (QAMP).

4. INTERFERENCES

4.1. Suspended matter may clog the system. Particulates may be removed by manual filtration.

4.2. High concentrations of calcium and magnesium may cause a precipitate to form. To prevent precipitation, use EDTA for river and industrial water and tartrate for sea water.

4.3. Turbidity may cause interferences in the samples. Turbidity is removed by manual filtration.

4.4. Color may cause interference in the samples. Color may be prevented by running the samples through the manifold as all the reagents are pumping through with deionized water. Non colored samples are run through the manifold as all the reagents are pumping through with *sodium hypochlorite*. The results from the colored samples are then subtracted from the non colored samples to obtain the corrected result.

4.4.1. NOTE: Highly colored samples must be diluted prior to analysis.

4.5. Ammonium hydroxide is a frequently used laboratory reagent so care must be taken when handling to prevent contamination of samples, reagents, and standards. Other commonly used cleaning compounds may also contain ammonia and contribute to contamination.

4.6. Since the method uses a heating coil, the carrier and buffer reagents must be degassed with helium. Bubble helium vigorously through solution for one minute to degas reagents.

5. SAFETY

5.1. Procedures shall be carried out in a manner that protects the health and safety of all Quanterra associates.

5.2. Eye protection that satisfied ANSI Z87.1 (as per the Chemical Hygiene Plan), laboratory coat, and chemically resistant gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.2.1. Neoprene, natural rubber, and butyl gloves provide varying degrees of protection against those chemicals listed. Refer to permeation/degradation charts for the actual data.

5.3. The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the Material Safety Data Sheets (MSDS) maintained in the laboratory.

5.3.1. The following materials are known to be **corrosive**: sodium phenolate, sodium hypochlorite, ammonia, sulfuric acid.

5.3.2. Phenol is highly toxic and can be readily absorbed through the skin. Wear gloves when handling. Avoid breathing the fumes by handling phenol in a fume hood. In case of skin contact, flush with water for at least 15 minutes. Notify your project manager or team leader or safety officer of any spills or exposures.

5.3.3. Sodium nitroprusside can release cyanide. Avoid contact with acids.

5.3.4. Exposure to chemicals must be maintained as low as reasonably achievable, therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.3.5. The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operations will permit.

5.3.6. All work must be stopped in the event of a known or potential compromise to the health and safety of a Quanterra associate. The situation must be reported immediately to a laboratory supervisor.

5.3.7. Wear gloves and apron when handling concentrated acid and bases. Transport only in approved carriers. Avoid breathing fumes and vapors; handle in a fume hood. Neutralize and clean up any spills immediately. In case of skin contact, flush affected area with water for at least 15 minutes. Notify your project manager or team leader or safety officer of any spills or exposures.

6. EQUIPMENT AND SUPPLIES

6.1. LACHAT QUIKCHEM AE Automated Flow Injection Ion Analyzer, consisting of:

6.1.1. Automatic sampler

6.1.2. Proportioning pump

6.1.3. Injection Module equipped with microloop

6.1.4. Colorimeter with 630 nm filter and 10 mm flow cell

6.1.5. Reaction module #10-107-06-1-A

6.1.6. Heating coil (60°C)

6.1.7. QuikCalc II Software System

6.1.8. Disposable autosampler vials

6.1.9. Miscellaneous laboratory apparatus and glassware.

7. REAGENTS AND STANDARDS

7.1. Sulfuric acid for Trace Element Analysis

7.1.1. Malinkrodt AR Select Sulfuric acid.

7.2. Sodium Phenolate:

7.2.1. In a hood, weigh and dissolve 83 g crystalline phenol (or 88 mL 88% liquefied phenol) in about 600 mL deionized water. While mixing, cautiously add 32 g sodium hydroxide and allow to dissolve. Cool to room temperature, dilute to 1 liter with deionized water, and mix well.

7.3. Sodium Hypochlorite:

7.3.1. Dilute 250 mL household bleach (5.25% NaOCl) to 500 mL with deionized water.

7.4. EDTA Buffer (5%):

7.4.1. Dissolve 50.0 g disodium ethylenediamine tetraacetate and 5.5 g sodium hydroxide in about 900 mL deionized water. Cool to room temperature, dilute to 1 liter, and mix well. Degas this reagent with helium just before use.

7.5. Sodium Nitroprusside:

7.5.1. Dissolve 3.5 g sodium nitroprusside in 1000 mL deionized water and mix.

7.6. Ammonia Stock Standard, 1000 mg/L as N:

7.6.1. Dry ammonium chloride at 105°C for one hour and cool in a desiccator. Dissolve 3.819 g in deionized water and dilute to 1 liter.

7.7. Intermediate Standard, 100 mg/L as N:

7.7.1. Dilute 10 mL 1000 mg/L Stock Standard to 100 mL with deionized water.

7.8. Working Standards:

7.8.1. Dilute the intermediate standard (10 mg/L) with deionized water as follows:

Std.	Aliquot (mL)	Final Volume (mL)	Conc. (mg/L as N)
A	5.0	100	5.0
B	2.0	100	2.0
C	1.0	100	1.0
D	0.5	100	0.5
F	0.1	100	0.1
G	0	---	0 (Blank)

7.9. DCS Solution: The DCS solution is a 2.0 mg/L mid-level standard.

7.10. A reference standard prepared from an independent source is also run.

7.10.1.1.NOTE: Store all reagents at room temperature.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Samples are to be collected in plastic or glass containers, acidified with sulfuric acid to pH < 2, and refrigerated to 4°C.

8.1.1. If 0.2 mL of sulfuric acid is added to the sample, then 0.2 mL of sulfuric acid must be added to the wash water and standards.

8.2. The holding time for Ammonia is 28 days.

9. QUALITY CONTROL

9.1. One method blank must be extracted with every process batch of similar matrix, not to exceed twenty (20) samples. The method blank is an aliquot of laboratory reagent water processed in the same manner and at the same time as the associated samples. Corrective actions must be documented on a Non-Conformance memo, then implemented when target analytes are detected in the method blank above the reporting limit or when surrogate recoveries are outside control limits. Re-extraction of the blank, other batch QC, and the affected samples are required when the method blank is deemed unacceptable. See Quanterra Inc. QA Policy 003 for specific acceptance criteria.

9.2. A Laboratory Control Sample (LCS) must be extracted with every process batch of similar matrix, not to exceed twenty (20) samples. The LCS is an aliquot of laboratory matrix (e.g. water, Ottawa sand, sodium sulfate, etc.) spiked with analytes of known identity and concentration. The LCS must be processed in the same manner and at the same time as the associated samples. Corrective actions must be documented on a Non-Conformance memo, then implemented when recoveries of any spiked analyte is outside control limits provided on the LIMS or by the client. Re-extraction of the blank, other batch QC and all associated samples are required if the LCS is deemed unacceptable. See Quanterra Inc. QA Policy 003 for specific acceptance criteria.

9.3. A Matrix Spike/Matrix Spike Duplicate (MS/MSD or MS/SD) pair must be extracted with every process batch of similar matrix, not to exceed twenty (20) samples. An MS/MSD pair are aliquots of a selected field sample spiked with analytes of known identity and concentration. The MS/MSD pair must be processed in the same manner and at the same time as the associated samples. Spiked analytes with recoveries or precision outside control limits must be within control limits in the LCS. Corrective actions must be documented on a Non-Conformance memo, then implemented when recoveries of any spike analyte is outside control limits provided on the LIMS or by the client. Re-extraction of the blank, an LCS, the selected field sample, the MS/MSD may be required after evaluation and review.

9.4. A duplicate control sample (LCSD or DCS) must be substituted when insufficient sample volume is provided to process an MS/MSD pair. The LCSD is evaluated in the same manner as the LCS. See Quanterra Inc. QA Policy 003 for specific acceptance criteria.

9.5. QC Samples

9.5.1. Aqueous samples require a reagent blank. For soil extractions, a prep blank is required with every batch of 20 or less samples.

9.5.2. Two DCS samples are required per 20 samples.

9.5.3. Duplicates may be required for project specific QC.

9.5.4. Matrix Spike and Matrix Spike Duplicate are prepared at a frequency of an MS/MSD pair per twenty samples as required by the California DOHS. Client requested QC may be used to satisfy this requirement.

9.6. Blanks must be less than the detection limit.

9.7. DCS/SCS recovery and DCS relative percent difference (RPD) must be within control limits. Current limits are in LIMS.

10. CALIBRATION AND STANDARDIZATION

10.1. Standard checks must be within 10% of the expected value.

10.2. The calibration curve must have a correlation coefficient of 0.995 or greater and must consist of a minimum of 5 standards in addition to the blank.

10.3. A reagent blank and an Initial Calibration Verification Standard (ICV) must be analyzed following initial calibration and prior to sample analysis.

10.4. A reagent blank and a Continuing Calibration Standard (CCV) are to be placed on the sampler after every 10 or less samples and also at the end of all samples. The CCV standard may be made from the same source as the initial calibration standards.

11. PROCEDURE

11.1. One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and is approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

11.2. Any unauthorized deviations from this procedure must also be documents as a nonconformance, with a cause and corrective action described.

11.3. Sample Preparation

11.3.1. Soils must be taken through the D.I. Leach procedure before analysis. See SOP # LM-CAL-1012.

11.3.2. Check the pH of all samples to ensure that they have been properly preserved. If necessary, add sulfuric acid to produce a pH of less than 2.

11.4. Instrument Start-up

11.4.1. Set up and operate the instrument in accordance to manufacturer's instructions. Install the Ammonia manifold according to manufacturer's instructions. Use deionized water as the carrier.

11.4.2. Inspect manifold for proper connections.

11.4.3. Prepare working standards. Refer to section 7.

11.4.4. Turn on the power to the system unit, pump, autosampler, and the software system. Allow the heating coil to warm up to 60°C. The control switch is located on the back of the system unit under the back cover.

11.4.5. Place reagent lines into proper containers. Fasten down the pump tube cassettes and tighten the tension one notch. Make sure the reagent tubes are in acceptable condition.

11.4.6. Pump reagents through all lines until all air bubbles have been pumped out.

11.4.7. Load the AMMONIA method onto the computer. The Ammonia background will appear on the slaved computer screen.

11.4.8. If the flow system is working properly, then the average baselines should be stable in approximately two minutes. No adjustment for the baseline locations or peak heights is necessary. The baseline locations and peak heights are already set in the software based on the highest standard solution. Depending on the age of the color reagent, peak heights might vary slightly.

11.5. Analysis

11.5.1. Fill the culture tubes with the working standards and place in the autosampler positions indicated by the letters corresponding to each standard in decreasing concentrations.

11.5.2. Place the ICV (DCS) in position 1 of the sampler, followed by the ICB and reference standard in positions 2 and 3 respectively. Additional blanks and DCS are to be analyzed at the beginning of every set of 20 or less samples. Reagent blanks and CCV are to be analyzed every 10 samples or less and at the end of all samples. If applicable, prep blank and DCS are run and counted as regular samples.

11.5.3. Load the autosampler with all samples, recording the location of each sample number, blank, DCS, etc. on the bench sheet. While the sampler is loading and is preparing to run, enter the recorded information into the computer. Be sure to record and enter any dilution or prep factors.

11.5.4. Start the analysis.

11.5.5. The instrument will analyze the standards and proceed with the samples if the correlation coefficient > 0.995. If the calibration curve is not acceptable, locate and correct the problem and then re-calibrate. The instrument should be monitored periodically to make sure that standard checks are in control and no other problems arise. The calibration statistics and raw data prints out at the end of the run.

11.5.6. Check the printout for off scale samples and rerun if necessary.

11.5.7. If additional samples need to be analyzed, reload the autosampler and enter the new tray information. Since the instrument is already calibrated, continue direct analysis of the new samples.

11.6. Instrument Shut-Down

11.6.1. Place all reagent lines in deionized water and pump for five minutes. Then remove the lines from the water and pump the system dry.

11.6.2. Drain the water out of the wash vessel.

11.6.3. Turn off the units and unfasten the pump tube cassettes from the pump. Release the tension levers.

11.6.4. Clean up the work area and replace any reagents which have been depleted.

12. METHOD PERFORMANCE

12.1. The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required expertise.

13. POLLUTION PREVENTION

13.1. No solvents of any kind or in any amount are to be disposed of in the sinks or evaporated in the hoods.

14. WASTE MANAGEMENT

14.1. Waste management in the procedure must be segregated and disposed according to the facility hazardous waste procedures. The Environmental Health and Safety Coordinator or Hazardous Materials Technician should be contacted if additional information is required.

14.2. All waste must be disposed of according to the facility hazardous waste management procedures, Attachment C of the Chemical Hygiene Plan, Section WS002, Table 1.

14.3. Samples and other solutions containing high concentrations of toxic materials must be disposed of according to the facility hazardous waste management procedures, Attachment C of the Chemical Hygiene Plan, Section WS003, Disposal of Samples After Analysis.

15. REFERENCES

15.1. Method source: Lachat Method 10-107-06-1-A (equivalent to EPA Method 350.1)

16. MISCELLANEOUS

16.1. Deviations from reference method.

16.1.1. The range was modified from 0.1-20 mg/L to 0.1-5.0 mg/L.

16.2. Summary of modifications to SOP from previous revisions.

16.2.1. This SOP was modified to be consistent with the Lachat AE system.

16.3. List of other SOPs cross-referenced in SOP.

16.3.1. SAC-QA-0041 Calibration and Calibration Check of Balances, Revision 1.1.

16.3.2. QA Policy 003 Quanterra Quality Control Program, Revision 1.

16.3.3. QA Policy 008 Data Recording Requirements, Rev. 1.

16.3.4. Chemical Hygiene Plan, Attachment C, Hazardous Waste Management Procedures, Revision 0.

16.3.5. LP-CAL-0001 Laboratory Documentation Practices, Revision 3.0.

16.3.6. LM-CAL-1012 Deionized Water Leach Extraction for Wet Chemistry Analyses, Revision Original.

16.4. Facility-specific information required to implement SOP.

16.5. Tables or figures referenced in body of SOP.

16.6. Method appendices.

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OPERATION-SPECIFIC STANDARD OPERATING PROCEDURE

TITLE: DETERMINATION OF NITRATE/NITRITE BY AUTOMATED COLORIMETRY-CADMIUM REDUCTION METHOD (MCAWW 353.2, SM4500-NO3 F)

(SUPERSEDES: LM-CAL-1036, REV. 2)

Prepared by: Hamid Foolad

Reviewed by: _____
Technical Specialist, Josefina P. Jones

Approved by: _____
Quality Assurance Manager, Gail Cho Celaschi

Approved by: _____
Environmental Health and Safety Coordinator, Cheryl Ann Stith

Approved by: _____
Laboratory Manager, Robert Dovi

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C4-573

1. SCOPE AND APPLICATION

1.1. This method determines nitrate and/or nitrite in surface and saline waters, domestic and industrial wastes. This SOP is based on EPA MCAWW 353.2 and Standard Method 4500-NO3 F.

1.2. Non-aqueous samples can be analyzed by this method following extraction using deionized water. See the SOP "Deionized Water Leaching Procedure for Non-aqueous Samples".

1.3. Nitrite is determined concurrently with nitrate + nitrite by the use of a separate manifold configured without the cadmium reduction column. Nitrate is calculated by subtracting the nitrite concentration from the combined nitrate + nitrite.

1.4. The analytical range of this method is 0.025 to 1.0 mg/L for nitrite and 0.05 to 2.0 mg/L for combined nitrate + nitrite. Samples that are over the linear range must be diluted and reanalyzed.

1.5. The reporting limit for each analyte is 0.05 mg/L as N.

1.6. This method is restricted to use by, or under the supervision of, analysts experience in the operation of Lachat Auto-Analyzer and the interpretation of its results.

2. SUMMARY OF METHOD

2.1. Samples are analyzed on the Lachat QuikChem AE, an automated colorimetric system configured with a cadmium reduction column. A filtered (optional) sample is passed through a column comprised of granulated copperized cadmium to reduce nitrate to nitrite. The reduced nitrate and any nitrite originally present in the sample are diazotized with the sulfanilamide, then coupled with N-(1-naphthyl)-ethylenediamine dihydrochloride to form a highly colored azo dye that is measured colorimetrically at 520 nm. The absorbance of this dye is directly proportional to the concentration of nitrate + nitrite as N.

3. DEFINITIONS

3.1. Definitions of terms used in this SOP may be found in the glossary of the Quality Assurance Management Plan (QAMP).

4. INTERFERENCES

4.1. Suspended matter builds up in the column and restricts samples flow. Filter the sample using 0.45 um filters to remove particulates.

4.2. Concentrations of iron, copper, or other metals above several milligrams per liter lower the reduction efficiency thus causing low results. EDTA is added to samples to eliminate this interference.

4.3. Oil and grease will coat the cadmium surface causing low results. This interference can be removed from samples by pre-extraction with organic solvent such as chloroform, methylene chloride, or another suitable solvent.

4.4. Samples preserved with mercuric chloride or sodium thiosulfate will degrade the reduction column and should not be analyzed by this method.

4.5. Physical interferences such as color and turbidity in the samples will cause high results and can be minimized by filtration or dilution.

4.6. Samples may contain interferences that cause the blue-colored azo-complex to dissipate upon extended exposure to air. It is recommended that the sample be loaded onto the autosampler near or just before injection to minimize exposure to air.

5. SAFETY

5.1. Procedures shall be carried out in a manner that protects the health and safety of all Quanterra associates.

5.2. Eye protection that satisfied ANSI Z87.1 (as per the Chemical Hygiene Plan), laboratory coat, and chemically resistant gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.2.1. Neoprene, natural rubber, and butyl gloves provide varying degrees of protection against those chemicals listed. Refer to permeation/degradation charts for the actual data.

5.3. The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the Material Safety Data Sheets (MSDS) maintained in the laboratory.

5.3.1. Chemicals that have been classified as **carcinogens**, or **potential carcinogens**, under OSHA include: cadmium.

5.3.2. The following materials are known to be **corrosive**: sodium hydroxide, hydrochloric acid, copper sulfate, and sulfanilamide reagent.

5.3.3. The following material is known to be **flammable**: acetone.

5.4. Exposure to chemicals must be maintained **as low as reasonably achievable**, therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.5. The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operation will permit.

5.6. All work must be stopped in the event of a known or potential compromise to the health and safety of a Quanterra associate. The situation must be reported **immediately** to a laboratory supervisor.

6. EQUIPMENT AND SUPPLIES

6.1. Lachat QuikChem AE Automated Flow Injection Ion Analyzer, consisting of:

6.1.1. Automatic sampler

6.1.2. Proportioning pump

6.1.3. Injection Module equipped with microloop

6.1.4. Colorimeter with 520 nm filter and 10 mm flow cell

6.1.5. Reaction module #10-107-04-1-C with Cd column

6.1.6. QuikChem II Software System

6.2. Disposable autosampler vials or culture tubes, 12 x 75 mm for samples and standards.

6.3. Acrodisk filters, 0.45 micron.

6.4. Miscellaneous laboratory apparatus and glassware.

7. REAGENTS AND STANDARDS

7.1. Sodium Hydroxide, 15 N: Add 150 g of sodium hydroxide pellets into a 250 mL volumetric flask containing at least 200 mL of deionized water and mix until dissolved. Cool to room temperature, bring to final volume of 250 mL. Store in a plastic bottle, cap tightly. This solution is stable for 1 year.

CAUTION: This mixture will get extremely hot and give off irritating fumes. Prepare in a hood.

7.2. Ammonium Chloride-EDTA Solution (Buffer solution): Dissolve 85.0 g ammonium chloride and 1.0 g disodium ethylenediamine tetraacetic acid dihydrate in about 800 mL deionized water in a 1 liter volumetric flask. Adjust the pH up to 8.5 by adding 15 N sodium hydroxide solution dropwise. Use pH meter and dilute to final volume of 1 liter with deionized water. This solution is stable up to one year.

7.3. Sulfanilamide Reagent: In a 1 liter flask containing at least 600 mL of deionized water, carefully add 100 mL of 85% phosphoric acid (H_3PO_4) and mix gently. Add 40.0 g sulfanilamide and 1.0 g N-1-naphylethylenediamine dihydrochloride (NED) and mix until dissolved. Bring the final volume to 1 liter. This solution is light sensitive, store this solution in a dark bottle. This solution is stable for at least one month. Discard if severe discoloration occurs prior to the one month shelf life.

7.4. Cadmium-Copper Reduction Column Preparation:

7.4.1. Hydrochloric Acid, 1 M: Carefully add 8 mL of concentrated hydrochloric acid (HCl) into a 100 mL volumetric flask or 100 mL graduated cylinder containing at least 90 mL of deionized water. Cap flask or cover the graduated cylinder with parafilm and mix solution gently. This solution is stable for at least 1 year.

7.4.2. Copper Sulfate, 2%: Dissolve 20 g $CuSO_4 \bullet 5\ H_2O$ in deionized water and dilute to 1000 mL.

7.4.3. Cadmium Preparation:

7.4.3.1. Place 10 to 20 g of cadmium granules (0.3 - 1.5 mm mesh diameter) into a 250 mL Erlenmeyer flask.

7.4.3.2. Wash with two 50 mL portions of acetone, then rinse with 50 mL deionized water for at least 3 times, followed by two 50 mL portions of 1 M hydrochloric acid.

7.4.3.3. Rinse several times with deionized water. Dispose of all washings and rinses in a suitable waste container.

7.4.4. Copperization of the cadmium

7.4.4.1. Pour about 50 mL 2% copper sulfate into the flask containing the prepared cadmium granules from 7.4.3.3 and swirl for 5 minutes.

7.4.4.2. Decant the liquid and repeat with another 50 mL portion of 2% copper sulfate. Continue this process until the blue color persists in the liquid.

7.4.4.3. Decant and wash with at least 5 portions of Ammonium Chloride-EDTA Solution (See Section 7.2). At this point, the cadmium granules should be black or dark gray. The copperized cadmium granules may be stored in a tightly capped bottle under Ammonium Chloride-EDTA Solution. Do not expose them to air. **Waste containing copper sulfate should not be poured down the drain, but placed in a suitable waste container for proper disposal.**

7.4.5. Packing Column: **Wear gloves!** Be careful not to chip or break the glass threads on the column.

7.4.5.1. Clamp the empty column in a upright position and unscrew the upper fitting.

7.4.5.2. Place a foam plug in the bottom end of the column. Fasten the fitting attached to the bottom end of the column with a disposable syringe and fill the column, tubing, and fittings with Ammonium Chloride-EDTA buffer solution.

7.4.5.3. Scoop the copperized cadmium granules with a spatula into the top end of the column so that they settle to the bottom of the column. Continue filling with cadmium granules, tapping the column with a

screwdriver handle to dislodge any air bubbles and prevent any gaps in the column.

7.4.5.4. When the column is filled to within about 2 mm of the top, cap the upper end of the column with the foam plug and reattach the fitting. Connect the two ends of the column to prevent air from getting inside.

7.4.5.5. Rinse the outside of the column with deionized water. If air remains in the column or is introduced accidentally, push the air out by using the disposable syringe filled with buffer solution and tapping the column gently with the screwdriver handle until all air bubbles are dislodged.

WARNING: Air bubbles interfere with sample flow. Do not let any air bubbles get into the reduction column. Cadmium granules should be kept in buffer solution at all times. The column must be taken off line through the blue valve while buffer is flowing through it.

7.5. Nitrate Stock Standard, 1000 mg/L as N: Dissolve 7.218 g KNO_3 in deionized water and dilute to 1 liter in a volumetric flask. This solution is stable for 6 months.

7.6. Nitrite Stock Standard, 1000 mg/L as N: Dissolve 4.925 g $NaNO_2$ in deionized water and dilute to 1 liter in a volumetric flask. This solution is stable for 6 months.

7.7. Nitrate + Nitrite Intermediate Standard, 10 mg/L NO_2/NO_3: Dilute 1.0 mL of each of the 1000 mg/L Stock Standards in Sections 7.5 and 7.6 to 100 mL with deionized water. Prepare fresh monthly.

7.8. Stock reference standards for Nitrate, Nitrite, Nitrate + Nitrite (1000 mg/L): Prepare as in sections 7.5, 7.6, 7.7 but use neat material from a second source (vendor or lot number). An ERA Nutrients reference standard or other premade standard can also be use as a second source verification standard.

7.9. Working Standards: Dilute the two intermediate standards (10 mg/L of NO_2+NO_3) with DI water into each volumetric flask as follows:

Std	Aliquot (mL)	Final Volume (mL)	Conc. (mg/L as N)*
A	10	100	1.0
B	5	100	0.5

C	2.5	100	0.25
D	1	100	0.10
E	0.5	100	0.05
F	0.25	100	0.025
G	0	---	BLANK

7.9.1. Working standards must be prepared monthly.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Samples are collected in plastic bottles and stored refrigerated at 4°C ± 2.

8.2. For nitrate **or** nitrite determination only, samples are unpreserved and must be analyzed within 2 days (48 hours) from the collection date.

8.3. For determination of nitrate + nitrite combined, samples are preserved with 2 mL of concentrated H_2SO_4 per liter of sample to pH $\leq$2. When preserved, the holding time is extended to 28 days from the collection date.

8.4. For leachates and extracts, analysis must be performed within 48 hours from completion of the extraction/preparation, including filtration.

9. QUALITY CONTROL

9.1. One method blank must be extracted with every process batch of similar matrix, not to exceed twenty (20) samples. The method blank is an aliquot of laboratory reagent water processed in the same manner and at the same time as the associated samples. Corrective actions must be documented on a Non-Conformance memo, then implemented when target analytes are detected in the method blank above the reporting limit. Re-extraction of the blank, other batch QC, and the affected samples are required when the method blank is deemed unacceptable. See Quanterra Inc. QA Policy 003 for specific acceptance criteria.

9.1.1. When no independant leaching or extraction procedure is performed, the ICB/CCB will be used and evaluated as the method blank.

9.2. A Laboratory Control Sample (LCS) must be extracted with every process batch of similar matrix, not to exceed twenty (20) samples. The LCS is an aliquot of laboratory matrix (e.g. water, Ottawa sand) spiked with analytes of known identity and concentration. The LCS must be processed in the same manner and at the

same time as the associated samples. Corrective actions must be documented on a Non-Conformance memo, then implemented when recoveries of any spiked analyte is outside control limits provided on the LIMS or by the client. Reextraction and/or reanalysis of the blank, other batch QC and all associated samples are required if the LCS is deemed unacceptable. See Quanterra Inc. QA Policy 003 for specific acceptance criteria.

9.2.1. When no independant leaching or extraction procedure is performed, the ICV/CCV will be used, evaluated, and reported as the LCS. ICV/CCV control limits will apply (i.e. ±10% from the true value) and corrective actions will be applied to affected samples, i.e. the associated and bracketed samples reanalyzed.

9.3. When solid samples are assayed by this procedure, a duplicate aliquot of a solid sample will be leached as part of the process batch, not to exceed 20 samples. The leachates will be the be spiked and then processed as MS/MSDs. The pair must be processed in the same manner and at the same time as the associated samples. Spiked analytes with recoveries or precision outside control limits must be within control limits in the LCS. Corrective actions must be documented on a Non-Conformance memo, then implemented when recoveries of any spike analyte is outside control limits provided on the LIMS or by the client. Re-extraction of the blank, an LCS, the selected field sample, the MS/MSD may be required after evaluation and review.

9.4. A duplicate control sample (LCSD or DCS) must be substituted when insufficient sample volume or matrix prevents processing of an MS/MSD pair. The LCSD is evaluated in the same manner as the LCS. See Quanterra Inc. QA Policy 003 for specific acceptance criteria.

9.4.1. When no independant extraction or leaching is performed, consecutive CCVs will be used, evaluated, and reported as the LCS/LCSD pair. The ICV/CCV acceptance criteria and corrective actions will be applied, i.e. ±10% from the true value, reanalyze associated and bracketed samples.

10. CALIBRATION AND STANDARDIZATION

10.1. The instrument maximization is part of the instrument set-up and pre-programmed internally.

10.2. Instrument calibration is done daily before any samples are analyzed. The calibration curve consists of five (5) standards and a blank. The correlation coefficient of the curve must be at least 0.995 or greater.

10.3. An Initial Calibration Verification Standard (ICV) and a reagent blank (ICB) must be analyzed immediately following initial calibration and prior to sample analysis.

10.3.1. The ICV/CCV standard concentration is the mid-point concentration of the calibration curve and must be within 10% of the expected value. If not, correct the problem and reanalyze any affected samples back to the last CCV. Perform a new multipoint curve if the ICV fails upon reanalysis.

10.3.2. The ICB/CCB value must be less than the reporting limit.

10.4. A Continuing Calibration Verification (CCV) and reagent blank (CCB) are to be placed on the autosampler after every ten or less samples and at the end of the run.

10.5. Corrective actions and suggestions for repeated failures of calibration verifications and calibration blanks.

10.5.1. Check for contamination in the reagents and standards.

10.5.2. Be sure all reagents were made correctly and have not exceeded their expiration dates.

10.5.3. Check system for obvious problems such as plugs, leaks, and pump tubes.

10.5.4. Identify samples may deactivate the reduction column. Samples may need to be diluted and reprepared. Consult with the Area Leader before continuing.

10.5.5. Analyze a 2 mg/L Nitrite-N standard and compare to a 2 mg/L Nitrate-N standard., If the results differ by more than 15%, the column has deteriorated and needs to be repacked.

10.5.6. If you are unable to locate and solve the problem, consult your supervisor for further corrective actions.

11. PROCEDURE

11.1. One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and approved by a Technical Specialist and QA Manager. If contractually required,

the client shall be notified. The Nonconformance Memo shall be filed in the project file.

11.2. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

11.3. This SOP was written explicitly for the Lachat QuikChem AE system and must be followed in order to work with the existing programs in the instrument. Any deviations from this SOP may necessitate modifications to the program. Also, any changes in the program may require alterations in this SOP.

11.4. Sample Preparation

11.4.1. Soils are leached following the SOP "Deionized Water Leaching Procedure for Non-Aqueous Samples".

11.5. Instrument Start-up

11.5.1. The instrument is to be set up and operated in accordance to the manufacturer's instructions. Install the nitrite and nitrate+nitrite manifolds according to manufacturer's instructions. Nitrite may be analyzed alone if the reduction column is bypassed. The carrier will be deionized water for unpreserved samples.

11.5.2. Inspect manifold for proper connections. Make sure that there is sufficient volume for all reagents for the entire run. All reagents and standards must be within the expiration dates.

11.5.3. Prepare working standards as stated in Section 7.

11.5.4. Turn on the power to the system unit, pump, autosampler, and the software system. The heating coil is not needed and must be turned off.

11.5.5. Place all lines into an Erlenmeyer flask containing deionized water for rinsing purposes only (approximately 5 min.). This step is recommended to ensure that the system does not retain any contaminants prior to initiation of the analysis.

11.5.6. Place reagent lines into proper containers. Fasten down the pump tube cassettes and tighten the tension one notch. Make sure the reagent tubes are in good working condition.

11.5.7. Pump reagents through all lines until all air bubbles have been pumped out.

11.5.8. Bring the cadmium column on line by turning on the blue valve on the NO_2+NO_3 manifold. Make sure there are no air bubbles inside the flow system.

11.5.9. Load the NITRATE+NITRITE method in the computer. You will see the NO_2+NO_3 and NO_2 backgrounds on the computer screen.

11.5.10. If the flow system is working properly, the average baselines should be stable in about two minutes. No adjustment for the baseline locations or peak heights is necessary. The baseline locations and peak heights are already set in the software based on the highest standard solution. Depending on the age of the color reagent, peak heights can be slightly different from previous runs.

11.6. Analysis

11.6.1. Check the pH of samples by using pH paper. If the sample pH is below 5 or above 9, adjust to between 5 and 9 with either conc. H_2SO_4 or conc. NaOH. Document the pH adjustment on the associated sample paperwork.

NOTE: If pH adjustment is not performed, then standards must be matrix-matched to the acid-preserved NO3 + NO2 samples. Adjust standards with dropwise additions of conc. H_2SO_4 to pH 2.

11.6.2. Fill the sample tubes with the working standards in **decreasing concentration** and place in the autosampler positions indicated by the letters corresponding to each standard. (Example: spot A should have the highest standard).

11.6.3. Place the ICV (LCS) in position 1 of the sampler, followed by the ICB and reference standard in positions 2 and 3 respectively. Additional blanks and LCSs are to be analyzed at the beginning of every 20 or less samples. If applicable, prep blanks and additional LCSs are run and counted as regular samples.

11.6.4. Load the autosampler with the samples, recording the location of each sample number, blank, LCS, etc., in the instrument run logbook. When the sampler is loaded and ready to run, enter this information into the computer. Be sure to record and enter any dilution or prep factors.

11.6.5. Start the analysis. The instrument will analyze the standards and proceed with the samples if the correlation coefficient > 0.995. If the calibration curve is not acceptable, locate and correct the problem and then recalibrate. The instrument should be monitored periodically to make sure that standard checks are in control and no other problems arise. The calibration statistics and raw data should be printed at the end of the run.

11.6.6. Check the printout for off scale samples and rerun if needed.

11.6.7. If additional samples are to be analyzed, reload the autosampler and enter the new tray information. Since the instrument is already calibrated, you may proceed directly to analysis of the new samples using a different batch identification.

11.7. Instrument Shut-Down

11.7.1. Take the cadmium column off line by turning the blue valve off. If the column is still in acceptable condition, you may leave it connected on the manifold for the next use.

11.7.2. Place all reagent lines in deionized water and pump at normal speed for at least 5 minutes. Then remove the lines from the water and pump the system dry.

11.7.3. Drain the water out of the wash vessel.

11.7.4. Turn off the units and unfasten the pump tube cassettes from the pump. Release the tension levers.

11.7.5. Clean up the work area and replace any reagents which have been depleted.

11.8. Calculations:

11.8.1. Nitrate is calculated manually by subtracting the Nitrite value from the Nitrate + Nitrite value.

11.8.2. Nitrocellulose (NC) calculation (see appropriate SOP):

$$\text{Conc. In sample extract (mg N / L)} = \frac{\text{Instrument Response} - \text{Y Intercept}}{\text{Slope}}$$

11.8.2.1.The final result is calculated by the following equation:

$$\text{Final Results (mg NC/g)} = A \times B \times C$$

Where:

A = Concentration in sample extract (mg N/L)

Conversion from mg/L to µg/L : mg/L × 1000 µg/mg = 1000 µg/L

B = Concentration Factor

$$= \frac{\text{Volume of Extract}}{\text{Weight of Sample taken for Analysis (g)}} = \frac{0.1\ \text{L}}{0.5\ \text{g}} = 0.2\ \text{L / g}$$

C = Conversion Factor

$$= \frac{\text{ug NC/ g}}{\text{ug N/ g}} = \frac{1}{0.1325} = 7.547$$

Note: Conversion Factor is based on the assumption that Nitrocellulose (NC) is 13.25% N.

11.8.3. For % recovery and %RPD calculations, see Policy QA-003.

12. METHOD PERFORMANCE

12.1. The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required expertise.

13. POLLUTION PREVENTION

13.1. No solvents of any kind or in any amount are to be disposed of in the sinks or evaporated in the hoods.

14. WASTE MANAGEMENT

14.1. Waste management in the procedure must be segregated and disposed according to the facility hazardous waste procedures. The Environmental Health and Safety Coordinator or Hazardous Materials Technician should be contacted if additional information is required.

14.2. All waste must be disposed of according to the facility hazardous waste management procedures, Attachment C of the Chemical Hygiene Plan, Section WS002, Table 1.

14.3. Samples and other solutions containing high concentrations of toxic materials must be disposed of according to the facility hazardous waste management procedures, Attachment C of the Chemical Hygiene Plan, Section WS003, Disposal of Samples After Analysis.

15. REFERENCES

15.1. EPA MCAWW 353.2 Nitrogen, Nitrate-Nitrite (Colorimetric, Automated, Cadmium Reduction).

15.2. Standard Method 4500-NO3 F. Automated Cadmium Reduction Method, 18th Edition 1992.

15.3. Method source: Lachat Method 10-107-04-1-C equivalent to EPA Method 353.2

15.4. The Determination of Nitrocellulose in water by Colorimetric Autoanalyzer. DataChem Laboratories, Version 1, January 15, 1991.

16. MISCELLANEOUS (TABLES, APPENDICES, ETC.)

16.1. Deviations from the referenced methods.

16.1.1. Instrumentation : Lachat Quickem AE instead of Technicon Autoanalyzer. Both instruments are compatible and designed for this method.

16.1.2. Linear range is designed based on the manufacturer's recommendation of 0.05 to 2.0 mg/L instead of 0.05 to 10.0 mg/L.

16.1.3. Absorbance is 520 nm instead of 540 nm.

16.1.4. Sodium Hydroxide is used in place of Ammonium Hydroxide for pH adjustment.

16.1.5. Sulfuric Acid is used in place of HCl for pH adjustment.

16.1.6. Reagent preparation are performed according to the manufacturer's instructions and may differ from the method.

16.1.7. Shelf life for standard curve of daily is changed to monthly and was based from the in-house stability study performed for these analytes.

16.1.8. Preservation with Chloroform is omitted. Shelf life on stock standards lasts for at least 1 year without preserving with chloroform.

16.1.9. From the reference material pertaining analysis of Nitrocellulose, the standard preparation, calibration and criteria is not clearly defined and therefore the analytical protocols for Nitrate are adopted for this analyte. Dedicated section for the conversion of Nitrate as N to Nitrocellulose (NC) is included in this SOP.

16.2. Summary of modifications to SOP from previous revisions.

16.2.1. The SOP was modified to be consistent with the use of the LACHAT AE Autoanalyzer.

16.2.2. The new QA-003 policy is incorporated.

16.2.3. Quanterra SOP format adopted.

16.3. List of other SOPs cross-referenced in SOP.

16.3.1. LP-CAL-0001 Laboratory Documentation Practices, Revision 3.0.

16.3.2. QA Policy 003 Quanterra Quality Control Program, Revision 1.

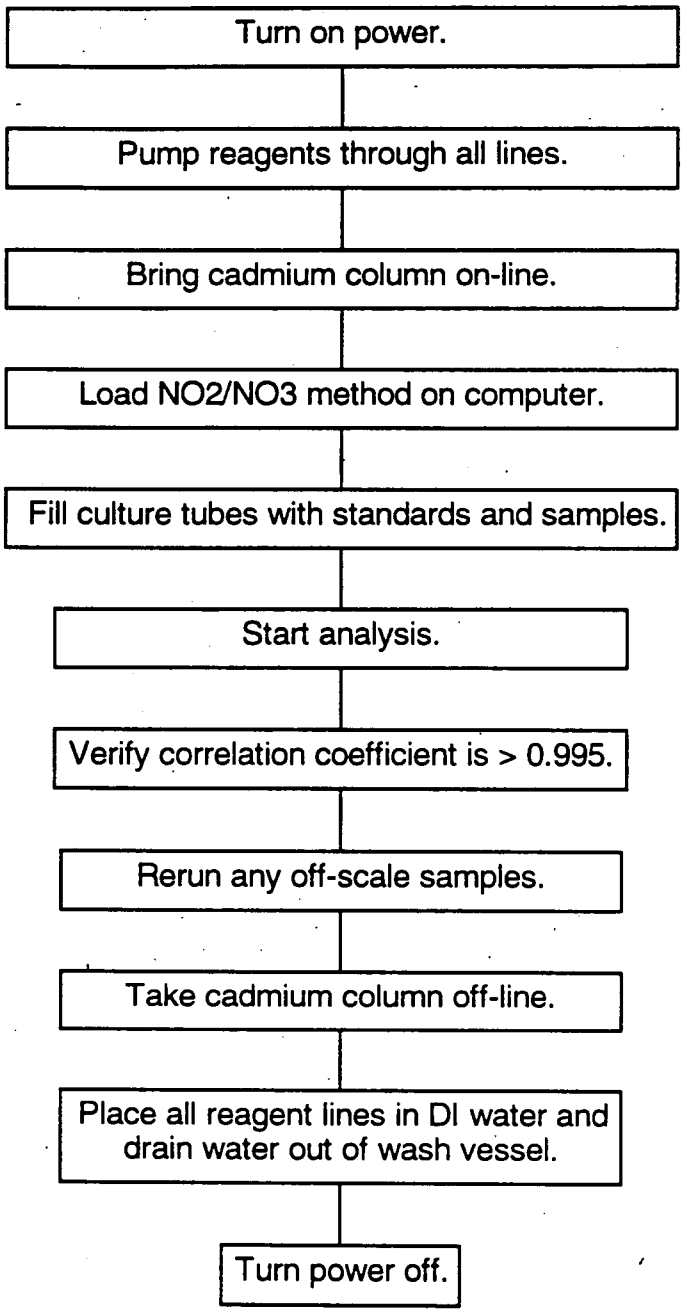
16.3.3. QA Policy 008 Data Recording Requirements, Rev. 0.

16.3.4. Chemical Hygiene Plan, Attachment C, Hazardous Waste Management Procedures, Revision 0.

16.3.5. Deionized Water Leaching Procedure for Non-Aqueous Samples.

16.3.6. Extraction of Nitrocellulose from water and soils samples.

Nitrate/Nitrite by Lachat AE Autoanalyzer
SAC-WC-0036



C4-589

Appendix 1

Example Forms

(Reviewer Checklist)
(Example Lachat Report)
(Instrument Run Log)
(Non-Metals Extraction Log)

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STANDARD OPERATING PROCEDURE

Subject of Title: **OIL AND GREASE, GRAVIMETRIC (EPA 413.1 AND EPA 9070)**

SOP No.:	Revision:	Effective Date:
LM-CRL-1026	0	February 1, 1992
	Supersedes: none	

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Prepared by: David C. Dawes — Date: 3/11/92

Management Approval: [signature] — Date: 3/23/92

QA Officer Approval: Paul Christi — Date: 3/27/92

C4-591

1. Scope and Application

1.1 This method includes the measurement for fluorocarbon-113 (freon) extractable matter from surface and saline waters, industrial and domestic wastes. It is applicable to the determination of relatively non-volatile hydrocarbons, vegetable oils, animal fats, waxes, soaps, greases and related matter. It is NOT applicable to the measurement of light hydrocarbons that volatilize at temperatures below 70 degrees Centigrade.

1.2 Reporting limits: 5 mg/L for liquids
100 mg/Kg for solids

1.3 Applicable matrices: Water, soil, solids, sludge and waste.

1.4 Dynamic range: This method covers the range from 5 to 1000 mg/L of extractable material for liquids. The range for solids is from 100 to 100,000+ mg/Kg.

1.5 Analysis time: Total hands-on time for analysis of samples is approximately 30 minutes per sample (15 samples + 3 QC per shift). Because of drying, total time for analysis is approximately 24 hours for 1 to 15 samples.

2. Method Summary

2.1 Oil and grease is determined differently depending on whether sample matrix is liquid or solid.

2.1.1 For liquids, an acidified sample (pH<2) is serially extracted with freon in a separatory funnel. The solvent is then evaporated from the extract and the residue weighed.

2.1.2 For solids, a representative aliquot of the sample is placed in a glass container with sodium sulfate and serially extracted with freon. The solvent is then evaporated from the extract and the residue weighed.

3. Comments

3.1 Interferences

3.1.1 Matrix interference is likely if samples contain any non-oil substances that dissolve in freon.

3.1.2 Scrupulous care must be taken to avoid any water being extracted which will lead to erroneously high results.

4. Safety Issues

4.1 All employees are expected to be familiar with and follow the procedures outlined in the Enseco safety plan. Lab coats and safety glasses are required in all laboratory areas at all times. Consult your supervisor or safety officer with any specific questions or safety concerns.

4.2 Wear gloves when handling concentrated acids and solvents. Transport them only in approved containers. Avoid breathing fumes and vapors of freon by venting separatory funnels and evaporating extracts inside a fume hood.

4.3 The toxicity or carcinogeneity of each chemical used in this procedure has not been precisely defined. Each chemical and sample should be treated as a potential health hazard.

4.4 A Material Safety Data Sheet (MSDS) is available for each laboratory reagent used in this analysis. These should be reviewed by the analyst before handling the chemicals.

5. Sample Collection and Preservation

5.1 Liquid samples are collected in 1 liter glass bottles. If analysis is to be delayed for more than a few hours, the sample is preserved by the addition of 1 mL concentrated sulfuric acid at the time of collection and refrigerated at 4° C.

5.2 Solid samples are to be sealed and refrigerated at 4° C.

5.3 The holding time for liquids is 28 days from the date of sampling. There is no defined holding time for solid samples.

6. Apparatus

6.1 Separatory funnel, 2000 mL with Teflon stopcock.

6.2 Boiling flask, 125 mL with ground glass joint.

6.3 Distilling arm to fit boiling flask and collection bottle.

6.4 Collection bottle, 1000 mL capacity.

6.5 Filter paper, Whatman 1/PS 11 cm diameter.

6.6 Glass funnel for filter paper.

6.7 Water bath, 70° C.

6.8 Glass bottles, 6 ounce, with screw cap (for solids).

6.9 pH indicator paper, 0-14 range.

6.10 Graduated cylinder, 1000 mL

7. Reagents and Standards

7.1 Sulfuric acid, concentrated.

7.2 Freon-113, 1,1,2-trichloro-1,2,2-trifluoroethane(freon), b.p.=48° C.
NOTE: Each new batch of freon must be analyzed by NDIR (EPA 413.2) to verify that it contains less than 5mg/L oil and grease.

7.3 Sodium sulfate, anhydrous crystal.

7.4 DCS solution. Weight 1 gram 3-in-1 oil to the nearest 0.1 mg into a 100mL volumetric flask and dilute to mark with freon. Calculate spike amount based on 5mLs in 1000mL DI water. Refrigerate at 4° C. EXPIRES IN 3 MONTHS.

8. Procedure

8.1 For liquid samples:

8.1.1 Mark the sample bottle at the water meniscus for later determination of sample volume.

8.1.2 Measure sample pH with indicator paper. If pH is not less than 2 then add 1mL concentrated sulfuric acid to sample and mix. Check pH again. If not less than 2, add subsequent mLs of acid until it is less than pH 2. Record on benchsheet any sample that is unpreserved.

8.1.3 Pour the sample into a separatory funnel.

8.1.4 Tare a boiling flask that has been pre-dried at 103° C. and desiccated until cool to the nearest 0.1 mg.

8.1.5 Add 30mL freon to the empty sample bottle and rotate to rinse the sides. Transfer the solvent into the separatory funnel. Extract by shaking vigorously for 2 minutes. Allow the layers to separate then filter the lower layer (freon) through 1 gram sodium sulfate salt placed in the filter paper cone. Both filter paper and salt should be moistened with solvent before sample is added.
Note 1: If an emulsion forms, drain it slowly through the sodium sulfate. Add more salt as required.
Note 2: To avoid having water penetrate the filter paper, leave a small amount of freon behind in the funnel after each extraction.

8.1.6 Repeat 8.1.5 twice more with additional portions of fresh solvent. (Do not rinse container further.) Combine all three extracts in the flask.

8.1.7 Rinse the separatory funnel tip, the filter paper and sodium sulfate with 10-20mLs freon. Add this to the extract already in the flask

8.1.8 Connect the boiling flask to the distilling arm and evaporate the solvent by immersing the lower half of the flask in water at 70° C.

8.1.9 When the flask appears dry, remove it from the water bath. Carefully wipe the flask down to remove excess water and then place it in a drying oven set at 60° C for 1 hour. Desiccate flask until cool.

8.1.10 Weigh the flask to the nearest 0.1 mg and record on benchsheet. If weight is not stable then return flask to desiccator for further cooling.

8.1.11 Determine volume of sample used by filling sample bottle to meniscus mark (8.1.1) with water and then transferring to a 1000mL graduated cylinder.

8.2 For soil samples:

8.2.1 Weigh out 10 grams sample to the nearest 0.1 gram. Place sample in a 6 ounce glass bottle.

8.2.2 Add 30mL freon to bottle and shake vigorously for 10-15 minutes (mechanical shaker can be used).

8.2.3 Pour extract through moistened sodium sulfate salt and filter paper into a tared flask as described in 8.1.5. Add more salt as needed to prevent water from passing through the filter.

8.2.4 Repeat 8.2.2 and 8.2.3 twice, combining all three extracts in the tared flask.

8.2.5 Rinse the salt and filter paper with 10-20 mLs of additional freon and combine with extracts.

8.2.6 Evaporate solvent and dry flask as described in 8.1.8 through 8.1.10.

9. Calculation

9.1

$$\text{mg/L or mg/Kg oil and grease} = \frac{R - T}{A} \times 1{,}000{,}000$$

where:

R = residue plus flask weight in grams
T = tare weight of flask in grams
A = aliquot of sample, liquids in mLs, solids in grams.

10. QA/QC Requirements

10.1 QC Samples

10.1.1 One blank is required with every batch of 20 or less samples. A blank consists of 1000mL acidified DI water which is taken through all steps of the analysis.

10.1.2 A pair of LCS samples (DCS) is required with every 20 or less samples. An LCS consists of 5mL DCS solution (7.4) added to 1000 mL DI water, acidified, and taken through all steps of the analysis.

10.2 Acceptance Criteria

10.2.1 Recovery and RPD on DCS samples must be within laboratory control limits which are updated periodically.

10.2.2 Blank result must be less than the reporting limit.

10.3 Corrective Action

10.3.1 If the blank reads greater than the reporting limit but is less than twice its value then the data can be reported with a notation. If the blank is greater than twice the reporting limit then the data is rejected. In either case, the solvent should be re-checked by NDIR and discarded if found to be greater than 5 mg/L. If the solvent is "clean" then all transfer containers should be checked for contamination and cleaned.

10.3.2 If the DCS average percent recovery and/or the RPD are outside of control limits then the data is rejected. The DCS solution should be discarded.

11. Reporting

11.1 Report all results as either mg/L or mg/Kg Oil and Grease.

11.2 Results below the detection limit are reported as ND.

11.3 Significant figures to report are 2 for all results less than 10 ppm and 3 for all results greater than or equal to 10 ppm.

12. References

12.1 Method Source:

12.1.1 EPA-600, 1974 Edition. Method 413.1.

12.1.2 SW-846, Third Edition 9/86. Method 9070.

12.2 Deviations from Source Method

12.2.1 1 mL concentrated sulfuric acid is used to acidify samples instead of 5 mLs 1:1 hydrochloric.

12.2.2 Whatman PS/1 filter paper used instead of No. 40.

12.2.3 Flasks with residue are dried in a 60° C oven for 1 hour and desiccated until cool instead of sweeping out with vacuum and desiccating for 30 minutes.

12.2.4 Samples are not blank subtracted.

12.2.5 Extraction procedure for soils is a modified addition to the method.

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STANDARD OPERATING PROCEDURE

Subject or Title:
Cyanide, Total: Manual Micro Distillation

SOP No.:	Revision No.:	Effective Date:
LM-CAL-1053	**Original**	**June 28, 1990**

Supersedes: **None**

1. SCOPE AND APPLICATION

1.1 This method is applicable to the determination of total cyanide in drinking, surface, and saline waters, domestic and industrial wastes, sludges, and soil samples. The method detects cyanide compounds that are present, as either simple soluble salts or complex cyanides. This is used to determine values for total cyanide.

1.2 The detection limit is 0.010 mg/L for aqueous samples and 0.5 mg/kg for non-aqueous samples, using the automated colorimetric method of analysis. The range extends to 0.5 mg/L for aqueous samples and to 25 mg/kg for non-aqueous samples.

1.3 A batch of 10 samples is processed through distillation in about 3 hours.

2. SUMMARY OF METHOD

2.1 The cyanide as hydrocyanic acid (HCN) is released from acidified cyanide complexes by means of a reflux distillation operation and is absorbed in a scrubber containing sodium hydroxide (NaOH) solution. The cyanide ion in the absorbing solution is then determined colorimetrically.

3. COMMENTS

3.1 Interferences

3.1.1 Physical interferences are eliminated or reduced during the distillation process.

Prepared By:	Date:
John Barnett	**May 25, 1990**
Management Approval:	Date:
[illegible]	June 28, 1990
QA Officer Approval:	Date:
B McNeill	June 28, 1990

C4-599

3.1.2 Sulfides can seriously affect the colorimetric analysis. Sulfide presence is indicated by lead acetate paper turning blue when moistened with a drop of sample. It can be eliminated by adding powdered cadmium carbonate to approximately 75 mL of sample. Yellow cadmium sulfide will precipitate. Add the cadmium carbonate until the sample no longer darkens the lead acetate paper. Filter the solution through filter paper into a graduated cylinder. Avoid a large excess of cadmium and a long contact time in order to minimize a loss by complexation or occlusion of cyanide (CN) on the precipitated material.

3.1.3 Oxidizing agents such as chlorine can destroy cyanide. Chlorine presence is indicated by potassium iodide-starch test paper turning blue when moistened with a drop of sample. Chlorine can be eliminated by adding ascorbic acid, a few crystals at a time until a drop of sample produces no color on the indicator paper. Then add an additional amount (approximately 0.06 g) of ascorbic acid to the sample. Maintain pH of sample at >12 during treatment by adding NaOH pellets if needed.

3.1.4 Carbonate in high concentration may affect the distillation by causing excessive gassing when acid is added. The carbon dioxide (CO2) released may significantly reduce the NaOH content of the scrubber. Bubbles formed in the flow cell may interfere with the automated colorimetric analysis.

3.1.5 The presence of surfactants may cause the sample to foam during refluxing. If this occurs, the addition of an agent such as Dow Corning 544 Antifoam Agent will prevent the foam from collecting in the condenser. This can be prevented in certain samples by first adding $MgCl_2$ and then Conc. H_2SO_4. The foaming can be eliminated or minimized.

3.1.6 Nitrate and/or nitrite may react with organic compounds during distillation and ultimately form cyanide. They may be removed prior to distillation with sulfamic acid.

4. SAFETY ISSUES

4.1 Follow Enseco-Cal Lab safety regulations (e.g., gloves, glasses, and lab coats (GGC), safety sheet, and Emergency Preparedness Plan).

4.2 Cyanide is highly toxic. Handle it only in a fume hood. Clean up spills immediately. If it comes into contact with acid the extremely toxic gas hydrocyanic acid forms.

4.3 In case of skin contact with cyanide, bases, or acids, flush area with water for 15 minutes.

4.4 Work with concentrated acids only in a fume hood; avoid breathing fumes. Neutralize and clean up spills immediately. Transport concentrated acid bottles only in impact-proof containers.

4.5 Conduct distillation only in a fume hood. Build up of pressure in the apparatus can cause hot acidic solution to spray out of the thistle tube. Keep the sash of the hood down while samples boil.

5. SAMPLE COLLECTION, PRESERVATION, CONTAINERS, AND HOLDING TIMES

5.1 Liquid samples should be collected in 1000 mL plastic or glass bottles with 2 mL of 10% N NaOH as preservative.

5.2 Solid samples can be collected in an 8 oz. jar, or a metal core tube.

5.3 All water and wastewater samples must be distilled as soon as possible and analyzed within 14 days after sampling.

5.4 Contract Laboratory Program (CLP) soil/sediment samples must be distilled and analyzed within 14 days of sample receipt.

5.5 Store at 4°C.

6. APPARATUS

6.1 Microdistillation set-up.

6.2 Heat controller.

6.3 Glassware: 100-mL and 1-L volumetric flasks
100-mL graduated cylinders.

6.4 Top pan balance.

6.5 125 mL Nalgene bottles

7. REAGENTS AND STANDARDS

7.1 Sodium hydroxide solution, 1.25 N: Dissolve 50 g of NaOH in approximately 300 mL of deionized water in a 1-liter volumetric flask. Dilute to the mark.

7.2 Sulfuric acid, concentrated.

7.3 Magnesium chloride solution: Dissolve 510 g of $MgCl_2$-$6H_2O$ in approximately 500 mL of deionized water into a 1-liter volumetric flask. Dilute to the mark.

7.4 Stock cyanide standard, 1000 ppm: Dissolve 2.51 g of KCN and 2.0 g of NaOH in about 300 mL deionized water in a 1-liter volumetric flask. Dilute to mark.

7.5 Working cyanide standard, 10 ppm: Add 1.0 mL of 1000ppm stock standard to 20 mL of 1.25 N NaOH in a 100-mL volumetric flask. Dilute to the mark with deionized water.

7.6 pH test strips.

7.7 Lead acetate paper.

7.8 KI-starch test paper.

7.9 Cadmium carbonate, powdered.

7.10 Ascorbic acid crystals.

7.11 Sulfamic acid solution, 0.4N: Dissolve 40g of sulfamic acid crystals in 500 mL of deionized water in 1 liter volumetric flask. Dilute to mark.

7.12 Dow Corning 544 antifoaming agent.

8. **PROCEDURE**

8.1 For aqueous samples:

8.1.1 Measure pH of samples with pH test strips. If >12 indicate so on log sheet. If <12 notify supervisor and indicate approximate pH on log sheet.

8.1.2 If samples are known or suspected to contain sulfide, test with lead acetate paper. If positive, proceed as in 3.1.2.

8.1.3 If samples are known or suspected to contain chlorine, proceed as in 3.1.3.

8.1.4 Pour 50 grams, or portion of the sample diluted to 50 grams, in the 100 mL microdistillation flask. Add a few boiling stones.

8.1.5 If samples are known or suspected to contain nitrate and or nitrite, add 5 mL of 0.4 N sulfamic acid solution.

8.2 For solid samples:

8.2.1 Measure 2.0 g of well-mixed sample into the microdistillation flask. Add a few boiling stones. Add 50 g deionized water.

8.2.2 If samples are known or suspected to contain sulfide, test with lead acetate paper. If positive, proceed as in 3.1.2.

8.2.3 If samples are known or suspected to contain chlorine, proceed as in 3.1.3.

8.2.4 If samples are known or suspected to contain nitrate and or nitrite, add 5 mL of 0.4 N sulfamic acid solution.

8.3 Connect the flask to the condenser. Fill the absorbing tube with 20 mL of 1.25 N NaOH and connect it to the scrubber. Connect air line and be sure all connections are airtight.

8.4 Turn on condenser water at a sufficient rate to cool condenser. Flow of water must be checked when solution starts refluxing. Be sure that condenser stays cool by increasing water flow.

8.5 Start a stream of air entering the boiling flask by adjusting the vacuum to approximately two bubbles per second. The air flow must be monitored throughout the analysis. The bubble rate will not remain constant after reagents are added and while heat is applied to the flask.

8.6 Slowly and carefully add 3.0 of concentrated H_2SO_4 to the inlet tube.

8.7 Slowly add 2.0 mL of magnesium chloride solution and follow with a distilled water rinse of the inlet tube. Swirl gently.

8.8 Heat the sample to boiling and reflux for one hour after condensation appears. Turn off heat and continue bubbling for 15 minutes.

8.9 Remove the absorption tube from the distillation apparatus. Rinse twice the inside and outside of the bubbler into the tube with dHOH. Pour the contents of the tube (1.25N NaOH) into a 125 mL Nalgene bottle. Rinse twice. Bring to a total volume of 100 grams.

8.10 Label the bottle with the original sample volume or weight, the final distillate volume (100g), and the sample number. If it is a redistillation, put that on the bottle along with the date. If it is a spike, label it as such and include the spike concentration as it applies to the sample. If it won't be analyzed that day, store in a refrigerator.

8.11 After distillation wash the inlet tube, flask, and absorbing tube with soap and water. Then rinse absorbing tube with HCl rinsewater to ensure complete removal of CN.

8.11.1 The rest of the apparatus can be rinsed with deionized water between distillations.

9. QUALITY ASSURANCE/QUALITY CONTROL (QA/QC) REQUIREMENTS

9.1 Definition

9.1.1 A batch is a group of samples processed at the same time.

9.2 QC Samples for Contract Laboratory Program (CLP)

9.2.1 One preparation blank per batch or per 20 samples, whichever is more frequent. Use deionized water.

9.2.2 The ICV solution must be distilled and analyzed with samples.

9.2.3 For each matrix and each concentration level, one sample duplicate per batch or per 20 samples, whichever is more frequent. Sample may be designated by EPA.

9.2.4 For each matrix and each concentration level, one sample spike (add 0.5 mL of 10 ppm CN to 50 g of aqueous sample or 2 g of solid sample) per batch or per 20 samples, whichever is more frequent. Sample may be designated by EPA.

9.3 QC Samples for Commercial Work (Non-EPA)

9.3.1 One preparation blank per batch or per 20 samples, whichever is more frequent. Use 50 g deionized water.

9.3.2 Duplicate Control Samples (DCS) per 20 samples. Add 0.5 mL of 10 ppm CN to 50 g deionized water.

9.3.3 A Single Control Sample (SCS) per analytical lot (here defined as all samples processed in a 24-hour period). Add 0.5 mL of 10 ppm CN to 50 g deionized water.

9.3.4 One sample duplicate - only when requested by client.

9.3.5 One sample spike (add 0.5 mL of 10 ppm CN to 50 g of aqueous sample or 2 g of solid sample) - only when requested by client.

10. REFERENCES

10.1 Method Sources

10.1.1 Method 335.2, Methods for Chemical Analysis of Waters and Wastes (EPA).

10.1.2 Standard Methods for the Examination of Water and Wastewater, 16th edition.

10.1.3 Method 9010, SW-846, 2nd edition.

10.1.4 CLP-M, Method 335.2, modified for the Contract Laboratory Program.

10.2 Deviations to Method Sources and Rationale

10.2.1 This method can be used for CLP-M, Method 335.2; MCAWW 335.2; SW846 9010; and Standard Methods. The method variances are minor.

10.2.2 This method uses a micro-distillation set-up so that smaller amounts of sample are used than in the quoted methods. This has been approved by EPA as an acceptable variance.

10.2.3 SW846 Method 9010 included both total cyanide and amenable cyanide. If only total cyanide is requested by 9010, we perform only the total cyanide test and quote Method 9010.

10.2.4 To reduce sulfide interferences when a sample contains sulfide, this method uses cadmium carbonate powder to precipitate the sulfide. This is from CLP-M Method 335.2. Method 9010 uses bismuth nitrate and Method MCAWW 335.2 uses lead acetate. The cadmium carbonate is an acceptable alternative for all methods.

C4-606

Quanterra Environmental Services

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STANDARD OPERATING PROCEDURE

Subject or Title: Cyanide, Total: Automated Analysis		Page 1 of 11
SOP No.: LM-CAL-1034	Revision No.: 2.0	Effective Date: October 12, 1993
Supersedes: June 15, 1989		

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1. SCOPE AND APPLICATION

1.1 This method is applicable to the determination of:

Total cyanide
Cyanide amenable to chlorination
Weak acid dissociable cyanide

1.2 Sample preparation for the above parameters is described in separate SOP's, i.e., SOP No. LM-CAL-1099 (total), LM-CAL-1102 (amenable to chlorination), LM-CAL-1101 (weak acid dissociable).

Prepared By: Don Fredrickson	Date: October 12, 1993
Management Approval: Lori Ann Upton	Date: 11-8-94
QA Officer Approval: [illegible]	Date: 11/8/94

C4-607

1.3 Detection Limits:

Parameter	Waters	Soil/Waste
Total cyanide	0.01 mg/L	0.5 mg/kg
Amenable to chlorination	0.01 mg/L	0.5 mg/kg
Weak acid dissociable	0.01 mg/L	0.5 mg/kg

1.4 Applicable matrices

1.4.1 This method is applicable to drinking, surface, and saline waters, domestic and industrial wastes, soil and other non-aqueous matrices.

1.5 Dynamic range

1.5.1 The range extends to 0.5 mg/L (aqueous samples) and to 25 mg/kg (non-aqueous samples). Higher concentrations are analyzed by dilution of the sample.

1.6 Approximate analytical time is 1 hour for a group of 20 samples, not including time for instrument set-up.

2. SUMMARY OF METHOD

2.1 Samples for total cyanide are prepared by distillation of the cyanide from an acidic medium.

2.2 Cyanide amenable to chlorination is prepared by chlorinating an aliquot of the sample and then distilling for total cyanide. A second aliquot is distilled without chlorination for total cyanide. Cyanide amenable to chlorination is defined as the difference between the two results.

2.3 Weak acid dissociable cyanide is prepared by distillation from a slightly acidified solution.

2.4 Samples prepared by the above methods are analyzed colorimetrically on a Autoanalyzer System. The sample is buffered and reacted with Choramine-T to produce cyanogen chloride. This then reacts with a pyridine-barbituric acid reagent to produce a red colored complex.

3. COMMENTS

3.1 Interferences

3.1.1 Oxidizing agents such as chlorine will destroy cyanide.

3.1.2 Some unidentified organic compounds may oxidize or form decomposition products during chlorination, giving higher results for cyanide after chlorination than before chlorination; this gives a negative value for cyanide amenable to chlorination. Examples include samples from petroleum refineries, the steel industry, and pulp and paper processing. The weak acid dissociable method should be used for these samples.

3.1.3 Sulfide may interfere with the analysis. Sulfide is tested for and removed during sample prep.

3.1.4 Chlorine added to the sample for amenable cyanide must be completely destroyed before distillation. Otherwise, it may distill over and destroy the non-amenable cyanide.

3.1.5 Nitrate and/or nitrite may react with organic compounds during distillation to form cyanide.

3.1.6 Samples containing surfactants may foam excessively during distillation.

3.1.7 The extent to which some metal-cyanide complexes are destroyed by chlorination depends on the amount of chlorine present and the time allowed for reaction.

3.1.8 High carbonate concentrations may react violently when sulfuric acid is added to the samples during distillation. It may also interfere with the automated colorimetric analysis by forming bubbles in the flow cell.

3.1.9 Aldehydes, glucose, and other sugars may convert cyanide to cyanohydrin during distillation.

3.1.10 Amino acids may distill with the cyanide and interfere with the analysis.

3.1.11 Fatty acids may interfere by forming soaps in the absorption solution.

3.2 Helpful hints

3.2.1 Cyanide amenable to chlorination is sometimes referred to as "Free Cyanide." The weak acid dissociable method also determines "Free Cyanide" and it is important to identify the method due to the different sample preparation.

4. SAFETY ISSUES

4.1 Follow Enseco-Cal Lab safety regulations (e.g., Gloves, Glasses, and Lab Coats (GGC), Safety Sheet, and Emergency Preparedness Plan).

4.2 Cyanide is highly toxic. Prepare standards in a hood. Clean up spills immediately. Do not allow acids to come into contact with cyanide salts or concentrated solutions - extremely toxic HCN gas will form from acid and cyanide.

4.3 Pyridine and chloramine-T are eye/skin/membrane irritants. The pyridine-barbituric acid and chloramine-T reagents should be prepared in a fume hood and kept covered as much as possible.

4.4 In case of skin contact with samples, standards, or reagents, flush area with water for at least 15 minutes.

4.5 The waste from the analysis is slightly acidic. Add sodium hydroxide to the waste container to keep any unreacted cyanide from escaping into the air.

5. SAMPLE COLLECTION, PRESERVATION, CONTAINERS, AND HOLDING TIMES

5.1 Water samples are to be collected in plastic bottles and preserved with 2 ml of 10 N sodium hydroxide per liter of sample. Refrigerate at 4°C.

5.2 Soil and waste samples should be collected in wide mouth jars and refrigerated at 4°C.

5.3 The holding time for water and soil samples is 14 days from collection. For CLP samples, the holding time is 12 days from receipt of samples.

6. APPARATUS

6.1 Lachat autoanalyzer consisting of auto-sampler, pump, injection module, colorimeter, and computer.

6.2 570 nm filter and 10 mm flow cell for colorimeter.

6.3 Miscellaneous laboratory apparatus and glassware.

7. REAGENTS AND STANDARDS

7.1 Pyridine-Barbituric Acid Reagent

7.1.1 Place 15g barbituric acid in a 1000 mL volumetric flask and add about 100 mL distilled water. Add 75 mL pyridine and mix. Carefully add 15 mL concentrated hydrochloric acid and mix. Dilute to volume with distilled water and store at 4°C.

7.2 Phosphate Buffer Solution

7.2.1 Dissolve 97g potassium dihydrogen phosphate momnohydrate in distilled water and dilute to 1000 mL.

7.3 Chloramine-T

7.3.1 Dissolve 4.0g chloramine-T in distilled water and dilute to 1000 mL. Prepare fresh daily and keep covered.

7.4 Sampler Wash--Sodium Hydroxide, 1%

7.4.1 Dissolve 10g sodium hydroxide in distilled water. Cool to room temperature, dilute to 1000 mL, and mix well. Store in a plastic bottle.

7.5 Calibration Standard Stock Solution, 1000 mg/L

7.5.1 Dissolve 2.51g potassium cyanide and 2g sodium hydroxide in distilled water and dilute to 1000 mL. Standardize using 0.0192 N $AgNO_3$. The titrated value should be within 5% of expected.

7.6 10 mg/L Intermediate Standard.

7.6.1 Pipette 1.0 mL Stock Standard (1000 mg/L) into a 100 mL volumetric flask. Add 20 mL 1.25 N sodium hydroxide, dilute to volume with distilled water and mix. Prepare fresh bi-weekly.

7.7 Calibration Standards

7.7.1 Using 100 mL volumetric flasks add 20 mL 1.25 N NaOH, add amount of intermediate standard as below, and dilute to volume with distilled water. Prepare these standards biweekly.

Aliquot, mL	Final Vol., mL	Concentration, mg/L
0	100	Blank
0.1	100	0.01
0.5	100	0.05
2.0	100	0.20
4.0	100	0.40
5.0	100	0.50

7.7.2 The blank also serves as the Continuing Calibration Blank (CCB).

7.7.3 A mid-range standard (typically the 0.2 mg/L) serves as the Continuing Calibration Verification (CCV).

7.8 Initial Calibration Verification (ICV) Standard

7.8.1 A second source is used for both CLP and non-CLP samples.

8. PROCEDURE

8.1 Set up the manifold. Pump reagents through the system until a steady baseline is obtained.

8.2 While the system is pumping, prepare the calibration standards.

8.3 When a steady baseline is obtained, adjust it to the bottom of the chart with the baseline control.

8.4 Start the sampler and adjust the peaks for the high standard to full scale with the standard cal control. Readjust the baseline if necessary.

8.5 Analyze the calibration standards in order of increasing concentration, followed by the ICV, ICB, Reference Standard, prep blank, DCS/SCS, and billable samples. A CCV and a CCB must be analyzed at least every 10 samples (distilled items), and again at the end of the run.

8.6 Each QC lot should be analyzed together as a group. Dilute any samples that are greater than the high standard and reanalyze. All dilutions must be performed with 1% sodium hydroxide. The sequence of reference, samples, etc. should be recorded on the bench sheet.

8.7 At the end of the run, flush the system with distilled water and turn off the instrument.

9. QA/QC REQUIREMENTS

9.1 QC for CLP Samples (Contract Laboratory Program)

9.1.1 A prep blank must be analyzed after the ICV.

9.1.2 As a control sample, an ICV, a 0.1 mg/L standard (mid-range) must be distilled and analyzed with every analytical run. This should be run immediately after the calibration.

9.1.3 After every 10 samples and at the end of the run a CCV and a CCB must be run.

9.1.4 A duplicate and a sample spike (0.10 ppm) must be run for each batch or for every 20 samples, whichever is more frequent.

9.2 QC for Non-CLP Samples

9.2.1 An ICV is run immediately after the calibration standards. The ICV must be distilled with the samples.

9.2.2 An ICB must be run after the ICV.

9.2.3 A prep blank, distilled with the samples, must be analyzed after the Reference Standard.

9.2.4 Duplicate Control Samples (DCS) and Single Control Samples (SCS), are spiked with CN at 0.10 ppm and prepared with the samples. DCS (two samples) are required for every 20 samples. An SCS is required with every analytical lot (here defined as all samples processed in a 24-hour period).

9.2.5 Duplicates may be required for project specific QC. Duplicates are to be taken through the entire prep and analysis together.

9.2.6 Sample spikes may be required for project specific QC. Spikes are prepared with the samples.

9.2.7 A CCV and CCB are required after every 10 samples and at the end of the run.

9.3 Acceptance Criteria

9.3.1 ICB's and CCB's must be less than the reporting limit.

9.3.2 The ICV must be within control limits of 85-115% for CLP-like projects. Alternatively, non-CLP projects will be controlled using historical control limits provided in LIMS.

9.3.3 DCS/SCS recovery and Relative Percent Difference (RPD) for DCS must be within control limits established according to the Laboratory Performance QC SOP, M-EQA-002. Current control limits are in LIMS.

9.3.4 Control limits for CLP sample spike recoveries and duplicate RPD's are, with certain exceptions described in the CLP Statement of Work, 75-125% and 20% respectively.

9.3.5 The standard curve must have a correlation coefficient of 0.995 or greater.

9.3.6 CCV's must be within 10% of the expected value.

9.4 Corrective action required

9.4.1 Check the dilutions made when preparing standards and redilute if necessary.

9.4.2 Reprep samples if DCS/SCS results are out of the control limits or if the blank shows excessive contamination.

9.4.3 If the sample spike recovery is not within the control limits a post-distillation spike must be run. This is done by spiking the unspiked sample distillate at twice the sample concentration or twice the reporting limit whichever is greater.

9.4.4 For cyanide amenable to chlorination, if the chlorinated aliquot shows more cyanide than the unchlorinated aliquot, the sample must be reprepped using the Weak and Dissociable method.

10. CALCULATIONS

10.1 Cyanide amenable to chlorination:

10.1.1 Subtract the result for the chlorinated aliquot from the unchlorinated aliquot. This difference is cyanide amendable to chlorination.

11. REPORTING

11.1 Reporting units are ug/L for waters and mg/kg for nonaqueous samples.

11.2 Reporting limits are 10 ug/L for waters and 0.5 mg/kg for nonaqueous samples. Samples less than the reporting limit are reported as ND.

11.3 Significant Figures:

	0.01-0.09	0.10-0.99	1.0-9.9	10-99	100-999	1000+
Waters	0.0X	0.XX	X.X	XX	XXX	XXX0
Soils	--	0.X	X.X	XX	XXX	XXX0
Reactive	--	0.X	X.X	XX	XXX	XXX0

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12. REFERENCES

12.1 Method sources: EPA Method 335.3 and EPA Method 335.2 CLP-modified.

12.1.1 Additional information was obtained from Standards Methods and ASTM.

12.2 Deviations from source method and rationale

12.2.1 The automated method is used following manual distillation. Automatic distillation is not performed.

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QUANTERRA® STANDARD OPERATING PROCEDURE

TITLE: NONCONFORMANCE AND CORRECTIVE ACTION

(SUPERSEDES: REVISION NO. 0 12/11/95)

Prepared by: Quanterra® QA Managers

Approved by: _____
Director of Quality Assurance

Approved by: _____
Director of Environmental Health and Safety

Approved by: _____
Vice President Operation Services

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1. PURPOSE

1.1. The purpose of this document is to establish procedures for the identification and documentation of nonconformances and the corrective actions taken as a result of these events. The Quanterra® Quality Assurance Management Plan (QAMP) requires documentation of instances of deviations from established control limits, approved SOPs, or client-specified requirements. The Nonconformance Memo (NCM) described in this procedure is used to document deviations from plan specifications together with their cause and correction. The NCM maybe a written document or a defined electronic formatted document transmitted via e-mail.

1.2. This document applies to procedures, services, data, reports, or materials purchased by the laboratory or supplied by the laboratory to its clients. Nonconformances related to incoming samples may be documented separately from the NCM process by use of the Condition Upon Receipt (CUR) Form as described in a facility-specific sample receiving/log-in standard operating procedure (SOP). Regardless of which system a lab chooses to use to handle these sample receipt client-related issues, including holding time violations (HTVs), the system must include and emphasize the immediate notification of the Project Manager (PM). This will allow the PM to initiate immediate client notification and resolution of how to proceed. See Section 5, Definitions, for further clarification of application.

1.3. Identification of suspected adverse conditions (deviations) can be made by laboratory employees in the course of their work or by individuals outside Quanterra's® laboratories through review of records, audit, or proficiency testing.

2. RESPONSIBILITIES

2.1. **Laboratory Associate:** During the course of their work, all employees are responsible for identifying and documenting problems, using a Nonconformance Memo, that might affect the quality of Quanterra's® product. They should also identify or attempt to seek out possible measures to correct the problem. By signing or initialing laboratory notebooks, forms, bench sheets, data reports, and other quality-related documents, associates are verifying that procedures have been followed. Any deviation that might render a measurement suspect shall be documented.

2.2. **Group Leader/Team Leader/Supervisor** (for purposes of this SOP, the term "Supervisor" will be used): Each supervisor is responsible for the review of NCMs to ensure that problems which might affect quality are adequately described and that personnel are assigned to correct them. Supervisors sign NCMs and forward them to a project manager. Together with project managers and Quality Assurance personnel,

supervisors are responsible for determining the appropriateness of planned corrective actions.

2.3. **Project Manager (PM):** The project manager is responsible for relaying project requirements to staff so that special project requirements are understood and nonconformances recognized. The project manager communicates conformance problems to clients and documents decisions made with clients. The project manager ensures that short-term corrective actions for routine analytical QC failures are completed. An example would be making sure that repreparation and analysis of a sample was done. The project manager can and must withhold final reports to clients until corrective actions agreed to with the client have been completed.

2.4. **QA Manager:** The Quality Assurance manager or his or her designee is responsible for assigning log numbers to the NCMs as received, reviewing all prepared NCMs to ensure that actions taken are appropriate, and assisting in resolving QA/QC discrepancies. The QA staff will maintain a nonconformance tracking system to guarantee that each nonconformance is brought to closure. The system will also be used to monitor for trends that might indicate long-term quality problems. Systematic problems are investigated, NCMs issued and reviewed, and audits conducted to ensure that long-term corrective actions have been successfully completed. If review of an area reveals a significant problem with data quality, the Quality Assurance manager has the authority and responsibility to stop production in that laboratory area.

2.5. **Operations/Systems Manager** : The operations/systems manager shall ensure that corrective actions are correct and have been implemented. The operations/systems manager shall document this review and concurrence by signing the NCM as the responsible manager, if QA-required, for a specific corrective action. Along with the laboratory manager, the operations/systems manager shall emphasize the importance of quality requirements and require all employees to report any problem that might adversely affect the quality of work.

2.6. **Laboratory Manager:** The laboratory manager shall emphasize the importance of quality requirements and require all employees to report any problem that might adversely affect the quality of work. The laboratory manager is also responsible for the implementation of the NCM system in the laboratory.

2.7. **Corporate QA Director**: The Quanterra® Quality Assurance Director should be notified of any continuing non-conformances that are not properly addressed by operations or where the root cause cannot be identified.

3. SAFETY

3.1. Normal office dependent safety precautions must be taken in performing this SOP. If personnel are required to perform any portion of the procedure in laboratory areas, appropriate personal protective equipment and precautions must be utilized.

3.2. Procedures shall be carried out in a manner that protects the health and safety of all Quanterra® associates.

3.3. All work must be stopped in the event of a known or potential compromise to the health and safety of a Quanterra® associate. The situation must be reported **immediately** to a laboratory supervisor.

4. PROCEDURE

4.1. When to Initiate a Nonconformance Memo

4.1.1. Lab associates are to prepare a nonconformance memo (NCM) whenever procedures, services, data, reports, electronic disk deliverables (EDDs), or materials deviate from established specifications. All nonconformances require an NCM (see definitions of nonconformance, anomalies, and deficiencies in Section 5 and Section 1.2 for exceptions).

4.1.2. All standard operating procedures (SOPs) shall be followed. By signing or initialing laboratory notebooks, forms, data reports, and other quality-related documents, employees are verifying that the SOPs have been followed with the exceptions of the pre-approved deviations (as described in QAPjPs or Quality Assurance Summaries). Any intentional deviation from an SOP must be pre-approved by the QA manager and technical specialist. Any deviation from a SOP or client requirement not previously approved must be documented on an NCM.

4.1.3. An NCM is to be completed for each instance of a nonconformance. A single NCM can be used for a single event affecting multiple project numbers and samples, but normally a separate NCM would be prepared for different nonconformance issues. If the nonconformance involves projects for multiple project managers, then the NCM will need to be communicated to each project manager. The laboratories may utilize any communication system they feel best satisfies this required communication and maintains the integrity of the NCM process.

4.2. How to Process the NCM Form

4.2.1. Enter the information required at the top of the form (see Example Form in Attachment A; printed forms are available from QA). Names or initials must be uniquely identified by using three initials or by spelling out the last name. Use specific test codes (when available) rather than generic codes such as "VOAs" or "Anions." At a minimum enter Project ID, QuantIMS batch number or LIMS QC Lot number if appropriate, in addition to sample numbers. The laboratory generating the NCM is identified as part of the log number (e.g., AUS-97-123). Therefore, separate laboratory location identification is not required on the form.

4.2.1.1. If the NCM is for a holding time violation, the project manager ***must*** be notified immediately.

4.2.1.2. An instrument tag-out does not require the completion of the NCM if there are no client samples involved (e.g., instrument out of service to perform routine maintenance.) See section 4.2.5.

4.2.2. Check appropriate NCM boxes. If box 31—"Other"—is checked, supply a brief description of the problem. Attach further information as necessary.

4.2.3. Complete the reverse side of the form concerning root cause and corrective actions. These sections may be filled out jointly with the supervisor. Consult the project manager or the operations/systems manager and QA manager if the supervisor and associate are uncertain of corrective actions. Be objective and specific but brief. Include enough information that decisions to approve the NCM can be made easily (include pertinent QC information).

4.2.3.1. Design corrective actions to correct the immediate problem (short-term corrective actions) and to minimize the possibility of its recurrence (long-term corrective actions). Examples of corrective actions are modifications to nonconforming procedures, repair or replacement of deficient equipment, training personnel, and reanalysis of any affected samples. Additional sheets documenting corrective action may be attached to the NCM. Reference the attachment on the NCM.

4.2.3.2. Where operational corrective actions are required, they shall be supported with reference to recovery data, control charts, or other documentation.

4.2.4. If the corrective action involved retraining, the training must be documented with the signatures of the trainees and submitted to the QA staff.

4.2.5. Instrument/Equipment Nonconformance Tag

4.2.5.1. Instruments and equipment which habitually fail to meet calibration criteria or are out of service due to needed repair or other reasons must be marked with a tag indicating the nonconforming condition. (See Example in Attachment B.)

4.2.5.2. If the reason for the nonconformance tag caused sample data to be impacted, initiate an NCM and mark box 30. Identify the instrument by name and/or identification number and briefly describe the problem. This information can be recorded on page 2 under "Problem Description/Root Cause". Submit the NCM to the group supervisor.

4.2.5.3. Upon receipt of the NCM, QA assigns the NCM number and provides the number to the analytical group so that the nonconformance tracking number can be recorded in the instrument's maintenance log.

4.2.5.4. The corrective action will be to either permanently remove the instrument from service or to have the instrument repaired. If an instrument is repaired, its reliability must be demonstrated through successful recalibration before the nonconformance can be closed. The nonconformance tag remains in effect during the demonstration period. Record this information in the instrument maintenance logbook. Reference the successful calibration on the tag and return the tag to the QA staff for closure of the NCM.

4.2.6. Submit the form to the supervisor responsible for the area indicated at the top of the form.

4.3. Supervisory Review, Approval, and Signature

4.3.1. Review the information provided. Complete or amend the descriptions of root cause and corrective action. If the corrective action has not been determined, the situation must be referred to the project manager and the operations/systems manager for resolution to ensure client requirements can be satisfied. The QA staff should be consulted if there are questions as to how to proceed. If the above input was not needed, the operations/systems manager does not need to have every NCM routed to him or her. If, upon receipt and review of the NCM by the QA staff, it is felt the operations/systems manager

needs to be made aware of the issues, the QA staff will notify and document the notification of the operations/systems manager on the form.

4.3.2. If the nonconformance is hardware/equipment related, the item shall be nonconformance tagged and segregated, if possible, to ensure that it is not used until repaired.

4.3.3. The supervisor will be responsible for the completion of the corrective action unless otherwise indicated. Enter the name of the person responsible for performing the corrective action if other than the supervisor. This is Operations' commitment to rectify the problem. This will be verified by the QA staff and/or the operations/systems managers.

4.3.4. The supervisor signs and dates the "Corrective Action Approved By" line and forwards the original form to the project manager. The project manager must receive the NCM in a timely manner, generally within 48 hours. If the NCM is for a holding time violation, a project manager ***must*** be notified ***immediately***.

4.4. Project Manager Review, Client Notification, and Project Documentation

4.4.1. The project manager shall determine if client notification is required to either assist in the definition of corrective action or to notify the client of problems related to sample analysis. The project manager shall indicate on the NCM whether client notification is required or not.

4.4.2. Record the result of the client contact or leave blank if client contact was not required. Sign and date the NCM form. This must be done whether client notification is completed or not. The signature documents the project manager as having read and reviewed the NCM.

4.4.3. Document any changes to the corrective action plan, and notify the supervisor. The project manager's signature on the front of the NCM serves as documentation that the PM has read and reviewed the NCM and is aware of the corrective action plan.

4.4.4. The project manager will forward the original to the QA office within 72 hours for review and closure. The PM may keep a copy for his or her own personal use, but the original must be forwarded to the QA office.

4.4.5. If the nonconformance involves analytical work in process, the **final** report cannot be released until a project manager has signed the NCM.

4.4.6. If it is found that erroneous analytical data (e.g., from data validation comments or phone requests, etc.; inaccurate chromatograms, spectra, calculations, or final reports) have been released by the laboratory, this fact must be documented on an NCM (items 25 and 26 on NCM form) and forwarded to the QA office. Prior to making the corrections, proper documentation shall be filled out and turned in to the QA staff if corrections are needed in the database (LIMS or QuantIMS). The original data shall be marked as unusable and maintained for historical purposes. The corrective action shall include prompt client notification and issuance of amended reports.

4.4.7. After the QA staff has closed the NCM, the original of the closed NCM will be filed either in the project file or QA files per laboratory procedure.

4.5. Quality Assurance Review and Tracking

4.5.1. The QA staff shall review all NCMs for conformance with standard laboratory practices.

4.5.2. NCMs will be reviewed to ensure that the corrective action was completed.

4.5.3. A tracking system will also be used to monitor for repetitive failures that might indicate systematic problems. Tracking records would (when applicable) include:

- NCM log number
- Date initiated
- Project number
- Lab sample ID numbers
- Method or parameter
- Nonconformance description and NCM box number
- Corrective action required
- Characterization as an anomaly or deficiency
- Closure of NCM

4.5.4. The QA staff shall identify repetitive quality issues that may be systematic in nature and may require corrective actions to prevent recurrence. Recurrent technical or Information Technology problems shall be referred to the appropriate technical group for corrective actions. Correction of systematic problems could take the form of modifications of nonconforming procedures, repair or replacement of deficient equipment, training or replacement of personnel. Findings and corrective actions from these investigations or audits shall also be documented. Resolution of corrective actions for systematic

problems must be documented by the responsible laboratory area along with supporting evidence.

4.5.5. The QA staff shall conduct follow-up assessments to confirm that correction of systematic problems is successful. These assessments can be done as part of the monthly spot assessment program at the laboratory.

4.5.6. The signature of the QA manager or designee is required for corrective actions to indicate that the issue has been closed.

4.5.7. The QA office shall maintain a central file/record of all NCMs.

4.6. Externally Generated Corrective Actions

4.6.1. Reports from external quality organizations, such as audit reports, data validation comments, and reports of proficiency tests, shall be considered externally generated. The QA staff is responsible for maintaining the files of externally generated quality reports. If the report is received by an associate other than the QA manager, the report (or copy) must be forwarded to the QA Manager.

4.6.2. Preformatted NCMs are not required to be used to document audit deficiencies or performance evaluation deficiencies. A memorandum format notification by the QA staff and responsible supervisor response with corrective action can be used to document the corrective action process.

4.6.3. The QA manager or designee shall review the reports to identify any deficiencies requiring action.

4.6.4. The QA manager or designee shall work with the responsible supervisor to formulate a corrective action plan. The plan shall be implemented and evidence of corrective action supplied to the QA office.

4.6.5. The QA staff shall verify that the problem has been corrected and prepare a written response to the external organization, if required. Data validation responses shall be coordinated through a project manager for the client.

5. DEFINITIONS

5.1. ***Nonconformance:*** an unplanned deviation from an established protocol or plan. The deviation may be the result of Quanterra's® actions, then termed a **deficiency**, or the result of events beyond the control of Quanterra®, then termed an **anomaly**.

A nonconformance exists when:

5.1.1. Any laboratory QC sample (e.g., method blank, laboratory control sample, duplicate laboratory control sample, matrix spike, matrix spike duplicate, and surrogate spike) component result is outside established control limits and demonstrate a systematic deficiency. Any matrix spike or matrix spike duplicate or sample related QC outside of established control limits attributed to matrix effects must be documented, but an NCM is not required.

5.1.2. A procedure is not performed as described in the applicable SOP or QA Policy, *except* in cases where the procedure has been performed according to a client-specified document Quanterra® has agreed to follow (e.g., EPA SOWs and QAPjPs).

5.1.3. A practice or procedure is not performed as described according to a client or project document that Quanterra® has agreed to follow.

5.1.4. Purchased materials or services are determined to be defective and their use would effect data quality.

5.1.5. Holding time violations occur regardless of what or whose actions caused them.

5.1.6. A formal NCM is not required for routine instrument maintenance, malfunctions, and power failures which can be documented in instrument maintenance logbooks.

5.2. ***Corrective action:*** Measures taken to rectify conditions adverse to quality and, where possible, to prevent their reoccurrence.

5.2.1. Corrective actions may vary from reporting the data as is—with appropriate documentation—to a complete reevaluation and restructure of a system.

5.2.2. Many corrective actions can be implemented immediately; however, some will take time to implement.

6. MISCELLANEOUS

6.1. Associated Documents

6.1.1. Quanterra® Quality Assurance Management Plan (QAMP), current revision.

6.1.2. ANSI/ASME NQA-1, Chapter II, Basic Requirement 15 "Control of Nonconforming Items." Supplement 15S-1 "Supplementary Requirements for the Control of Nonconforming Items."

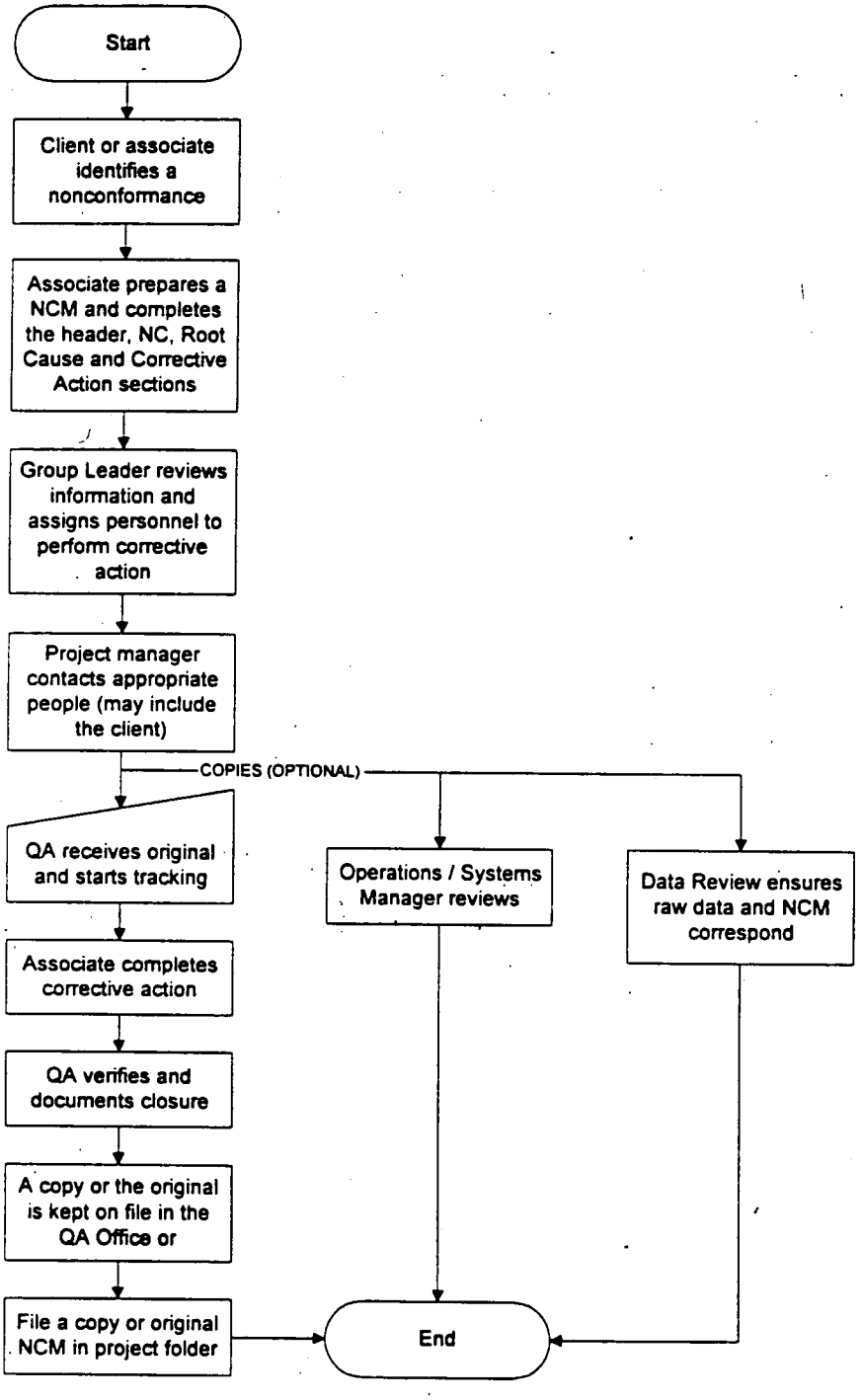
6.1.3. ANSI/ASQC Q94-1987, "Quality Management and Quality System Elements - Guidelines," Section 14.0 "Nonconformity" and Section 15.0 "Corrective Action."

6.2. Appendices

6.2.1. Attachment A: Laboratory Nonconformance Memo (NCM).

6.2.2. Attachment B: Instrument/Equipment Nonconformance Tag Form

6.3. Flow Chart for Internal NCM



ATTACHMENT A LOG #: _____

LABORATORY NONCONFORMANCE MEMO (NCM)

Quanterra® Incorporated

Project ID/Client: _____

NCM Initiated by/Date: _____ Project Manager: _____

Sample Numbers/QC batch or lot numbers _____

Tests: _____

Analytical Area (check appropriate area):

- ❑ Sample control
- ❑ Organic preparation
- ❑ Inorganic preparation
- ❑ GC
- ❑ HPLC
- ❑ GC/MS
- ❑ Wet chemistry
- ❑ Metals
- ❑ Reporting
- ❑ Data review
- ❑ Radiochemistry
- ❑ _____

Nonconformance (check appropriate area): *To be completed by analyst*

Holding Time Violations (exceeded by _____ days)

Category I: Laboratory Independent

- ❑ 1. Holding time expired in transit
- ❑ 2. Sample rec'd > 48 hrs after sampling. or ½ holding time has expired
- ❑ 3. Test added by client after expiration

Category II: Laboratory Dependent

- ❑ 4. Instrument failure
- ❑ 5. Analyst error
- ❑ 6. Log-in error
- ❑ 7. Miscommunication
- ❑ 8. Other (explanation required)_____

_____

Category III: Analysis Reruns (QA/QC)

- ❑ 9. Surrogates
- ❑ 10. Internal standards
- ❑ 11. Spike recoveries
- ❑ 12. Blank contamination

Category IV: Analysis Reruns (Confirmation)

- ❑ 13. Second column
- ❑ 14. Contamination check
- ❑ 15. Confirmation of matrix effects
- ❑ 16. Other (explanation required)_____

Category V: Analysis Reruns (Dilution)

- ❑ 17. Over calibration
- ❑ 18. Under calibration
- ❑ 19. Other (explanation required)_____

Quality Assurance/Quality Control

- ❑ 20. QC data reported outside of controls
- ❑ 21. Incorrect procedure used
- ❑ 22. SOP intentionally modified with QA and tech approval
- ❑ 23. Invalid instrument calibration
- ❑ 24. Received insufficient sample for proper analysis

Incorrect or Incomplete Client Deliverable

- ❑ 25. Hardcopy deliverable error
- ❑ 26. Electronic deliverable error

Reported Detection Limits Elevated Due to:

- ❑ 27. Sample matrix: Does not include high analyte content
- ❑ 28. Insufficient sample volume
- ❑ 29. Other (explanation required)

Miscellaneous

- ❑ 30. Instrument/equipment Tag-out
- ❑ 31. Other (explanation required)

_____

Notification (check appropriate area): ❑ **Required** ❑ **Not Required** *To be completed by project manager*

Client notified by (name and date): _____

- ❑ In writing
- ❑ By telephone
- ❑ By facsimile
- ❑ Other (explain)

Client's name and response: _____

- ❑ Process "as is"
- ❑ On hold until _____
- ❑ Re-sample
- ❑ Other (explain)

Project manager (signature and date): _____

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QUANTERRA® LABORATORY NONCONFORMANCE MEMO

LOG #: _____

Corrective Action: *To be completed and reviewed by all associates involved*

Problem Description/Root Cause Author's initials and date: _____

Corrective Actions (Short Term) Author's initials and date: _____

Corrective Actions to Prevent Reoccurrence (Long Term) _____

Corrective Action approved by (Supervisor/Group Leader) and date: _____

Additional Comments: _____

Corrective Action to be completed by (if other than Supervisor/Group Leader): _____

Date Corrective Action is to be completed: _____

Quality Assurance Review *To be completed by a QA associate*

❑ Anomaly ❑ Deficiency ❑ Notified Ops/Sys Manager (Initials) _____

❑ Further action required: _____

Further action assigned to: _____

QA signature: _____ Date: _____

Corrective Action Verification: *To be completed by a QA associate*

❑ Verification not required or requested

❑ Verified / CA completed on: _____ by _____

❑ Cannot verify (specify reason) _____

Verified by: _____ Date: _____

Nonconformance Memo Closure:

QA signature: _____ Date: _____

The Office of Quality Assurance maintains a copy or the original of this NCM indicating its final status.

L4-633

ATTACHMENT B

EXAMPLE

INSTRUMENT/EQUIPMENT NONCONFORMANCE TAG FORM

QUANTERRA®

CAUTION

DO NOT USE

NONCONFORMING ITEM

NCM NUMBER _____

AFFECTED ITEM _____

_____

_____

_____ _____
ANALYST DATE

WORK MAY NOT PROCEED ON THIS ITEM UNTIL SUCCESSFUL CALIBRATION IS DOCUMENTED.

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APPENDIX D
LABORATORY CUSTODY PROCEDURES

APPENDIX D
LABORATORY CUSTODY PROCEDURES

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OFFICIAL COPY RED STAMP INDICATES ORIGINAL

SOUTHWEST LABORATORY OF OKLAHOMA, INC./AMERICAN ANALYTICAL & TECHNICAL SERVICES

STANDARD OPERATING PROCEDURE FOR LABORATORY DOCUMENTATION OF SAMPLE CUSTODY

REV 2.1— 7/25/96

DOCUMENT CONTROL # AATSWL-GENCOC_____

CONTROLLED DOCUMENT: DO *NOT* COPY!

APPROVALS BY:

Name: Mike Butler _____ Title: Sample Receiving Manager _____

Signature: Mike Butler

Name: Chuck Hoover _____ Title: QA/QC Officer _____

Signature: Chuck Hoover

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I. CHAIN-OF-CUSTODY

A. *Summary*

1. A critical aspect of sound sample collection and analysis protocols is the maintenance of strict COC procedures. COC procedures include inventorying and documentation during sample collection, shipment, and laboratory processing. A sample is considered to be in an individual's custody if the sample is: (1) in the physical possession or view of the responsible party; (2) secured to prevent tampering; or (3) placed in a restricted area by the responsible party. The laboratory is responsible for this documentation of sample custody throughout the handling, processing, and anaslysis of samples.

II. SAMPLE LABEL

A. *Description-Field Sample Label*

1. A label is attached to all sample containers at the time of collection. The label is written in indelible ink and contains the following information:

   a. Sample number/identification

   b. Date and time collected

   c. Purpose of the sample (analyte and sample group)

   d. Source/location and location of the sample

   e. Contract task number and title of project

   f. Preservative used (if any)

   g. Collector's name or initials

2. An example of a sample label is presented in Figure 3.1-A.

III. CHAIN-OF-CUSTODY RECORD-EXTERNAL COC

A. *Description*

1. Sample custody is initiated with the detailed record keeping by the field sampling personnel. COC establishes the documentation and control necessary to identify and trace a sample from sample collection to final analysis. It includes field sample labeling to prevent mix-up, custody seals to prevent sample tampering, secure custody, and provide the recorded support information for potential litigation.

2. COC forms are used to document the integrity of all samples. To maintain a record of sample collection, transfer between personnel, shipment, and receipt by the laboratory, a COC form will be filled out for each sample set at each sampling location. The COC form will contain the following information:

a. Sample number (for each sample in shipment)

b. Collection date (for each sample shipment)

c. Time sample was obtained/or collected

d. Number of containers of each sample

e. Sample description (environmental matrix)

f. Analyses required for each sample

g. Shipment number

h. Shipping address of the laboratory

i. Date, time and method of shipment

j. Spaces to be signed as custody is transferred.

B. Procedure

1. The individual in charge of shipping samples to the laboratory is also responsible for completing the COC form. This individual will also inspect the form for completeness and accuracy. Any changes made to the COC form shall be initialed by the person making the change. An example of the COC form is presented in Figure 3.2.

IV. TRANSFER OF CUSTODY AND SHIPMENT-EXTERNAL

A. Procedure

1. Samples are to be accompanied by an approved COC record. When the possession of samples are transferred, the individual relinquishing the samples signs and records the date and time on the COC document. The individual receiving the samples repeats the procedure. This record represents the official documentation for all transference of the sample custody until the samples have arrived at the laboratory.

2. If samples are to be split with another laboratory facility or governmental agency, a separate COC record is prepared for those samples. This COC record indicates with whom the samples have been split and is appropriately signed and dated with the time of transfer of splits.

V. LABORATORY CUSTODY PROCEDURES-INTERNAL

A. Procedure

1. The Sample Control program describes the laboratory custody procedures associated with sample receipt, storage, preparation, analysis and security. Sample control is maintained at SWLO through the use of several tracking systems designed to protect sample integrity. Tracking systems include the use of laboratory COC procedures and sample analysis requests (in the form of work sheets), Internal Chain-of-Custody forms, the Laboratory Information Management

System (LIMS), Laboratory Log Books (Extraction, Digestion and Analytical Run Logs).

B. Sample Tracking

1. An overview of the sample tracking and Internal Chain of Custody, (ICOC) procedure to be employed is presented in the Figure 3.3 flow diagram. It includes the following components:

   a. Laboratory COC documentation is initiated by the SC when the sample is relinquished by the courier.

   b. After sample shipment arrival, the SC begins sample inspection and log-in. All samples are inspected: comparisons are made between the clients paperwork and that paperwork supplied by the Project Officer (i.e., Analytical Request Form, Figure 3.6). Anomalies are noted in the sample log in sheet (Figure 3.8) form and the Client/Lab Communication sheet (Figure 3.5) as the client is notified.

   c. Suspected Radioactive samples are handled and "screened" by the Radioation Safety Officer or Designee as per SOP, SWL-RO139 in the Radiation Control Area.

   d. Each sample is assigned a unique SWLO laboratory identification number which is cross-coded with the client's identification. Sample identification information is entered into the computerized laboratory data base, and the assigned number is used to track sample locations and status throughout the analytical process.

2. The following sample information is recorded into the computerized laboratory database system:

   a. Customer and project information

   b. Date of receipt

   c. Client identification

   d. Date sampled

   e. Matrix Type

   f. Number of containers

   g. Analytical requirements

   h. Other pertinent comments

   i. Radiation Screening results (RAD samples only)

3. The SC logs in samples with the tests and test code information supplied by the project officer on the Analytical Request Form (figure 3.6).

4. The field COC document is completed and copies are returned to the appropriate party(s).

5. After the sample is logged in, a LIMS generated work sheet (figure 3.7) and an Internal COC form is generated (figure 3.9).

6. The work sheet informs the analysts/departments of samples in-house.

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7. This ICOC documents the movement of the sample from storage to sample preparation and back to sample storage.

8. When the sample preparation is concluded, Sample Preparation Logs are completed and filed in the appropriate project file, along with analytical instrument logs (see figures in the analytical SOPs),

9. Suffixes are assigned to sample IDs to indicate special treatment/analysis. These suffixes include the following:

   a. MS — Matrix Spike

   b. MSD — Matrix Spike Duplicate

   c. DL — Dilution

   d. RE — Reextraction/Analysis

   e. S — Spike (Inorganics)

   f. D — Duplicate (Inorganics)

   g. L — Serial Dilution (Inorganics)

   h. A — Post Digestion Spike (Inorganics)

10. When all analyses are complete ICOC documents are filed with appropriate data in the report file.

11. A monthly archive list is generated from LIMS (see figure 3.14). Sample disposal is documented on the Sample Archive List (figure 3.14).

12. In addition to the internal and external COC documents, a computer-generated listing of the sample analysis parameters is used to control sample flow and facilitate tracking within the laboratory. Each laboratory unit is given the list of parameters and is responsible for maintaining sample integrity (holding time), fulfilling COC requirements, scheduling sample flow, and tracking sample status.

SOUTHWEST LABORATORY OF OKLAHOMA, INC.
(918)251-2858

SITE NAME	DATE
ANALYSIS	TIME
	PRESERVATIVE

SPECIALTY CLEANED CONTAINER

SWLO
(918) 251-2858

Designate comp / grab	Samplers	Analyses
Time		
mo/day/year	Preserved yes / no	
Station No.	Station Loc.	
Project Code	Remarks	
	Tag No.	

FIG. 3.1-A
Example of Sample Bottle Label (top) & Sample Tag (right).

CUSTODY SEAL
Date _____
Signature _____
Southwest Laboratory of Oklahoma

CUSTODY SEAL
Date _____
Signature
Southwest Laboratory of Oklahoma

FIG. 3.1-B Example Custody Seal for Sample Containers/Coolers

CHAIN OF CUSTODY RECORD

SOUTHWEST LABORATORY OF OKLAHOMA, INC.

SAMPLING FIRM | CLIENT CONTACT | PHONE NUMBER

P.O. OR PROPOSAL NUMBER | PROJECT NAME

SAMPLER (Signature)

ANALYTICAL TESTS REQUESTED

SAMPLE NO	DATE	TIME	COMP. GRAB	LOCATION	MATRIX	NUMBER OF CONTAINERS							REMARKS

RELINQUISHED BY (Signature)	DATE	TIME	RECEIVED BY (Signature)	RELINQUISHED BY (Signature)	DATE	TIME	RECEIVED BY (Signature)

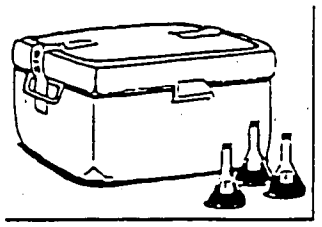
RELINQUISHED BY (Signature)	DATE	TIME	RECEIVED BY (Signature)	RELINQUISHED BY (Signature)	DATE	TIME	RECEIVED FOR LABORATORY BY (Signature)

RELINQUISHED BY (Signature)	DATE	TIME	RECEIVED BY (Signature)	REMARKS

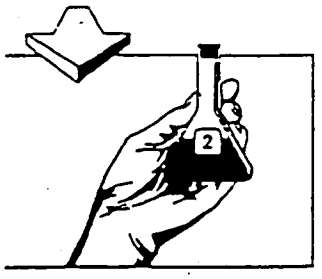
SR012-1192-031

FIG. 3.2 Example of Chain of Custody Record

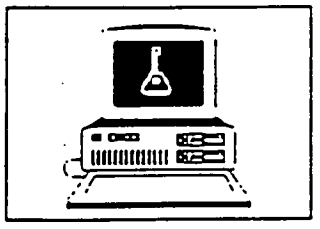
FIG. 3.3 Chain of Custody Sample Tracking Flow Diagram



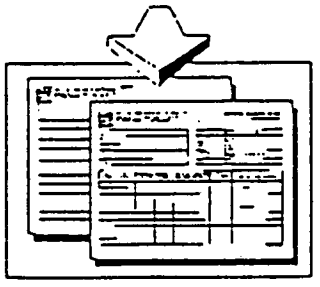
Sample Receipt



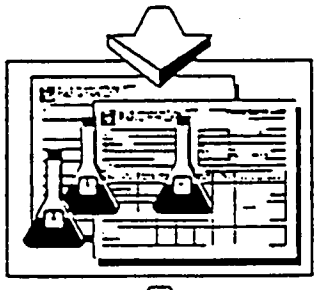
Sample Inspection
(Primary & Secondary Containers)
Radiation screening, RAD Samples Only



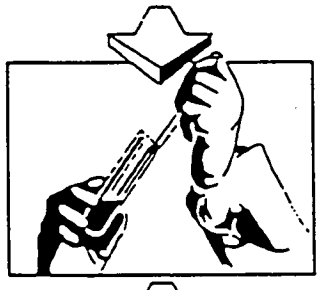
Sample Log-In
(Assigned Unique ID Number)



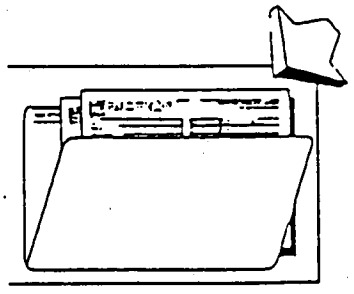
Initiate Internal Chain-of Custody Documentation



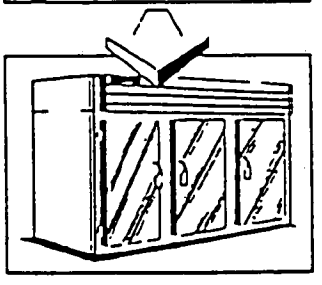
Sample Relinquished to Appropriate Laboratory Technician. (Sample maintained in locked storage)



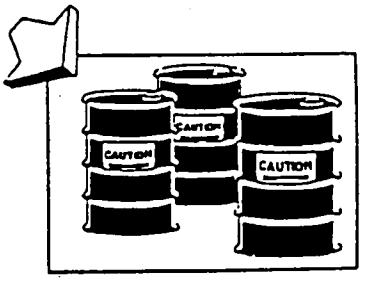
Sample Preparation and Analysis
(Sample maintained in locked storage)



Chain of Custody Documents completed and filed with appropriate data in Report File



Archive Sample/Extracts in freezer



Non-essential Sample Remains Disposed of
(Hazardous sample remains incinerated in controlled circumstances, or returned to client.)

SOUTHWEST LABORATORY OF OKLAHOMA, INC.
1700 W. Albany, Suite C • Broken Arrow, Oklahoma, 74012 • Office: 918-251-2858 • Fax 918-251-2599

CLIENT/LABORATORY COMMUNICATION SYSTEM

➤ In Reference to Case Contract/Proposal:

TELEPHONE RECORD LOG

➤ Date of Call:

➤ Client Name:

➤ Client Contact:

➤ Call Initiated By: ☐ Client ☐ Laboratory

➤ In reference to data for the following sample number(s):

➤ Summary of Questions/Issues Discussed:

➤ Summary of Resolution:

Signature: *Date:*

➤ Distribution: ☐ Lab Copy ☐ Client Copy ☐ Project Officer Copy

FIG. 3.5 Client/Laboratory Communication System

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SOUTHWEST LABORATORY OF OKLAHOMA
1700 W. Albany, Suite C • Broken Arrow, Oklahoma, 74012 • Office: 918-251-2858 • Fax 918-251-2599

ANALYTICAL REQUEST FORMS

➤ Client:	➤ Code:
➤ Client Contact:	➤ Phone No.:
➤ Project Name:	➤ P.O. #:
➤ Shipping Info.	➤ Projected Receipt Date:

➤ Date of Contact:

ANALYTICAL REQUESTS

Quantity.	Matrix.	Code	Code	Code	Code	Code	TAT	Format

➤ Special Provisions

➤ Reporting Address:

➤ Billing Address:

➤ SWLO Contact

FIG. 3.6 Sample Request Form/Analysis Request Form

SOUTHWEST LABORATORY OF OKLAHOMA, INC.
1700 W. ALBANY SUITE C
BROKEN ARROW, OK 74012-1421

Date: 10/27/93
Episode: 16075
Client: EPA
Project: 21052

SAMPLE LOG-IN RECORD

SAMPLE #	DATE IN	DESCRIPTION	SDS	MA	NC	TEST	FRI	DUE	CONTAINER	DESCRIPTION	RESULTS	ANALYST	DATE/TIME
16075.01	10/25/93	FT919 (GA/SC		W	19	GC815	4	11/14/93	MNOPQR	PEST CLP OLM016			
										EXTRACTION		MT	10/26/93
						MS515	4	11/14/93	ABCDEF	VOA-CLP OLM01.8	_____	_____	_____
						MS515	4	11/14/93	GHIJKL	BNA CLP OLM01.8			
										EXTRACTION		TB	10/25/93
16075.02	10/25/93	FT920 (CASE#210521		W	6	GC815	4	11/14/93	EF	PEST CLP OLM016			
										EXTRACTION		MT	10/26/93
						MS515	4	11/14/93	AB	VOA-CLP OLM01.8	_____	_____	_____
						MS515	4	11/14/93	CD	BNA CLP OLM01.8			
										EXTRACTION		TB	10/25/93
16075.03	10/25/93	FT921 (CASE#210521		W	6	GC815	4	11/14/93	EF	PEST CLP OLM016			
										EXTRACTION		MT	10/26/93
						MS515	4	11/14/93	AB	VOA-CLP OLM01.8	_____	_____	_____
						MS515	4	11/14/93	CD	BNA CLP OLM01.8			
										EXTRACTION		TB	10/25/93
16075.04	10/25/93	FT922 (CASE#210521		W	6	GC815	4	11/14/93	EF	PEST CLP OLM016			
										EXTRACTION		MT	10/26/93
						MS515	4	11/14/93	AB	VOA-CLP OLM01.8	_____	_____	_____
						MS515	4	11/14/93	CD	BNA CLP OLM01.8			
										EXTRACTION		TB	10/25/93

Total samples = 4

FIG. 3.7 LIMS Generated Sample Worksheet

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SAMPLE LOG-IN SHEET

Lab Name: ______ Page ___ of ___

Received By (Print Name): ______ Log-in Date: ______

Received By: (Signature): ______

Case Number: ______

Sample Delivery Group No: ______

SAS Number: ______

REMARKS:

1. Custody Seal(s) Present/Absent* Intact/Broken
2. Custody Seal Nos.: ______ ______
3. Chain-of-Custody Records Present/Absent*
4. Traffic Reports or Packing List Present/Absent*
5. Airbill Airbill/Sticker Present/Absent*
5. Airbill No: ______ ______
7. Sample Tags Present/Absent
   Sample Tag Numbers Listed/Not Listed on Chain-of-Custody
8. Sample Condition: Intact/Broken*/Leaking
9. Does information on custody records, traffic reports, and sample tags agree? Yes/No*
10. Date Received at Lab: ______
11. Time Received ______

Sample Transfer

Fraction: ______ ______
Area #: ______ ______
By: ______ ______
On: ______ ______

	CORRESPONDING			
EPA SAMPLE #	SAMPLE TAG #	ASSIGNED LAB #	REMARKS: CONDITION OF SAMPLE SHIPMENT, ETC.

* Contact SMO and attach record of resolution

Received By: ______
Date: ______

Logbook No: ______
Logbook Page No: ______

FORM DC-1

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FIG. 3.8 EPA-CLP Sample Log-In Sheet

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SOUTHWEST LABORATORY OF OKLAHOMA, INC.
1700 W. ALBANY SUITE C
BROKEN ARROW, OK 74012-1421

Date: 09/27/95
Episode: 23643
Client:
Project:

INTERNAL CHAIN-OF-CUSTODY

SAMPLE #	DATE IN	DESCRIPTION	SDG	MA	MC	TEST	PRI	DUE	CONTAINER	DESCRIPTION	LOG-OUT	ANALYST	LOG-IN
23643.01	09/27/95				0	GC817	4	10/17/95	CD	PEST CLP OLM03			
										EXTRACTION			
						MS317	4	10/17/95	AB	VOA-CLP OLM03.			
						MS317	4	10/17/95	CD	BNA CLP OLM03.			
										EXTRACTION			
23643.02	09/27/95				0	GC817	4	10/17/95	CD	PEST CLP OLM03			
										EXTRACTION			
						MS317	4	10/17/95	AB	VOA-CLP OLM03.			
						MS317	4	10/17/95	CD	BNA CLP OLM03.			
										EXTRACTION			
23643.03	09/27/95				0	GC817	4	10/17/95	GHIJ	PEST CLP OLM03			
										EXTRACTION			
						MS317	4	10/17/95	ABCDEF	VOA-CLP OLM03.			
						MS317	4	10/17/95	GHIJ	BNA CLP OLM03.			
										EXTRACTION			
23643.04	09/27/95				0	GC817	4	10/17/95	CD	PEST CLP OLM03			
										EXTRACTION			
						MS317	4	10/17/95	AB	VOA-CLP OLM03.			
						MS317	4	10/17/95	CD	BNA CLP OLM03.			
										EXTRACTION			
23643.05	09/27/95				0	MS317	4	10/17/95	AB	VOA-CLP OLM03.			

FIG. 3.9 Internal Chain of Custody

SWLO / 6ATS Sample Digestion Record

Date : 04/05/1994 Batch: 04059411 Analyst: TS
Type : MT600 ACID DIGESTION - ICP/GA Client: CLP-PRI MSH-TI Matrix: W

LAB ID	Client ID	TYP	Initial Weight	Final Weight	Color Before	Color After	Texture	pH	Comments
PBW		LB	100 ML	100 ML					
LCSW		BS	100 ML	100 ML					1.0 ML W-1,W-2,B5
17824.01	9403080000275		50 ML	50 ML	CO/CL	CO/CL		<2	
17824.01MS	9403080000275		50 ML	50 ML	CO/CL	CO/CL		<2	0.5 ML W-1,W-2,B5
17824.01MSD	9403080000275		50 ML	50 ML	CO/CL	CO/CL		<2	0.5 ML W-1,W-2,B5
17824.02	9403069A05376		100 ML	100 ML	CO/CL	CO/CL		<2	
17824.03	9403069A05373		100 ML	100 ML	CO/CL	CO/CL		<2	
17824.04	9403069A05376		100 ML	100 ML	CO/CL	CO/CL		<2	
17836.01	9403090000282		100 ML	100 ML	CO/CL	CO/CL		<2	
17836.02	9403090000283		100 ML	100 ML	CO/CL	CO/CL		<2	
17836.02MS	9403090000283		100 ML	100 ML	CO/CL	CO/CL		<2	1.0 ML W-1,W-2,B5
17836.02MSD	9403090000283		100 ML	100 ML	CO/CL	CO/CL		<2	1.0 ML W-1,W-2,B5
17836.03	9403100000284		100 ML	100 ML	CO/CL	CO/CL		<2	
17836.04	9403116A05412		100 ML	100 ML	CO/CL	CO/CL		<2	
17836.05	9403116A05413		100 ML	100 ML	CO/CL	CO/CL		<2	
17836.06	9403116A05415		100 ML	100 ML	CO/CL	CO/CL		<2	
17836.07	9403106A05394		100 ML	100 ML	CO/CL	CO/CL		<2	
17836.08	9403116A05414		100 ML	100 ML	CO/CL	CO/CL		<2	
17860.01	9403140000285		50 ML	50 ML	CO/CL	CO/CL		<2	
17860.01MS	9403140000285		50 ML	50 ML	CO/CL	CO/CL		<2	0.5 ML W-1,W-2,B5
17860.01MSD	9403140000285		50 ML	50 ML	CO/CL	CO/CL		<2	0.5 ML W-1,W-2,B5
17860.02	9403140000286		100 ML	100 ML	CO/CL	CO/CL		<2	
17860.03	9403140000287		100 ML	100 ML	CO/CL	CO/CL		<2	

FIG. 3.10 Digestion Record

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SAMPLE DISTILLATION RECORD

PAGE ____ OF BOOK ____

INORGANIC DEPARTMENT

Distillation Date _____ Distillation Batch No. _____ Analyst _____

Type of Analysis _____ Client _____ Matrix _____

Sample ID	Client ID	Initial wt/vol (g/mL)	Final Vol. (mL)	Description (Water color, Clarity, Soil color, Texture Artifacts)	pH	Sulfides Present (Y or N)	Chlorine Present (Y or N)	Comments (Solving Solution)

SOUTHWEST LABORATORY OF OKLAHOMA, INC. • 1700 W. ALBANY • BROKEN ARROW, OKLAHOMA 74012 • OFFICE (918) 251-2858 • FAX (918) 251-2599 (IN018-029301)

FIG. 3.11 Sample Distillation Record

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METHOD

	SV		OTHER
CONT. LIQ			
SEP F			
SONIC.			

☐ RUSH ☐ REEXTRACT ☐ TCLP ☐ DIOXIN

SAMPLE EXTRACTION LOG
SWOK • AATS
Southwest Laboratory of Oklahoma, Inc.
American Analytical & Technical Services, Inc.

CLIENT: _____ DATE RECEIVED: _____

CASE: _____ EXTRACT DATE: _____

SAMPLE ID (EPISODE #)	CLIENT ID	MATRIX - LEVEL	SAMPLE pH	% MOISTURE	DRY WT EQ	AMOUNT EXTRACTED	EXTRACTED VOLUME	GPC VOLUME	GPC FLUTE VOL	FLORISIL VOL CONC	FLORISIL FA VOL CONC	VOL SV - P	FA PROCESS VOLUME	SOXLET VOL	VOL SV - P	SUMA SV - P	SUMA pH CONFIRMATION	ACID pH CONFIRMATION	COMMENTS
				%	%	g - mL	mL	mL	mL	mL	mL	mL	mL		mL	mL			

INITIALS

WEIGHING:	EXTRACTION:
CLEANUP:	KD:
RI DOWDOWN	
SURROG. SPIKER:	VERIFIER
MATRIX SPIKER:	VERIFIER:
SUBMITTED BY:	REVIEWED BY:

ID NUMBERS

FLORISIL CARTRIDGE #:
GPC COLUMN #:
SURROGATES ID CONC #:
MATRIX SPIKE SOL ID/CONC #:
MISC:

COMMENTS

33050

FIG. 3.12 Sample Extraction Log

VOLATILES GC/MS RUN LOG

VOLATILES GC/MS DEPARTMENT

SWOK / AATS

TM

TUNE FILE ID: _____ METHOD FILES _____

INSTRUMENT: _____ DATE _____

CB/ID FILE ID: _____ ARCHIVE TAPE #: _____

Time	File	SWOK/AATS Sample ID	Client ID	Vol.	IS Area	Surr. Rec.	Lin. Chk.	Comments	OK/ RR

Analyst Signature

FIG. 3.13 Volatiles GC/MS Run Log

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SOUTHWEST LABORATORY OF OKLAHOMA, INC.

Page 1

SAMPLES READY TO BE ARCHIVED — PRODUCED ON 04/04/91

SAMPLE	CLIENT	DESCRIPTION	MATRIX	REPORTED
5447.08	RS&A	MW-2	W	04/03/91
5447.09	RS&A	MW-21A	W	04/03/91
5447.10	RS&A	MW-21B	W	04/03/91
5447.11	RS&A	MW 18/B	W	04/03/91
5447.12	RS&A	MW-30	W	04/03/91
5447.13	RS&A	MW-5	W	04/03/91
5447.14	RS&A	MW-3	W	04/03/91
5447.15	RS&A	MW-4	W	04/03/91
5447.16	RS&A	MW-14	W	04/03/91
5447.17	RS&A	MW-26B	W	04/03/91
5447.18	RS&A	MW-15A	W	04/03/91
5447.19	RS&A	MW-25	W	04/03/91
5447.20	RS&A	MW-34	W	04/03/91
5447.21	RS&A	TRIP BLANK	W	04/03/91
5447.22	RS&A	MW-6	W	04/03/91
5447.23	RS&A	MW-24	W	04/03/91
5447.24	RS&A	MW-23	W	04/03/91
5447.25	RS&A	MW-22A	W	04/03/91
5447.26	RS&A	MW-10B	W	04/03/91
5447.27	RS&A	MW-11B	W	04/03/91
5447.28	RS&A	MW-29	W	04/03/91
5447.29	RS&A	MW-33	W	04/03/91
5447.30	RS&A	MW-32	W	04/03/91
5447.31	RS&A	MW-30	W	04/03/91
5447.32	RS&A	EQUIP BLANK	W	04/03/91
5447.33	RS&A	MW-11A	W	04/03/91
5447.34	RS&A	MW-10T	W	04/03/91
5447.35	RS&A	MW-19	W	04/03/91
5447.36	RS&A	MW-26	W	04/03/91
5447.37	RS&A	MW-27	W	04/03/91
5447.38	RS&A	MW-28	W	04/03/91
5447.39	RS&A	TRIP BLANK	W	04/03/91
5450.01	KIMCLARK	OUTFALL001	W	04/01/91
5452.01	FHC	5-1	S	04/03/91
5452.02	FHC	5-2	S	04/03/91
5452.03	FHC	5-3	S	04/03/91
5452.04	FHC	5-4	S	04/03/91
5452.05	FHC	5-5	S	04/03/91
5452.06	FHC	6-1	S	04/03/91
5452.07	FHC	6-2	S	04/03/91
5452.08	FHC	6-3	S	04/03/91
5452.09	FHC	6-4	S	04/03/91
5452.10	FHC	6-5	S	04/03/91
5461.01	RS&ASSOC	5211.01	W	04/01/91
5466.01	ATAS	1788.01	S	04/02/91
5466.02	ATAS	1788.02	S	04/02/91
5466.03	ATAS	1788.03	S	04/02/91
5466.04	ATAS	1788.04	S	04/02/91
5467.01	ATAS	1789.16	S	04/03/91

FIG. 3.14 Sample Archive Record Sheet

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3.0 Sample Handling Practices & C-O-C

A critical aspect of sound sample collection and analysis protocols is the maintenance of strict COC procedures. COC procedures include inventorying and documentation during sample collection, shipment, and laboratory processing. A sample is considered to be in an individual's custody if the sample is: (1) in the physical possession or view of the responsible party; (2) secured to prevent tampering; or (3) placed in a restricted area by the responsible party.

3.1 CHAIN-OF-CUSTODY

Sample Label

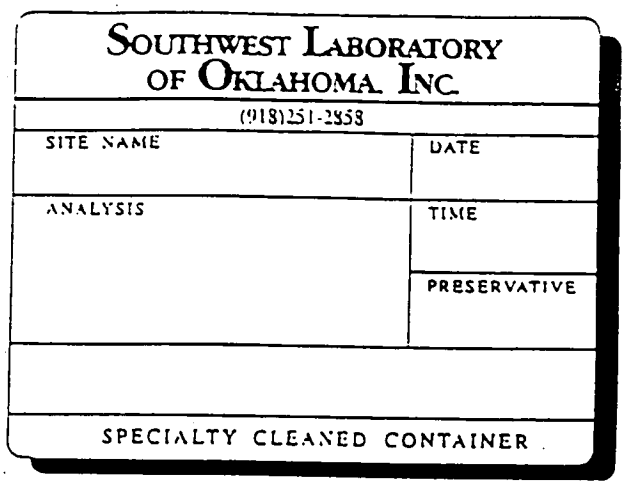
A label is attached to all sample containers at the time of collection. The label is written in indelible ink and contains the following information:

- Sample number/identification
- Date and time collected
- Purpose of the sample (analyte and sample group)
- Source/location and location of the sample
- Contract task number and title of project
- Preservative used (if any)
- Collector's name or initials

An example of a sample label is presented in Figure 3.1.

Chain-of-Custody Record

Sample custody is initiated with the detailed record keeping by the field sampling personnel. COC establishes the documentation and control necessary to identify and trace a sample from sample collection to final analysis. It includes field sample labeling to prevent mix-up, custody seals to prevent sample tampering, secure custody, and provide the recorded support information for potential litigation.



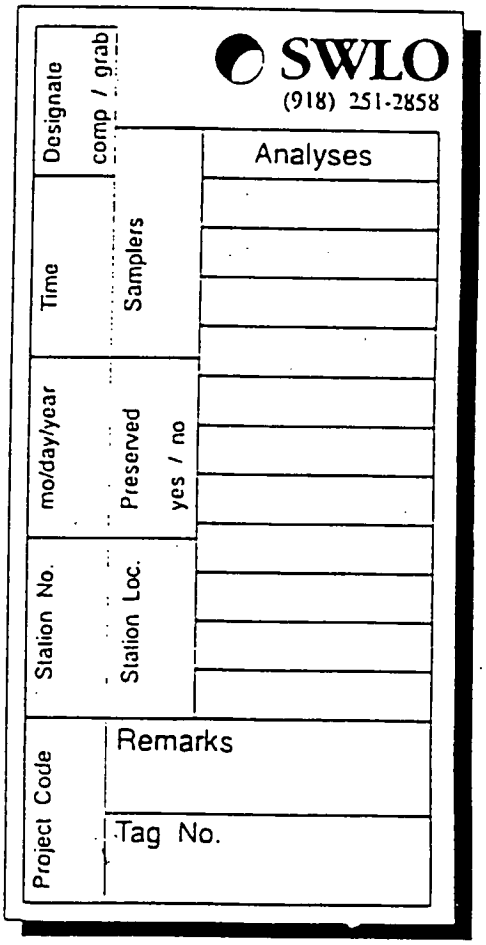


FIG.3.1 Example of Sample Bottle Label & Sample Tag.

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COC forms are used to document the integrity of all samples. To maintain a record of sample collection, transfer between personnel, shipment, and receipt by the laboratory, a COC form will be filled out for each sample set at each sampling location. The COC form will contain the following information:

- Sample number (for each sample in shipment)
- Collection date (for each sample shipment)
- Time sample was obtained/or collected
- Number of containers of each sample
- Sample description (environmental matrix)
- Analyses required for each sample
- Shipment number
- Shipping address of the laboratory
- Date, time and method of shipment
- Spaces to be signed as custody is transferred.

The individual in charge of shipping samples to the laboratory is also responsible for completing the COC form. This individual will also inspect the form for completeness and accuracy. Any changes made to the COC form shall be initialed by the person making the change. An example of the COC form is presented in Figure 3.2.

Transfer of Custody and Shipment

Samples are to be accompanied by an approved COC record. When the possession of samples is transferred, the individual relinquishing the samples signs and records the date and time on the COC document. The individual receiving the samples repeats the procedure. This record represents the official documentation for all transference of the sample custody until the samples have arrived at the laboratory.

If samples are to be split with another laboratory facility or governmental agency, a separate COC record is prepared for those samples. This COC record indicates with whom the samples have been split and is appropriately signed and dated with the time of transfer of splits.

Laboratory Custody Procedures

The Sample Control program describes the laboratory custody procedures associated with sample receipt, storage, preparation, analysis and security. Sample control is maintained at SWLO through the use of several tracking systems designed to protect sample integrity.

Laboratory COC procedures include sample inventory and record maintenance during sample collection, shipment and laboratory processing. The Sample Custodian (SC) manages and tracks the storage and distribution of samples after their arrival.

An overview of the sample tracking and COC procedure to be employed is presented in the Figure 3.3 flow diagram. It includes the following components:

1. Laboratory COC documentation is initiated by the SC when the sample is relinquished by the courier.
2. After sample shipment arrival, the SC begins sample inspection and log-in. Cooler temperatures are recorded for those clients requesting it, otherwise samples received warm will be noted on the Chain-of-custody. Samples are checked for preservation in the sample preparation area and recorded on Extraction/Digestion Logs. All samples are inspected: comparisons are made between the clients paperwork and that paperwork supplied by the Project Officer (i.e., Sample booking Information, Figure 3.5). Anomalies are noted in the COC form and the Client/Lab Communication sheet (Figure 3.6) as the client is notified.
3. Each sample is assigned a unique SWLO laboratory identification number which is cross-coded with the client's identification. Sample identification information is entered into the computerized laboratory data base, and the assigned number is used to track sample locations and status throughout the analytical process.
   The following sample information is recorded into the computerized laboratory data base system:
   - Customer and project information

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CHAIN OF CUSTODY RECORD

SOUTHWEST LABORATORY OF OKLAHOMA, INC.

SAMPLING FIRM | CLIENT CONTACT | PHONE NUMBER

P.O. NUMBER/COST NUMBER | PROJECT NAME

SAMPLER (Signature)

ANALYTICAL TESTS REQUESTED

SAMPLE NO	DATE	TIME	COMP. GRAB	LOCATION	MATRIX	NUMBER OF CONTAINERS							REMARKS

RELINQUISHED BY (Signature)	DATE	TIME	RECEIVED BY (Signature)	RELINQUISHED BY (Signature)	DATE	TIME	RECEIVED BY: (Signature)

RELINQUISHED BY (Signature)	DATE	TIME	RECEIVED BY (Signature)	RELINQUISHED BY (Signature)	DATE	TIME	RECEIVED FOR LABORATORY BY (Signature)

RELINQUISHED BY (Signature)	DATE	TIME	RECEIVED BY (Signature)	REMARKS:

SR012-1192-03

FIG.3.2 Example of Chain of Custody Record

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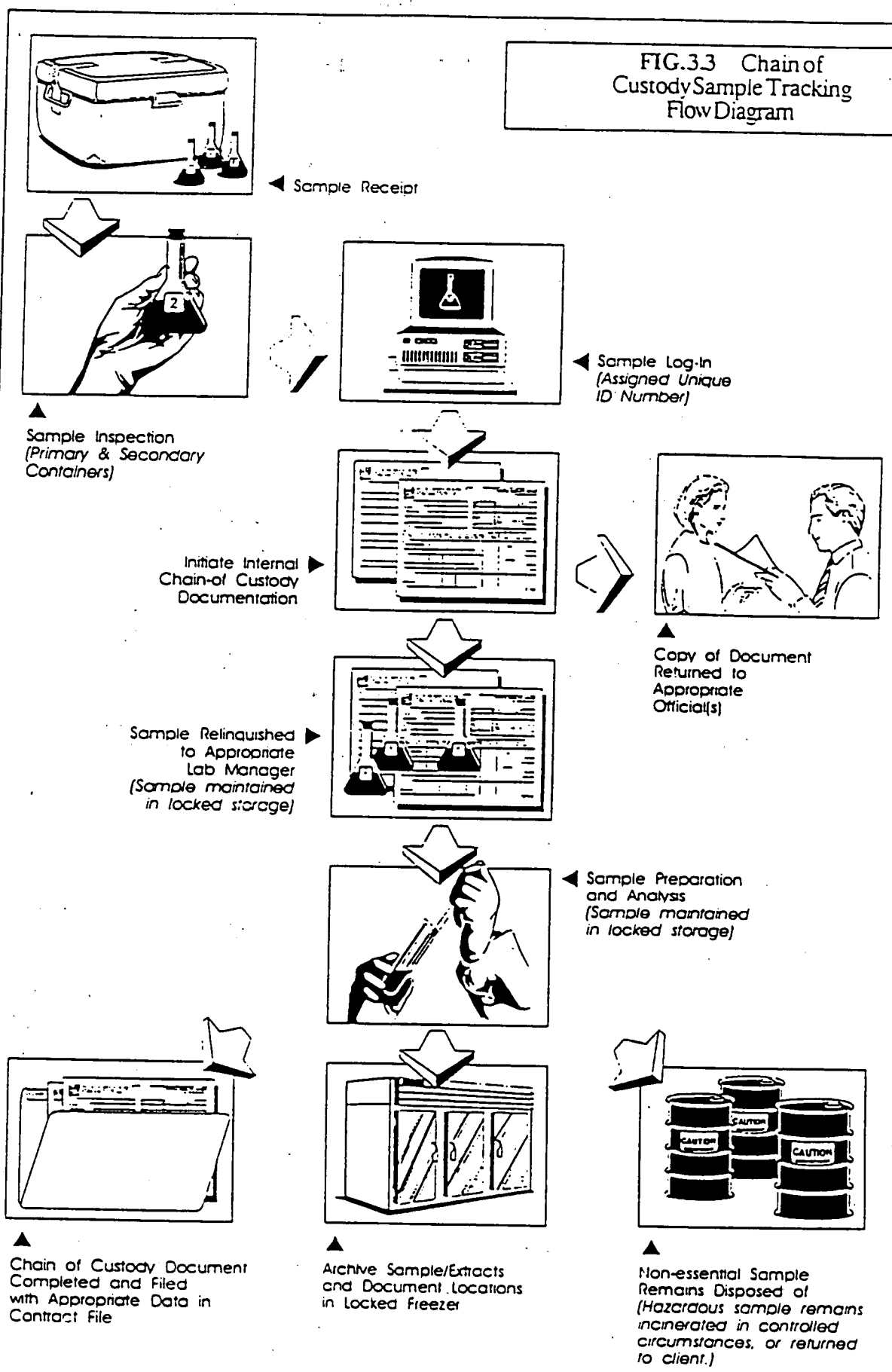
FIG.3.3 Chain of Custody Sample Tracking Flow Diagram



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SOUTHWEST LABORATORY OF OKLAHOMA, INC.

1700 W. Albany, Suite C • Broken Arrow, Oklahoma, 74012 • Office: 918-251-2858 • Fax 918-251-2599

INITIAL CONTACT FORM

➤ Client:

➤ Client Contact:

➤ Client P.O. No: | ➤ Date:

➤ Billing Instructions: | ➤ Phone No:

➤ Proposal:

➤ Analytical Requests:

➤ Method Requests

➤ Detection Limits:

➤ Additional QC:

➤ Number of Samples: | ➤ Matrix/Matrices

➤ Shipping Date: | ➤ VTSR:

➤ Turn-around Time: | ➤ Duration of Project:

➤ Bid Price: | ➤ Bid Due Date:

➤ Comments:

➤ Approximate $ Volume:

➤ Award Date: | ➤ Follow-up Date:

➤ Price: | ➤ SWL Contact

➤ Additional Information:

➤ Required Qualifications:

➤ Decision Criteria:

➤ Other Bidders:

➤ Data Package:

FIG.3.4 Project Sheet (Initial Contact Form)

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- Date of receipt
- Client identification
- Date sampled
- Matrix Type
- Number of containers
- Analytical requirements
- Other pertinent comments

4. The SC logs in samples with the tests and test code information supplied by the project officer (figure 3.5 & 3.7)

5. The field COC document is completed and copies are returned to the appropriate party(s).

6. After the sample is logged in, a LIMS generated work sheet is generated (Figure 3.9).

7. The work sheet informs the analysts/departments of samples in-house.

8. A copy of the worksheet also documents the movement of the sample from storage to sample preparation and back to sample storage. These are maintained ouside the sample storage areas where the analyst initials and dates their removal & return.

9. Based on specific contract requirements, any remaining samples are either archived in locked storage areas or disposed of properly.

In addition to the internal and external COC documents, a computer-generated listing of the sample analysis parameters is used to control sample flow and facilitate tracking within the laboratory. Each laboratory unit is given the list of parameters and is responsible for maintaining sample integrity (holding time), fulfilling COC requirements, scheduling sample flow, and tracking sample status.

3.2 PROJECT INITIALIZATION

Sample Custodian

The SC for the laboratory has duties and responsibilities that include but are not limited to:

- Receiving samples
- Inspecting sample shipping containers for presence, absence, and condition of:

  Custody seals, locks, "evidence tapes", etc.

  Container breakage and/or container integrity
- Recording the condition of both shipping containers and sample containers (bottles, jars, cans, etc.) on appropriate forms
- Verifying and recording agreement or nonagreement of information on sample documents (i.e., sample tags, COC records, traffic reports, airbills, etc.) on appropriate forms; if there is a variance, the Project Officer (PO) is notified immediately
- Signing appropriate documents shipped with samples (i.e., airbills, COC record(s), traffic reports, etc.)
- Initiating the paperwork for sample analyses on appropriate laboratory documents
- Labeling samples with laboratory sample numbers and cross-referencing laboratory numbers with client numbers and sample tags (Figure 3.8.)
- Placing samples, sample extracts, and spent samples into appropriate storage and/or secure areas
- Controlling access to samples in storage and assuring that laboratory standard operating procedures are followed during sample movement
- Monitoring sample COC in the laboratory
- Monitoring sample tags
- Monitoring storage conditions for proper sample preservation
- Returning shipping containers to sampling teams or clients.

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3.3 SAMPLE RECEIPT

Upon arrival at the laboratory, all sample shipping containers are opened and inspected. Field sampling personnel are notified on the same day of any problems concerning the samples or documents associated with the shipment. If samples arrive on a Saturday and field sampling personnel are unavailable, notification is made on the next working day.

Initial Receipt

All samples will be received by one of the Sample Custodians (SC). For weekend sample receipt, a designated person shall receive the samples and store them properly for sample log-in processing the next business day.

All samples received shall be considered to be hazardous samples, and all shipping containers shall be opened in an approved exhaust hood or an approved, well-ventilated area. All personnel associated with sample receipt are required to become familiar with safety procedures for the handling of hazardous samples.

The objective of the sample receipt procedure is to ensure that all pertinent information about the condition of the sample is recorded.

Examination of Shipping Container

The SC or will examine the shipping container and shall record the following information on the COC sample log-in sheet. Only one project or sample batch may be recorded per sheet.

- Condition of container, noting any damage, etc.
- Presence/absence of custody seals and their condition
- Labeling on shipping container

Opening Shipping Container

No shipping containers should be opened except under an approved hood or in an approved, well-ventilated area. Approved hood space and/or approved, well ventilated areas shall be determined by the Laboratory's Health and Safety Officer or the Corporate Health and Safety Officer. The SC shall note on the sample log-in form the following:

- Presence/absence of the COC record(s)
- Presence/absence of airbills and/or bills of lading documenting shipment of samples
- Necessary project and sample information enclosed with the shipment
- The temperature of the samples upon arrival is documented on the chain-of-custody form. Notification of the Project Officer and Client is made when temperature limits are exceeded.

Sample Removal

The SC shall note on the COC form the following:

- Condition of samples (intact, broken, leaking, cold or ambient, headspace in VOA vials, etc.)
- Presence/absence of sample tags

If the sample tags are present:

- Record sample tag document control numbers
- Compare sample tags with COC record(s)
- Document whether these numbers agree
- If the sample tags are not listed on the COC record, record this fact.

If an odor is noticed after opening the shipping container prior to sample removal, it must be noted on the COC.

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Sample Document Verification

The SC will compare the following documents to verify agreement among the information contained on them: (a) COC; (b) sample tags; (c) prepared project sheets; and (d) contract requirements. The SC shall document agreement among the forms and shall note any discrepancies found on the sample log-in sheet.

- If all samples recorded on the COC record were received and no problems observed, the SC will sign the COC record in the "received for laboratory by" box.

- If problems are noted, the SC will sign the COC record and note problems in the "remarks" box or reference another form that details the problems.

- If discrepancies are found, they shall be reported to the PO for clarification.

In addition, samples to be analyzed are checked for holding time requirements. When sample preservation and/or sample holding time requirements are not in accordance with the table, the appropriate PO is notified.

3.4 SAMPLE LOG-IN

Following inspection of shipping containers, records, and samples, the sample information will be added to the project information on the project sheet by the SC. Should any of the project information be incomplete or any other problems arise, the sample shall be placed on hold and the problems will be noted on the Client/Laboratory Communication System Sheet (Figure 3.6). This problem sheet shall be forwarded to the PO for resolution. The samples shall remain on hold until all information necessary for log-in is received. Information concerning the sample will be entered into the laboratory data base to maintain an official record of receipt of the sample. Corrections will be made once the PO resolves all problems.

Any samples not properly preserved will be noted on the COC and the field sampling manager will be notified immediately of the problem. He will determine the necessary corrective action.

In the event holding times may be exceeded, the PO shall contact the field sampling manager or the client immediately to correct any log-in problems. In the event holding time for analysis is exceeded, the PO or SC shall notify the field sampling manager or the client and request either resampling or instruction for proceding with the analysis. If analysis is to be performed on the original samples, the PO shall note the fact that the holding time has been exceeded, and a comment to this effect shall be added to the final report.

Samples Labeling/Identification

The SC shall assign laboratory sample numbers. These numbers will be listed on a document to cross-reference with the client number, sample tag number, and laboratory sample number.

Laboratory sample numbering is comprised of an "Episode" number (Episode meaning client batch) followed by .01 for the first sample, .02 designating second sample, etc. These numbers are sequentially generated by the SWLO "LIMS" system.

	SWLO ID	Client ID
Example:	3001.01	Sample A
	3001.02	Sample B
	3001.03	Sample C

This unique sample number shall be used for sample identification during storage, analysis and data reduction, data validation, and reporting.

3.5 SAMPLE SPLITTING

When clients supply their own containers or when bulk samples are received, the SC shall split the samples to provide sufficient aliquots for each analytical procedure that is to be performed. The following guidelines shall be used to determine the manner in which samples are split.

Water samples - Inorganic Parameters Only

The SC with the appropriate laboratory section manager determine the minimum sample quantities required for analysis. If insufficient sample exists to produce the aliquots needed, the SC shall contact the PO for a priority list of parameters, shall split the samples into proper containers, and shall complete the log-in procedure.

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CLIENT BOOKING SHEET

QA/QC DEPARTMENT

DATE OF CALL: _____ CLIENT: _____

CLIENT CONTACT: _____ PROJECT: _____

SHIP DATES: _____ REQUIRED TAT: _____

DELIVERABLES: CLP ☐ TAB—QC ☐ SPECIAL ☐ CONTRACT: _____

MS DEPARTMENT

VOA	BNA	DIOXIN	OTHER
W ☐ S ☐ O ☐	W ☐ S ☐ O ☐	W ☐ S ☐ O ☐	W ☐ S ☐ O ☐

GC DEPARTMENT

PEST/PCB	HERB	PAH	EXPLOS.	OTHER
W ☐ S ☐ O ☐	W ☐ S ☐ O ☐	W ☐ S ☐ O ☐	W ☐ S ☐ O ☐	W ☐ S ☐ O ☐

INORGANIC DEPARTMENT

ICP	FURNACE	Hg-CV	CN	WET CHEM.
W ☐ S ☐ O ☐	W ☐ S ☐ O ☐	W ☐ S ☐ O ☐	W ☐ S ☐ O ☐	W ☐ S ☐ O ☐

AATS

BTEX	TPH MOD-8015	TPH 418.1	O & G	OTHER
W ☐ S ☐ O ☐	W ☐ S ☐ O ☐	W ☐ S ☐ O ☐	W ☐ S ☐ O ☐	W ☐ S ☐ O ☐

TEST CODES/COMMENTS: _____

SOUTHWEST LABORATORY OF OKLAHOMA, INC./AMERICAN ANALYTICAL & TECHNICAL SERVICES, INC.
1700 W. ALBANY • BROKEN ARROW, OKLAHOMA 74012 • OFFICE (918) 251-2858 • FAX (918) 251-2599 [QA010-0492-01]

FIG.3.5 Client Booking Sheet

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CLIENT/LABORATORY COMMUNICATION SYSTEM

PROJECT OFFICERS DEPARTMENT

SWL____ /AATS____

TELEPHONE RECORD LOG

➤ In Reference to Case Contract/Proposal:

➤ Date of Call:

➤ Client Name:

➤ Client Contact:

➤ Call Initiated By: ☐ Client ☐ Laboratory

➤ In reference to data for the following sample number(s):

➤ Sumary of Questions/Issues Discussed:

➤ Summary of Resolution:

Signature: Date:

➤ Distribution: ☐ Lab Copy ☐ Client Copy ☐ Project Officer Copy

SOUTHWEST LABORATORY OF OKLAHOMA, INC. • AMERICAN ANALYTICAL & TECHNICAL SERVICES, INC.
1700 W. ALBANY • BROKEN ARROW, OKLAHOMA 74012 • OFFICE (918) 251-2858 • FAX (918) 251-2599 [P]001-0492-01]

FIG.3.6 Client/Laboratory Communication System

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ANALYTICAL REQUEST FORM

SAMPLE RECEIVING DEPARTMENT

➤ Client:	➤ Code:
➤ Client Contact:	➤ Phone No.:
➤ Project Name:	➤ P.O. #:
➤ Location:.	➤ Receipt Date:

➤ Deliverables:

ANALYTICAL REQUESTS

Sample ID	Quantity.	Matrix	Code	Code	Code	Code	TAT

➤ Special Provisions

➤ Reporting Address:

➤ Billing Address:

➤ SWL Contact: ➤ Date Prepared:

SOUTHWEST LABORATORY OF OKLAHOMA, INC.
1700 W. ALBANY • BROKEN ARROW, OKLAHOMA 74012 • OFFICE (918) 251-2858 • FAX (918) 251-2599 (SR002-0492-01)

FIG.3.7 Sample Request Form/Analysis Request Form

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SOUTHWEST LABORATORY OF OKLAHOMA, INC.
1700 WEST ALBANY • BROKEN ARROW, OK 74012-1421
OFFICE (918) 251-2058 • FAX (918) 251-2599

SAMPLE LOG-IN SHEET

SAMPLE RECEIVING DEPARTMENT

Date:

Custodian: (Signature)

Document Control #:

Case Number:

Airbill Number:

CUSTODY SEAL: ☐ Present ☐ Absent ☐ Intact ☐ Not Intact

CHAIN OF CUSTODY: ☐ Present ☐ Absent

SAMPLE TAGS: ☐ Present ☐ Absent

SAMPLE TAG NUMBERS: ☐ Listed ☐ Not Listed (On Chain-of-Custody)

SMO FORMS: ☐ Present ☐ Absent

Date Received	Time Received	Chain-of-Custody Record Number	SMO Sample Numbers	Corresponding Sample Tag Nos.	Corresponding Assigned Lab Nos	Does Info. on Custody Rec, Traffic Rept. and Sample Tags Agree?	REMARKS (Condition of Sample Shipment, etc.)

[SR005-0492-01]

FIG. 3.8 Sample Log-In Sheet

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SAMPLE LOG-IN SHEET

Lab Name: ______________________ Page ____ of ____

Received By (Print Name): ______________________ Log-in Date: ____________

Received By: (Signature): ______________________

Case Number: ____________

Sample Delivery Group No: ____________

SAS Number: ____________

REMARKS:

1. Custody Seal(s) Present/Absent* Intact/Broken
2. Custody Seal Nos.: ________ ________
3. Chain-of-Custody Records Present/Absent*
4. Traffic Reports or Packing List Present/Absent*
5. Airbill Airbill/Sticker Present/Absent*
6. Airbill No: ________ ________
7. Sample Tags Present/Absent
   Sample Tag Numbers Listed/Not Listed on Chain-of-Custody
8. Sample Condition: Intact/Broken*/ Leaking
9. Does information on custody records, traffic reports, and sample tags agree? Yes/No*
10. Date Received at Lab: ________
11. Time Received ________

Sample Transfer

Fraction: ________ ________

Area #: ________ ________

By: ________ ________

On: ________ ________

	CORRESPONDING		
EPA SAMPLE #	SAMPLE TAG #	ASSIGNED LAB #	REMARKS: CONDITION OF SAMPLE SHIPMENT, ETC.

* Contact SMO and attach record of resolution

Received By: ______________ Logbook No: ______________

Date: ______________ Logbook Page No: ______________

FORM DC-1

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FIG.3.8 (cont.) EPA-CLP Sample Log-In Sheet

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SOUTHWEST LABORATORY OF OKLAHOMA, INC.
1700 W. ALGONQUIN SUITE C
BROKEN ARROW, OK 74012-1421

Date: 10/25/93
Customer: 16075
Client: EPA
Project: [illegible]

SAMPLE LOG-IN RECORD

SAMPLE #	DATE IN	DESCRIPTION	SDG	MA	NC	TEST	PRI	DUE	CONTAINER	DESCRIPTION	RESULTS	ANALYST	DATE/TIME
16075.01	10/25/93	FT919 10A/SC		W	10	GC815	4	11/14/93	MNOPQR	PEST CLP OLM016			
										EXTRACTION		MT	10/26/93
						MS315	4	11/14/93	ABCDEF	VOA-CLP OLM01.2			
						MS515	4	11/14/93	GHIJKL	BNA CLP OLM01.2			
										EXTRACTION		TB	10/25/93
16075.02	10/25/93	FT920 (CASE#210321)		W	6	GC815	4	11/14/93	EF	PEST CLP OLM016			
										EXTRACTION		MT	10/26/93
						MS315	4	11/14/93	AB	VOA-CLP OLM01.3			
						MS515	4	11/14/93	CD	BNA CLP OLM01.3			
										EXTRACTION		TB	10/25/93
16075.03	10/25/93	FT921 (CASE#210321)		W	6	GC815	4	11/14/93	EF	PEST CLP OLM016			
										EXTRACTION		MT	10/26/93
						MS315	4	11/14/93	AB	VOA-CLP OLM01.3			
						MS515	4	11/14/93	CD	BNA CLP OLM01.3			
										EXTRACTION		TB	10/25/93
16075.04	10/25/93	FT922 (CASE#210321)		W	6	GC815	4	11/14/93	EF	PEST CLP OLM016			
										EXTRACTION		MT	10/26/93
						MS315	4	11/14/93	AB	VOA-CLP OLM01.3			
						MS515	4	11/14/93	CD	BNA CLP OLM01.3			
										EXTRACTION		TB	10/25/93

Total samples = 4

FIG. 3.9 Sample Work Sheet

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METHOD

CONT. EX			
SEP F			
SONIC.			

☐ RUSH ☐ REEXTRACT ☐ TCLP ☐ DIOXIN

SAMPLE EXTRACTION LOG

SWOK • AATS

CLIENT: _____ DATE RECEIVED: _____

CASE: _____ EXTRACT DATE: _____

SAMPLE ID (EMSDOE #)	CLIENT ID	Matrix - Liquid	Sample Dry	% Moisture	Wet Weight	Weight Extracted	Extracted Volume	GPC Volume	GPC Elute Vol	Florisil Vol Conc	Florisil Vol Elute	Vol. Extr.	Final Prep Volume	Vol. Extr.	Vol. Extr.	Date of Extraction	Date of Final Extraction	Comments

INITIALS

WEIGHING:	EXTRACTION
CLEANUP:	KD
BLOWDOWN:	
SURROG SPIKER	VERIFIER
MATRIX SPIKER	VERIFIER
SUBMITTED BY	REVIEWED BY

ID NUMBERS

FLORISIL CARTRIDGE #:

GPC COLUMN #:

SURROGATES ID CONC #:

MATRIX SPIKE SOL ID/CONC #(S):

SONIC.

COMMENTS

FIG. 3.10 Internal SampleTracking (Extraction Log)

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VOLATILES GC/MS RUN LOG

VOLATILES GC/MS DEPARTMENT

SWOK / AATS

TUNE FILE ID: _____ METHOD FILES _____

INSTRUMENT: _____ DATE _____

CB/ID FILE ID: _____ ARCHIVE TAPE #: _____

Time	File	SWOK/AATS Sample ID	Client ID	Vol.	IS Area	Surr. Rec.	Lin. Chk.	Comments	OK/ RR

Analyst Signature

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|VGM008-0492-01|

FIG.3.11 Internal Sample Tracking (Volatiles GC/MS Run Log)

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SW10 / 6475 Sample Digestion Record

Date : 04/05/1994 Batch: 94057411 Analyst: TS
Type : 87600 ACID DIGESTION - 125.0A Client: CLP-FRI MSW-TI Matrix: W

LAB ID	Client ID	Typ	Initial Vol	Final Vol	Color Before	Color After	Texture	pH	Comments
PBW		LB	100 ML	100 ML					
LCSW		BS	100 ML	100 ML					1.0 ML W-1.W-2.05
17824.01	9403080000275		50 ML	50 ML	CO/CL	CO/CL		<2	
17824.01MS	9403080000275		50 ML	50 ML	CO/CL	CO/CL		<2	0.5 ML W-1.W-2.05
17824.01MSD	9403080000275		50 ML	50 ML	CO/CL	CO/CL		<2	0.5 ML W-1.W-2.05
17824.02	9403096005370		100 ML	100 ML	CO/CL	CO/CL		<2	
17824.03	9403096005372		100 ML	100 ML	CO/CL	CO/CL		<2	
17824.04	9403096005374		100 ML	100 ML	CO/CL	CO/CL		2	
17836.01	9403080000282		100 ML	100 ML	CO/CL	CO/CL		<2	
17836.02	9403080000283		100 ML	100 ML	CO/CL	CO/CL		<2	
17836.02MS	9403080000283		100 ML	100 ML	CO/CL	CO/CL		<2	1.0 ML W-1.W-2.05
17836.02MSD	9403080000283		100 ML	100 ML	CO/CL	CO/CL		<2	1.0 ML W-1.W-2.05
17836.03	9403160000284		100 ML	100 ML	CO/CL	CO/CL		<2	
17836.04	9403116005412		100 ML	100 ML	CO/CL	CO/CL		<2	
17836.05	9403116005413		100 ML	100 ML	CO/CL	CO/CL		<2	
17836.06	9403116005415		100 ML	100 ML	CO/CL	CO/CL		<2	
17836.07	9403116005394		100 ML	100 ML	CO/CL	CO/CL		<2	
17836.08	9403116005414		100 ML	100 ML	CO/CL	CO/CL		<2	
17860.01	9403140000285		50 ML	50 ML	CO/CL	CO/CL		<2	
17860.01MS	9403140000285		50 ML	50 ML	CO/CL	CO/CL		<2	0.5 ML W-1.W-2.05
17860.01MSD	9403140000285		50 ML	50 ML	CO/CL	CO/CL		<2	0.5 ML W-1.W-2.05
17860.02	9403140000286		100 ML	100 ML	CO/CL	CO/CL		<2	
17860.03	9403140000287		100 ML	100 ML	CO/CL	CO/CL		2	

FIG. 3.12 Internal SampleTracking (Sample Digestion Record)

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SAMPLE DISTILLATION RECORD

PAGE ____ OF BOOK ____

INORGANIC DEPARTMENT

Distillation Date _____ Distillation Batch No. _____ Analyst _____

Type of Analysis _____ Client _____ Matrix _____

Sample ID	Client ID	Initial Vol. (g/mL)	Final Vol. (mL)	Description (Water color, Clarity, Soil color, Texture Artifacts)	pH	Sulfide Present (Y or N)	Chlorine Present (Y or N)	Comments (Spiking Solution)

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FIG. 3.13 Internal SampleTracking (Sample Distillation Record)

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SOUTHWEST LABORATORY OF OKLAHOMA, INC.

Page 1

SAMPLES READY TO BE ARCHIVED

PRODUCED ON 04/04/91

SAMPLE	CLIENT	DESCRIPTION	MATRIX	REPORTED
5447.08	RS&A	MW-7	W	04/03/91
5447.09	RS&A	MW-21A	W	04/03/91
5447.10	RS&A	MW-21B	W	04/03/91
5447.11	RS&A	MW-10/B	W	04/03/91
5447.12	RS&A	MW-3B	W	04/03/91
5447.13	RS&A	MW-5	W	04/03/91
5447.14	RS&A	MW-3	W	04/03/91
5447.15	RS&A	MW-4	W	04/03/91
5447.16	RS&A	MW-14	W	04/03/91
5447.17	RS&A	MW-26B	W	04/03/91
5447.18	RS&A	MW-15A	W	04/03/91
5447.19	RS&A	MW-25	W	04/03/91
5447.20	RS&A	MW-34	W	04/03/91
5447.21	RS&A	TRIP BLANK	W	04/03/91
5447.22	RS&A	MW-6	W	04/03/91
5447.23	RS&A	MW-24	W	04/03/91
5447.24	RS&A	MW-23	W	04/03/91
5447.25	RS&A	MW-22A	W	04/03/91
5447.26	RS&A	MW-10B	W	04/03/91
5447.27	RS&A	MW-11B	W	04/03/91
5447.28	RS&A	MW-29	W	04/03/91
5447.29	RS&A	MW-33	W	04/03/91
5447.30	RS&A	MW-32	W	04/03/91
5447.31	RS&A	MW-30	W	04/03/91
5447.32	RS&A	EQUIP BLANK	W	04/03/91
5447.33	RS&A	MW-11A	W	04/03/91
5447.34	RS&A	MW-10T	W	04/03/91
5447.35	RS&A	MW-19	W	04/03/91
5447.36	RS&A	MW-26	W	04/03/91
5447.37	RS&A	MW-27	W	04/03/91
5447.38	RS&A	MW-20	W	04/03/91
5447.39	RS&A	TRIP BLANK	W	04/03/91
5450.01	KIMCLARK	OUTFALL001	W	04/01/91
5452.01	FHC	5-1	S	04/03/91
5452.02	FHC	5-2	S	04/03/91
5452.03	FHC	5-3	S	04/03/91
5452.04	FHC	5-4	S	04/03/91
5452.05	FHC	5-5	S	04/03/91
5452.06	FHC	6-1	S	04/03/91
5452.07	FHC	6-2	S	04/03/91
5452.08	FHC	6-3	S	04/03/91
5452.09	FHC	6-4	S	04/03/91
5452.10	FHC	6-5	S	04/03/91
5461.01	RS&ASSOC	5211.01	W	04/01/91
5466.01	ATAS	1788.01	S	04/02/91
5466.02	ATAS	1788.02	S	04/02/91
5466.03	ATAS	1788.03	S	04/02/91
5466.04	ATAS	1788.04	S	04/02/91
5467.01	ATAS	1789.16	S	04/03/91

FIG. 3.14 Sample Archive Record Sheet

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Water Samples - Organic and Inorganic Parameters

When bulk samples arrive for both inorganic and organic analysis, the SC or personnel doing volatile analyses shall split out a portion for any required volatile analysis and transfer the samples to the Process Laboratory where the remainder of the organic split shall be made by the Organic Extraction Group Leader. The sample will be returned to the SC to complete samples splitting for the inorganic portion, as described above.

Sediments/Soil Samples

Every effort will be made to acquire duplicate aliquots of these matrices. The sample will be made homogeneous after any portion required for volatile analysis has been removed by one or all of the following procedures:

- Stirring
- Air drying and grinding
- Particle separation
- Quartering

The Organic Extraction Group Leader shall obtain the aliquot for organic analysis after any portion required for volatile analysis has been removed. The remainder shall be given to the Inorganic Laboratory Supervisor for further splitting. If insufficient material exists to provide minimum quantities, the PO shall provide a priority list of parameters.

3.6 SAMPLE SECURITY

Southwest Laboratory is a secured facility. Samples are maintained in storage areas except during laboratory analysis. The work sheet informs the analyst of what samples are needed for sample preparation and/or analysis (Figure 3.8). Internal custody of the sample is documented in the Sample Digestion/Extraction Logs, or in the case of Volatiles, the GC/MS Run Log (see Figure 3.9 through Figure 3.12.) All laboratory personnel who receive samples are responsible for the care and custody of samples from the time each sample is received until samples (or appropriate documentation as to disposition of the empty containers) are returned to storage. All subsets (extraction, digestates, etc.) of the samples shall be kept in locked storage which is controlled by the appropriate laboratory manager.

3.7 SAMPLE STORAGE

Once the samples have been logged into the computer system, the SC shall be responsible for the following:

Sample Storage

1. Samples and extracts shall be stored in designated areas.
2. Samples shall be removed from the shipping container and stored in their original containers unless damaged.
3. Damaged samples are to be documented and PO or the client is contacted immediately to notify him of the damaged samples.
4. Storage area temperatures are to be monitored daily. (see Figure 3.16).
5. Samples removed from storage will be documented. All transfers of samples are documented in the internal COC.
6. VOA samples will be stored separately from other samples.
7. Standards are not stored with samples.

3.8 LABORATORY DOCUMENTATION

Filling of Log-in Information

Work sheets and internal chain-of-custody are sent to the various departments (i.e. inorganics, GC/MS, GC, etc.)

The SC or a designee will log in samples. The date on the receipt form will be the date of sample receipt in the laboratory. The time, date of collection, and recipient

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name will be noted in the remarks column of a COC sample log-in sheet (Figure 3.8); and the original receipt documentation will be attached to the COC record.

Sample Log-in Document Storage

There will be two repositories for documents associated with a project. The first repository is the project file. This file will contain the following documents:

- Contracts, purchase orders, task order, and/or other work authorization
- Original project sheet
- Computer-generated project sheet
- Project modification forms

The second repository for related documentation is the analytical data file, which will contain a copy of the final project report/QC report and any other documents related to the project analysis (i.e. 1) signed airbill; 2) signed chain-of-custodies; 3) work sheet; 4) sample tags; 5) traffic reports; 6) bench sheets; 7) raw data).

Corrections to Documentation

When it becomes necessary to make corrections to any form of documentation (e.g., sample tags, COC forms, daily logbooks), the obsolete information is crossed out with a single line and the changes are made and initialed by the person making the change.

3.9 SAMPLE DISPOSAL

Sample Disposal

1. Upon completion of the analysis, an archive li[illegible] generated each month (Figure 3.14).
2. When sample analysis and all QC checks have been completed and a final report has been issued, the unused sample portion shall be archived for a period of not less than 60 days after the report has been issued.
3. The SC shall be responsible for returning all unused bottles, shipping containers, packing materials, blue ice packs, and if requested, the unused sample portions to the client.
4. Any sample remains shall be properly disposed of after a 120-day period, unless further instructions are received from the PO or client. Sample disposal shall be documented on the archive list (Figure 3.14.)
5. For more information (Details on Disposal of Samples & Waste) see Appendix B.

FIG.3.15 Example Custody Seal for Sample Containers/Coolers

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FREEZER TEMPERATURE RECORD

QA/QC DEPARTMENT

CONTROL LIMITS: -2°C to -10°C/-4°F to 14°F

DATE	TIME	ANALYST	TEMP °C / °F	WITHIN LIMITS?	CORRECTIVE ACTION* (If Needed)
1				Yes No	
2				Yes No	
3				Yes No	
4				Yes No	
5				Yes No	
6				Yes No	
7				Yes No	
8				Yes No	
9				Yes No	
10				Yes No	
11				Yes No	
12				Yes No	
13				Yes No	
14				Yes No	
15				Yes No	
16				Yes No	
17				Yes No	
18				Yes No	
19				Yes No	
20				Yes No	
21				Yes No	
22				Yes No	
23				Yes No	
24				Yes No	
25				Yes No	
26				Yes No	
27				Yes No	
28				Yes No	
29				Yes No	
30				Yes No	
31				Yes No	

* REPAIR SERVICE: Tobey's Heating & A/C • Ken Tobey • 664-1166 • OK Refrigeration Co. • Gary Stout • 582-5214

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(OA03-0992-02)

Figure 3.16 Freezer Temperature Record

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Environmental Services

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SOP No. SAC-QA-0003
Revision No. 5
Revision Date: 7/3/96

OPERATION-SPECIFIC STANDARD OPERATING PROCEDURE

TITLE: SAMPLE RECEIPT AND PROCEDURES (SUPERSEDES: LP-CAL-0007, REVISION 4)

Prepared by: Peter Isaeff

Reviewed by: _____
Technical Specialist, Roger Freize

Approved by: _____
Quality Assurance Manager, Gail Cho Celaschi

Approved by: _____
Environmental Health and Safety Coordinator, Cheryl Ann Stith

Approved by: _____
Laboratory Director, Bruce Warden

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1. PURPOSE

1.1. This SOP describes the procedures for laboratory chain-of-custody, including receipt and acceptance of sample shipments, storage requirements, generation of computer records, and corrective actions for sample receipt anomalies.

2. RESPONSIBILITIES

2.1. Sample Administrator

2.1.1. Sample Administrators are responsible for following the procedures and policies set forth in this SOP.

2.1.2. Primary responsibilities include: sample receipt and acceptance, documentation of sample identity, preservation of custody chains, annotation of discrepancies and obvious anomalies, maintenance of proper sample storage conditions, and creation of computer records which integrate laboratory expectations with specifications from shipment documentation.

2.2. Project Manager (PM)

2.2.1. The PM is responsible for verifying the accuracy of all computer records entered at project creation.

2.2.2. The PM is responsible for providing essential information in order for acceptance to proceed.

2.2.3. The PM is responsible for resolving discrepancies with client, Sample Management Office (EPA), or Contracting Officer in order for work to proceed.

2.3. Laboratory Analysts

2.3.1. Analysts are responsible for ensuring the integrity of the internal chain of custody of each sample they have signed out of or returned to sample control. Refer to Section 4.12.

3. SAFETY

3.1. Procedures shall be carried out in a manner that protects the health and safety of all Quanterra associates.

3.2. Eye protection that satisfied ANSI Z87.1 (as per the Chemical Hygiene Plan), laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

3.3. The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the Material Safety Data Sheets (MSDS) maintained in the laboratory.

3.4. Exposure to samples must be maintained **as low as reasonably achievable**, therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation.

3.5. All subsampling activities will be conducted in a fume hood with the sash closed as far as the operation will permit.

3.6. All work must be stopped in the event of a known or potential compromise to the health and safety of a Quanterra associate. The situation must be reported **immediately** to a laboratory supervisor.

3.7. Safety policies pertain to all Sample Administration visitors, including employees of clients who deliver samples.

3.8. All samples are to be considered hazardous and must be handled according to safety policies and any pertinent SOP.

4. PROCEDURE

4.1. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

4.2. Receiving Shipment

4.2.1. Verify accuracy of each container's delivery address.

4.2.2. Sign bills of lading, if present, including date, time, and custody seal condition. In the event of multiple package deliveries, the receipt time is the carrier's time of arrival, not the time when each package is handled.

4.2.3. Record each project received in the Project Receipt Logbook (PRL). A single shipping container may be listed as one or several projects. Record the client and/or shipper name(s), receipt date and time. If the project number is generated at this time, record the number in the far right column. If a single shipment is subdivided into multiple projects at a later time, all project numbers may be listed on the original logbook entry. The PRL (Example 1) is used to track the flow of incoming work and as a cross reference between shipments and computer records.

4.2.4. All packages received must be entered in the PRL, whether samples are present or not. If the shipment is not a project, enter a comment explaining why no other receipt records were generated (e.g. "empty returned cooler", "technical components", "standards", or "personal").

4.2.5. Initiate a Project Receipt Checklist (PRC) for each sample shipment (Example 2). Each person who completes an entry must also enter initials and date in the corresponding columns. For not applicable items, enter "N/A", with initials and date. The initiator must complete entries A, B, and F-K, unless responsibility for the shipment is immediately passed to another technician.

- Item A: Enter client/shipper name, as entered in PRL.
- Item B: Enter PRL number. PRL number consists of the logbook page number and line number (e.g. 1-1, 1-2, etc.).
- Item F: Enter date shipment is received.
- Item G: Enter time shipment is received.

- Item H: Enter carrier (method of delivery). "Over the counter" indicates a delivery made directly to the laboratory by a client.

- Item I: Enter condition of custody seal (if present). Enter pre-printed number of custody seal or "N/A". Custody seals are defined by the presence of the shipper's signature accompanied with date/time. Note the presence of custody tape (which will have no space for entries and crumbles when removed).

- Item J: Record ownership/disposition of shipping container. Non-company containers must be returned to client if possible.

4.3. Opening Shipment - Prioritizing Workload

4.3.1. Following safety policies, open the shipping container, remove paper work, and check interior condition. Shipments accompanied by MSDS documentation, shipments of concentrated product, or exhibiting strong/noxious odors must be treated with extra care. Open any suspect shipments only after placing the container in hood H1.

NOTE: If a shipment contains broken or leaking samples, place the container in hood H1 and notify the project manager. Proceed with disposal of broken samples in accordance with "Response to Samples Broken Upon Arrival", SOP LP-CAL-0005.

4.3.2. Immediately after opening, ascertain if ice or artificial coolants are present. Measure temperature and record results under Item: K of the PRC.

4.3.3. Note pre-printed Chain-of-Custody (COC) number if a COC is present and possesses a sequential document number.

4.3.4. Use the infra-red thermometer to determine temperature. Measure by directing the thermometer at sample containers. Do not measure the temperature of any cooling agents (ice, dry ice, artificial ice). Do not allow packing to interfere. One layer of plastic, such as a ziplock bag may cause a discrepancy of two or three degrees. [The IR thermometer is calibrated quarterly against a "NIST" thermometer. All filament thermometers are calibrated annually. The calibration documentation resides with the Quality

Assurance Department.] If the COC lists a temperature blank, locate the blank and measure the temperature.

4.3.5. Record result under Item K on the PRC.

4.3.5.1. An anomaly must be entered if the temperature reading is below 2°C or above 6°C. Use one of the anomaly selections available with the LABEL program (Example 6). This notifies the project manager and provides a hard copy to include with the project folder. Some sample matrices do not require cooling during transit. Anomalies need not be filed for these samples and "N/A" may be entered for Item: K. Situations where acceptable temperature range exceedence may be expected include, but are not limited to:

- samples delivered within six hours of close of daily sampling event; an anomaly must be filed in this case
- paperboard and dry pulp samples;
- dry incinerator ashes;
- filters, PUF and XAD cartridges;
- dry product samples.

4.3.6. If the shipment shows any sign of matrix degradation, an anomaly must be filed.

4.3.7. Read accompanying documentation. If documentation is absent and the shipment arrival was unexpected, contact the lead project manager for assistance.

4.3.8. Examine the analyses requested and the sampling dates/times. Be aware of any notation for expected Turn-Around-Time (TAT) or a due date which is less than 14 days.

4.3.9. Most aqueous organic analyses require extraction within seven days of sampling. If three days or less are remaining, consider the project to be a priority and file in an orange folder. [Methods 8280 and 8290 (dioxin/furan analyses) are an exception, requiring extraction within thirty days.]

4.3.10. Any analysis which has a standard holding time of forty-eight hours or less is considered a "Short-Holding Time" (SHT) analysis (Example 3). These are reported to the General Chemistry Department immediately. [Reporting SHT analyses does not require complete entry of project records on the computer.]

4.3.11. Any requested TAT of fourteen days or less is also considered a priority.

4.3.12. Prioritize the shipment according to the TAT, while ensuring extraction holding times have not been exceeded. "RUSH" projects are placed in a red folder, SHT analyses are placed in orange folders, while all other projects are placed in manila folders. If a project with a short TAT also includes SHT analyses, file in a red folder.

4.4. Sample Processing

Select the project with the highest priority:

1. orange folder;
2. red folder;
3. manila folder.

4.4.1. In circumstances where chemists have arranged to extract samples immediately, project TAT is forty-eight hours or less, or SHT analyses will be handled at an arranged time (so that holding times do not expire), some flexibility is allowed in processing the workload.

4.4.2. Read shipment documentation. Ideally, samples are to have possession documented on a Chain-of-Custody form. Chains-of-Custody will identify samples individually by alpha-numeric designators, list sampling dates/times for each sample, requested analyses and document possession. Signatures of possession qualify samples as having been "received under Chain-of-Custody". A Letter-of-Transmittal is also accepted as definitive documentation. Other forms of documentation include Request for Analysis, Shipping Order, Purchase Order, and various computer listings of sample information. If no documentation which lists sample identifications exists, complete a COC when accepting the samples.

4.4.3. Remove the samples from the shipping container. If the temperature has not been measured, do so and record under Item: K, as in Section 4.3.4. If a

temperature blank is discovered during unpacking, measure it and record under Item K also.

4.4.4. Align containers from left to right in rows, with additional containers for any sample placed behind the first in columns.

4.4.5. Check the samples against the COC for accuracy (e.g., sample ID, collection date/time, etc.). If the samples have been received in good condition, document this on the COC. All discrepancies must be noted as well, including the lack of relinquishing signature of the shipper.

NOTE: Good condition is loosely defined as all containers intact with no obvious discrepancies present. The shipment is estimated at this time as viable; what is being requested coincides with what has been received. Temperature exceedences and minor discrepancies become issues when so specified by contract or client instruction. The presence of bubbles in volatile containers is documented. Head space present in organic lead samples originating from U.S.Navy Program work is anomalous and must be documented on custody chains, the PRC (Item: Q), and by using the Anomaly option of the LABEL program.

4.4.6. Complete the bottle inventory on the back of the project receipt checklist (Example 5).

4.4.7. Compare the containers and their state of preservation with the list of analyses requested, if present on receipt documents. Although some exceptions are permitted regarding particular clients, container types must compare to the Quanterra QA (Quality Assurance) Plan. Discrepancies of this nature must also be annotated on the COC and an anomaly filed.

4.4.8. Assume custody of the samples by signing and dating the COC in the section marked "Received for Lab By" or "Received By". For documentation that is not very specific, specify what was received. [Example: Two solid product samples received in good condition.] Complete Item: P and/or Q.

4.4.9. Affix a color dot to the lid of each sample container. The color corresponds to the receipt week on the disposal calendar. The dot color aids in sample rotation.

4.4.10. For all work sampled at or concerning government property, or as specified in client QAPjPs, the pH of the preserved aqueous samples must be checked and recorded except for the VOA vials. The pH is noted as <2 or >12 with the preservative type following. [Example: pH<2 H2SO4).

4.4.10.1. Ascertain the pH by inverting the sample container three times, removing the cap and inserting a clean pipette into the sample container, removing a small amount. Release a droplet of the aliquot on a fresh piece of pH indicator paper. Compare against the pH Color Grid Table. Do not reuse the pipet. Use a clean pipet for each container. Recheck readings that indicate samples were unpreserved when they should have been. Note discrepancies on the COC. Notify the Project Manager and file an anomaly for the concerned sample.

4.4.10.2. Transport samples to the General Chemistry or Metals Departments when sample preservation is necessary as a corrective action. Complete Item: L

4.4.11. Referring to Section 4.7, obtain a project number.

4.4.12. Generate labels for the sample containers on the computer terminal next to the label printer. The user name and password is "labels". The LABELS program will automatically come on the screen (Example 6).

4.4.13. Some usage is necessary to understand LABELS. One must have pre-selected the storage unit location, and know the Project Manager and appropriate disposal codes before use.

4.4.14. To designate a storage location, use the following guide:

- VOA(V) - volatile containers awaiting transfer to the VOA group refrigerators and organic lead samples received under the U.S.Navy Program.
- W1 through W3 - environmental soil samples;
- W4 through W10, W17 through W26 - environmental aqueous samples;

- W11 through W13 - Advanced Tech aqueous and solid samples;
- W14 - incoming/outgoing shipments;
- W15 - product/known hazard/air toxics/oversized containers (Specialty) samples;
- W16 - extended storage, disposal codes exceeding thirty day retention time;
- F2 - any samples requiring freezing, including plant/animal tissues;
- R2 - any samples where secondary containment in ziplock bags is deemed necessary due to smells, spillage, degradation of container exterior;
- C1 - dry pulp/paper samples;
- EPA1 - soil samples received from the Environmental Protection Agency or clients requiring dioxin work under SOW DFLM01.1.
- WR1/WF1 - refrigerator/freezer storage dedicated to Explosives & Specialty Chemicals projects.
- MA - room temperature storage for aqueous samples (excluding Hg, CRVI, Organic), located in Metals Department and dedicated to U.S.Navy Program projects.

NOTE: Location selections (specify shelf by letter, Example: W4A).

4.4.15. Label sample containers. Be certain the labels adhere to containers. Attempt to leave all client label information exposed. However, when impossible to do so, affix label so that at least the client ID, sampling date/time, and preservative are showing. Preprinted laboratory names may be covered with no consequence. If label adhesive is insufficient, use cellophane tape to secure label. Place sample in ziplock bag when storage location is R2.

4.4.16. Initial/date the PRC (Item: M).

NOTE: U.S.Navy Program requires that each bottle for each sample be designated with an alpha character. The first container for the -001 will be "a", the second "b", etc. Sample -002 will repeat with "a","b",etc.

4.4.17. Project/sample labeling may be peer reviewed whenever the complexity of the project warrants a second check to ensure accuracy. A peer compares the order of the samples to the documentation and ensures that all containers of a sample have the same sample number and are sequential. The reviewer then initials/dates the PRC (Item: N). If no review is performed, the labeler completes line N.

4.4.17.1.Circumstances where a review is warranted include:

a) a large number of samples are present;
b) many containers per sample are present;
c) VOA containers are present;
d) client identifications are illegible or confusing:
e) a review is requested by a Quanterra employee

4.4.18. Anomalies can be filed using the LABELS program. Shipment discrepancies as well as temperature exceedences must be conveyed to Project Managers promptly. Holding-times that expire prior to receipt or result due to half the holding-time expiring prior to receipt require documentation using the ANOMALY Program (Example 7). Holding-time violations can only be entered after LIMS entry is complete.

4.5. Shipping Container Return

4.5.1. Containers in which sample containers have broken or in which sample volume has mixed with packing material or melted ice must be handled according to LP-CAL-0005, Response to Samples Broken Upon Arrival.

4.5.2. Except when discarded, shipping containers are cleaned in Sample Administration and either taken to Bottle Prep or sent to the client. Containers belonging to Quanterra Incorporated will be marked in felt pen with an easily identified tracking number. If the container belongs to a client, be certain the return address is recorded before stripping the container of used tape and labels.

4.5.3. Strip the container of tape and shipping labels. Older containers may be covered with aged tape and labels that are not easily removed. Do not spend excessive amounts of time with these. All hazardous materials labeling must be removed or defaced in some way. Courier/shipping companies consider intact hazard labels valid, and will refuse to carry containers displaying hazard labels unless accompanied by proper documentation.

4.5.4. Blue foam inserts can be placed in a hood or on a counter to dry when received wet. They may also be stored in the warehouse outside Bottle Prep if necessary. If the inserts are dry they may be returned to Bottle Prep in the container after it has been cleaned.

4.5.5. Dry the interior of the container if wet, replace packing and return to Bottle Prep. A cart is located in the room for transporting containers. Place the container on the cart. If the cart is full, take the entire cart load to Bottle Prep. Transfer load from cart to storage room.

4.5.6. Client containers are to be returned in as satisfactory condition as possible. Packing/artificial ice and temperature blanks are returned to clients. Packing which resembles trash or is ruined during unpacking is disposed as trash. Packing which is deemed re-usable may be returned also. Drain any water from the container before sealing it closed. Due to partially frozen artificial ice, client containers may not be perfectly dry when returned.

4.5.7. Complete a return mailing label. Secure it with cellophane tape and seal the container with tape also. Return packages are listed in the UPS Logbook and set near the door. Be certain to complete both address labels and the UPS shipping logbook, including weights and zone codes.

4.5.8. When ice is present, it is often enclosed in plastic bags. A basin used for dumping ice is located in Sample Administration. Open the bags and dump ice into the basin. Draining into a closed system, water may not be left running in the basin. When excessive amounts of ice are received, collect the ice in an ice chest and dump it over the storm drains along the sidewalk outside the building. **This is permissible only if the ice and coolers are uncontaminated and the amounts disposed in this way are recorded.** A

logbook located in Sample Administration records the date/time, person and amount of ice.

4.6. Client Deliveries ("Over the Counter")

4.6.1. Samples received over the counter are hand delivered by the client/sampler or by a courier directly employed by the client's company. The pivotal question in such deliveries is whether the person possessing the shipment is in custodial possession of the samples or merely transporting the shipment. If in custodial possession, the deliverer will wish to relinquish custody to you before departing and will probably retain a copy of the documentation. To determine the matter, simply ask the deliverer if he/she had signed any of the sample documents.

4.6.2. If the "client" does not wish to remain while the samples are unpacked and verified, sign the COC somewhere other than the "Received By" section. Include a statement that only the shipping container was received. [Example: One cooler received from Company X in good condition. MMDDYY/ Initials]

4.6.3. Generally, the client/sampler will wish to remain, have the samples verified and relinquish custody to this laboratory. Follow Sections 4.2 through 4.4 as applicable. [Over the counter receipts are a condensed, abbreviated form of standard delivery practices. As such, they require more skill and expertise to accomplish necessary tasks without prolonging the client's involvement beyond what is necessary.]

4.6.4. Mention any discrepancies and allow corrections to be made by the client/sampler. Also ask for any special instructions or important aspects of the project (i.e. rush turn-around times, impending hold-time violations, sample matrix specifics, etc.).

4.6.5. Solicit the client's wishes regarding shipping container and/or any other needs, such as bottle order pick-up or a need to speak with a project manager.

4.6.6. After the client's departure, complete any aspects of Sections 4.2 through 4.4 left unfinished. Prioritize the new project within the existing workload and proceed with the highest priority.

4.7. Project Generation

4.7.1. Log on to the VAX/VMS computer system and enter the Laboratory Information Management System (LIMS) by typing: LIMSMENU <R>. For the basic use of the LIMS system, refer to the LIMS user manual.

4.7.2. Projects can be created using a variety of resources. Minimal project login consists of the Project Screen (pages 1 and 2), Sample List and Group Codes (Example 8). Frameworks for new projects may be dictated using instructions from project managers, copying templates or existing projects.

4.7.3. If project information is not available and a project must be setup from scratch, determine the name and location of client. This information can be found on the COC, shipping labels, or obtained from the project manager.

4.7.4. Pull up the appropriate client screen.

4.7.5. If the client does not exist in the database, a new one must be created using the protocol in the LIMS user manual. New JDE IDs (for billing) need to be requested of corporate by the project manager and are not necessary to complete login at this time.

4.7.6. Search the programs listing of the client and choose the appropriate one for the project. If one does not exist, confer with the project manager whether a new program needs to be created or not.

4.7.7. Once the appropriate program screen is linked with the client, search the project listing for a template or similar project. If none is found a project must be created.

NOTE: Duplication of templates is the most desirable method of project set up as the template contains all necessary information needed to accurately create a project.

4.7.8. The necessary LIMS fields that must be filled in are as follows:

- Project name: follow the naming procedures used for previous projects belonging to the same client. If none exist, list the client's project name and number, and the date.
- Matrices and brief analytical list may also be added if room is available in the data block.
- P.O. number: if no P.O. number is available, list LIMS contact name (i.e. D.Client).
- Description: list the matrix type and analyses requested. Also note anomalies, special requests, and/or any hazards of which analyst(s) should be aware.
- Contact: enter LIMS ID of contact, the person who will receive the final report.
- JDE ID: J.D.Edwards ID of company and/or individual who meets credit requirements.
- Mail To: Address to which invoice is mailed. This may be identical to JDE ID, or completely different. Third party billing and larger companies often use separate address.
- Team ID:
  - 11 - Environmental Department
  - 40 - Advanced Tech, Dioxins
  - 70 - Energetics
  - 561- Air Toxics
- Calculate holding times from collection dates?: If the answer is Yes(Y), LIMS will calculate all analytical holding times from the collection (sampling) date. If the answer is No (N), then LIMS will calculate the holding times from the date of receipt to the laboratory. At present, only projects requiring CLP protocol will have a "No" answer.

- Primary PA: the project manager who is responsible for daily monitoring of project.

- Turnaround Class:
  - NORMAL - Standard Deliverable;
  - RUSH - Faxed Deliverable;
  - EXTENDED - Custom Deliverable (e.g., CLP, IRPMS, or Raw Data).
  - [If unknown, default is "NORMAL".]

- Promised Turnaround Time: enter number of calendar days in order to produce the report one day before "Due Date" specified by client on COC. Default is "20" for NORMAL TAT.

- Actual Start Date: date from which the project TAT is calculated. The project has met all sample delivery acceptance criteria as stated in the Quanterra terms and conditions.

- Receipt Date and Time: actual date and time the shipment arrived at the laboratory.

- Storage location: unit/shelf where samples are stored until rotation or disposal. See listing in Section 4.4.15.

- Comments: short description of the number of samples, matrices, condition upon receipt, delivery method and whether the samples were received under chain of custody. [Examples: Two aqueous samples received under Chain-of-Custody in good condition. Delivered by Federal Express. Soil samples (103) received in good condition, listed on purchase order. Delivered by client.]

4.8. Sample Login

4.8.1. Refer to the LIMS manual section for entering sample information into LIMS.

4.8.2. Creation of group codes depends upon information available when the samples arrive. Each group code lists analyses to be entered against a particular sample. Group codes are constructed to the best of the technician's ability, but final responsibility lies with the Project Manager.

D7 1/n

4.8.3. The Sample List is a series of Sample Detail screens. Each screen records the information for a particular sample. Each screen records client identification, sampling date/time, receipt date/time, due date and container types. This screen does draw information from other screens, but must be individualized to the sample it describes.

NOTE: Sample screens may also indicate a formal record of quality assurance aliquots extracted in order to monitor occurrence of contamination. Dioxin projects always have one method blank logged per matrix type (Example 8). U.S.Navy Program projects require a "VOA Holding Blank" assigned to every SDG (Sample Delivery Group) received.

4.9. Folder Assembly: Use an appropriately colored folder (Red - Rush, Manila -Normal) for project information and manila/any color for sample tracking and complete as follows:

4.9.1. Project Manager Folder

4.9.1.1. Staple the air bill, secondary documentation, the original COC and a ticket report in reverse order to the left hand side of the folder. Tab with project label is to be on right hand side.

4.9.1.2. In reverse order, place the following on the right-hand side of the folder:

a) Project Review Checklist #1 (completed);
b) Data Control Instruction Sheet (Example 9);
c) Ticket Report (for projects with method blank aliquots listed);
d) Sample List with Group Code List underneath (stapled together);
e) Duplicate copy of COC stapled underneath a Ticket Report;
f) Project Receipt Checklist (completed).

4.9.2. In-House COC Folder Assembly

4.9.2.1. Align folder as with the project folder. Tab is on right-hand side.

4.9.2.2. On the right-hand side of the folder, staple the In-House COC form QA188 (Example 10).

4.9.2.3. On the left-hand side, staple a Ticket Report. If the entire bottle inventory for any sample is not complete on the ticket report, include a copy of the bottle inventory (reverse side of PRC).

4.9.2.4. If an MSDS is provided in shipment, staple a copy beneath the ticket report on the right-hand side of the folder.

4.10. Final Tasks

4.10.1. Record project number in PRL on the line corresponding to the shipment's arrival.

4.10.2. Complete Project Review Checklist #1 QA-235 (Example 11).

4.10.3. File an HTV, if necessary, by entering "Anomaly" and the project number at your prompt. Answer the queries, and be certain to mail the HTV to the department supervisor and project manager involved. The HTV is filed by Sample Administration when a holding time is expired upon receipt. If the holding times have not completely expired for a project, the department handling the analysis in question files the HTV.

4.10.4. Place the Project Manager Folder in the project manager's tray and the Internal Tracking Folder in the check-out boxes (both located in Sample Administration).

4.11. Sample Storage

4.11.1. Refrigerator and freezer temperatures will be monitored and recorded once daily Monday through Saturday. Any temperature below 2°C or above 6°C for the refrigerators and above -10°C for the freezer will need corrective action. Refer to "Corrective Action for Refrigerators Out of Acceptable Temperature Range", SOP LP-CAL-0014.

4.11.2. Temperatures will be recorded on the appropriate charts (Example 12). Temperature charts will then be stored in the temperature logbook. All charts for a particular refrigeration unit will be stored together in chronological order.

4.12. Internal Sample Tracking

4.12.1. Analyst selects sample check-out folder for the project of interest and ascertains storage location.

4.12.2. Analyst removes appropriate containers from storage unit and organizes them by project/sample number.

4.12.3. Analyst completes check-out portion of Internal COC and has the record and container labels verified by another Quanterra employee (Example 9). The witness must initial the internal chain of custody. Any laboratory personnel may verify the transaction. If the containers are not to be returned and will enter the wastestream through an extraction lab, the analyst will also enter this information during check-out.

4.12.4. Analyst files the check-out folder before leaving the room.

4.12.5. At return, the analyst completes the check-in portion of the Internal COC.

4.12.6. Analyst returns samples to their storage location.

4.12.7. If subsampling or aliquoting is performed, whether outside the area or not, containers must be checked out by the analyst. The original containers' whereabouts and those of any other containers generated must be documented.

4.12.8. Transfer of volatile containers from VOA to RD and RF do not require verification.

4.12.9. Empty containers are not returned to Sample Administration.

5. DEFINITIONS

5.1. Refer to Glossary in the Quanterra QAMP.

5.2. Chain of Custody (COC): A system of documentation demonstrating the physical custody and traceability of samples.

5.3. A sample is considered in your custody if the following conditions are met:

5.3.1. The sample is in your possession.

5.3.2. The sample is in your view.

5.3.3. The sample was in your possession and was returned to a secured area.

5.3.4. The sample was placed in a secure area accessible to authorized personnel only.

5.4. HTV: Holding Time Violation

6. MISCELLANEOUS (TABLES, APPENDICES, ETC...)

6.1. Ensure client confidentiality at all times, giving special attention during facility audits conducted by clients and regulatory agencies.

6.2. Procedural Additions for Receipt/Acceptance of EPA sample shipments.

6.2.1. Shipment documentation should also include an official EPA Traffic Report, if it has not been incorporated with the Chain-of-Custody. The Traffic Report (TR) is the equivalent of the COC and is to be handled identically.

NOTE: The lowest alpha-numeric identification is used to designate the Sample Delivery Group (SDG). The SDG will be used to identify the twenty samples that are received during the sampling event. A single SDG may be received at one time, or several groups may arrive over a short period of time. If so, every twentieth sample will complete the on-going SDG.

6.2.2. Sample identification also consists of an EPA Sample Tag, which will be fastened to each sample container, either by string/wire ties or by tape. Verify the preprinted tag number against the COC and/or TR listing. Remove each tag and seal inside a ziplock bag. Note the Quanterra project/sample number on the bag as well as the SDG designation. The bags containing the tags are included within the Project Manager Folder.

6.2.3. Volatile holding blanks, if received with a shipment, are labeled with the project number of the applicable SDG and are stored with the samples. Holding blanks will record exposure of samples to contamination and must accompany them during storage. Do not separate holding blanks by assigning them to "V" (VOA).

6.2.4. Shipment anomalies must also be recorded on the DC-1 or EPA/CLP form (Example 13). The DC-1 is generated by the EPA, while the EPA/CLP is Quanterra specific. Each records receipt conditions and discrepancies with associated shipment documentation. Both forms coincide with the PRC, except that sequential airbill/waybill numbers are recorded and therefore must be verified. The completed form is included in the Project Manager Folder, stapled beneath the COC/TR on the left side.

6.2.5. Calculate holding times from collection dates? The answer is "No", according to protocol requirements.

6.2.6. Turnaround class is "EXTENDED" as EPA reports are due thirty-five days from the actual start date. A TAT of forty-five days applies under DFLM01.1 (dioxins).

6.2.7. Actual start date is entered with the completion of an SDG. The actual start date may be postponed pending the resolution of discrepancies between the laboratory and the Sample Management Office (SMO).

6.2.8. All EPA samples and empty containers are to be stored at the laboratory for no less than sixty days following the submission of the respective report. Insert the "DISP-60" status on the Group Code screen during LIMS records creation (Example 8).

EXAMPLE 1

QUANTERRA - West Sacramento
SAMPLE RECEIVING LOG

EXAMPLE 1

Init	CLIENT	DATE	TIME	PROJECT#	COMMENTS	
						1
						2
						3
						4
						5
						6
						7
						8
						9
						10
						11
						12
						13
						14
						15
						16
						17
						18
						19
						20
						21
						22
						23
						24
						25
						26
						27
						28
						29
						30
						31
						32
						33
						34

EXAMPLE 2

Quanterra Incorporated - West Sacramento
PROJECT RECEIPT CHECKLIST

EXAMPLE 2

Client Name: Ice: A Log #: B

Project # (LIMS ID): C Project name: D

Lessee(s): F Initials Date

Date Received: F Time Received: G

Delivered by: H ☐ Federal Express ☐ Airborne
☐ Courier Express ☐ DHL
☐ White Cotton Delivery ☐ UPS
☐ Over the counter (OTC) ☐ Co-Cotton
Other:

Custody Seal Intact: I ☐ Intact ☐ Broken ☐ N/A
Custody Seal Number(s):

Shipping Container(s): J ☐ Quanterra ☐ Client ☐ N/A

Temperature Record (in °C): K
COC#:
Temp Blank:
Ambient Temp:

pH Measured: L ☐ Yes ☐ Annually ☐ N/A

Sample(s) Labeled By: M .....

Sample Labeling Checked By: N .....

Short Hold Time Notification: O Sample Receiving ☐ N/A
Wet Chem ☐ N/A
Metals (Filt/Pres) ☐ N/A

P ☐ Complete shipment received in good condition, with appropriate temperatures, containers, and preservatives. ☐ N/A

☐ Annually (-ins)/monitoring: ☐ Temperature exceeded (2°C-6°C)

Q _____ ☐ PM notified ☐ N/A

Leave no spaces blank. Use "N/A" if not applicable. Initial and date all "N/A" entries. QA125 02/96 MCD

D723

EXAMPLE 3

SUBJECT - Short Holding Time Analysis

EXAMPLE 3

INORGANICS - AQUEOUS SAMPLES

Parameter	Method Number	Holding Times From Collection
pH	150.1	ASAP
Coliform	909A/909C/SM9221	*ASAP
Sulfite	377.1	*ASAP
Chromium III/VI	7196 (water & soil)	24 hours
Chlorine, Residual	330.4	*24 hours
BOD	405.1	*48 hours
Color	110.2	48 hours
Nitrate	300.0/353.2 (unpreserved)	48 hours
Nitrite	300.0/353.2 (unpreserved)	48 hours
Odor	140.1	48 hours
Orthophosphate	300.0/365.3 (unpreserved)	48 hours
Settleable Solids	160.5	48 hours
Surfactants (MBAS)	425.1	*48 hours
Turbidity	180.1	48 hours
AOX	9562	3 days
Sulfide (Waters)	376.2/9030A	7 days
Total Dissolved Solids (TDS)	160.1	7 days
Total Suspended Solids (TSS)	160.2	7 days
Total Solids	160.3	7 days
Total Volatile Solids	160.4	7 days
Alkalinity (all types)	310.1	14 days
Cyanide	335.2/9010	14 days
Free Cyanide	412M(STD)	14 days
Sulfide (Soils)	9030A	14 days

* These analyses not performed at this facility.

ORGANICS - AQUEOUS SAMPLES (Except CLP samples)

Volatiles	8010/8020/8240/8260	7 days
OC Pesticides/PCB's	8080	7 days
Polyaromatic Hydrocarbons (PAH)	8310	7 days
OP Pesticides	8140	7 days
Phenoxy Acid Herbicides	8150	7 days
Semivolatiles by GC/MS	8270	7 days
Volatiles	8010/8020/8240/8260(preserved)	14 days

All organic analyses on soil samples (except CLP samples) require extraction within 14 days after collection. An exception is that soil samples for TCLP-8270 must be TCLP extracted within 7 days of sampling.

ORGANICS - CLP AQUEOUS SAMPLES (Holding Times calculated from receipt date)

OC Pesticides/PCB's	8080	5 days
Volatiles	8240/8260	10 days
Semivolatiles by GC/MS	8270	5 days

ALL CLP organic analyses on soil samples require extraction within 10 days after receipt.
Dissolved Metals - if unfiltered or unpreserved -- ASAP!
Remember to add the P-FILTM or P-FILTPRESM tests in LIMS if the samples need to filtered and/or preserved.

EXAMPLE 3 (cont.)

EXAMPLE 3

Quanterra Incorporated - West Sacramento
SHORT HOLDING TEST NOTIFICATION

CLIENT:_____ ☐ SHT ANALYSIS

PROJECT ID:_____ ☐ PENDING HTV

DATE:_____ ☐ RUSH (24-72 Hr TAT)

TIME:_____ ☐ SEE ATTACHED

CHEMIST NOTIFIED:_____ QAS#_____

GENCHEM ANALYSIS:		SAMPLE #:	ASSIGNED CHEMIST:
☐ pH	☐ ALK	_____	_____
☐ Cr6	☐ EC	_____	_____
☐ TURB	☐ COLOR	_____	_____
☐ SS	☐ SO3	_____	_____
Lachat			
☐ NO2+NO3-A		_____	_____
☐ NO2-A		_____	_____
☐ NO3-A		_____	_____
Method 365.3			
☐ OPO4		_____	_____
Method 300.0			
☐ NO3 ☐ NO2 ☐ OPO4		_____	_____
☐ BR ☐ CL ☐ SO4 ☐ F			
☐ OTHER: _____		_____	_____
_____		_____	_____
_____		_____	_____

HOLDING TIME ALREADY VIOLATED? ☐ YES ☐ NO

HTV FILED? ☐ YES ☐ NO ☐ N/A

☐ MS/SD ☐ MS/DU ☐ CLIENT REQUESTED (Sample#_____) ☐ N/A

COMMENTS:_____

9/96 MCD QA-3[illegible]

EXAMPLE 4

EXAMPLE 4

[redacted]

in of Custody
ord

Quanterra Environmental Services

50444

Sample I.D. No. and Description

Date

Time

Matrix

Containers & Preservatives

Special Instructions/ Conditions of Receipt

Sample Disposal

Return To Client

Disposal By Lab

Archive For

Months

Page of

DISTRIBUTION: WHITE - Stays with the Sample; CANARY - Returned to Client with Report; PINK - Field Copy

Df-26

EXAMPLE 5

EXAMPLE 5

	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20
VOA																				
VOAh																				
AGB																				
AGBn																				
250AGB																				
250AGBn																				
250AGBn																				
250AGBnn																				
AGJ																				
500AGJ																				
250AGJ																				
CGJ																				
500CGJ																				
250CGJ																				
125CGJ																				
PB																				
PBn																				
Pjn																				
500PB																				
500PBn																				
500PBns																				
500PBzn/ns																				
250PB																				
250PBn																				
250PBns																				
250PBzn/ns																				
°CT																				
Folder/Filter																				
PUF																				
PETRJ																				
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20

D727

EXAMPLE 6

LABEL PROGRAM EXAMPLE 6

```
CTRL-Y to exit
Make you selection to print labels

Just the label ma'am!!

0.  Send Shipment Anomaly
1.  For series of samples
2.  For one sample
3.  For specified set of sample #s (up to 25)
4.  For specified starting and ending sample
5.  For set of Custody Seals

Enter your selection:
```

[The most common usages of the selections are explained below. Most selections include shipment anomaly queries.]

0. Asks if shipment was received two and six degrees Celsius. Also provides an edit screen for messages. Sends E-mail to Project Manager and prints hardcopy on SAMPRINT.
1. Prints title labels for Project Manager and Internal Sample Tracking folders. Creates the same number of labels per sample. for each sample beginning with -001. "Notes" query may be used for disposal codes. hazard cautions or accelerated TAT.
2. Prints a number of labels per sample number selected.
3. Similar to selection 1. but allows the number of labels to vary per sample number selected. The link between queries is linear. If 1.5.6.9 and 2.3.4.5 are entered. two labels for sample -001. three labels for sample -005. etc are produced.
4. Allows for the same number of labels to be printed per sample. for sequential samples between the beginning and ending samples selected. (Example: four labels for each sample between -045 and -057).
5. Prints a number of labels which read "Custody Seal".

EXAMPLE 6 (cont.)

LABEL PROGRAM (continued) EXAMPLE 6

```
Selection 1:

Enter project#:  087261
Enter location(s) of sample(s):  N/A
Enter Notes (ie. Hazard info. MS/SD. Rush):  HAZ-MAT
Enter # of samples:  4
Enter # of labels:  3
Enter the client (max 15 chrs):  Quanterra-Macinaw Textile
Any shipment anomalies? (y,n)  ["y" will initiate E-mail option]
Print Labels (y,n):
Can project be rotated?:        [refer to disposal code listed in LIMS]


Anomaly Option:

Enter PM:  KVerrue
Was temperature out of range (acceptable is 4+2 )? (y,n): y
Enter temperature(s): 12
Enter affected sample(s): -001.2.3.4

SHIPMENT RECEIPT ANOMALY(-IES)

            CLIENT:  Quanterra-Macinaw Textile
           PROJECT:  087261
   PROJECT MANAGER:  KVERRUE
AFFECTED SAMPLE(S):  -001.2.3.4
    TEMPERATURE(S):  12

        RECEIPT ANOMALY(-IES)

[Any length message recording poor or unusual shipment
 conditions may be entered here.
```

EXAMPLE 7

```
ANOMALY PROGRAM                                          EXAMPLE 7

[Anomalies may be filed only after login is complete.  Samples must
 have analyses logged against them in order to operate this program.]

      +--086803_INITIAL_MENU------+
      | A. Anomaly Report         |
      | H. HTV Report             |
      |                           |
      | X. EXIT Program           |
      +---------------------------+
      |  : Enter Selection        |
      +---------------------------+
```

```
+--086803_DEPT_MENU------+
| A. Metals              |
| B. Metals_Prep         |
| C. GCMS                |
| D. Special_Services    |L_MENU-------+
| E. Wetchem             |ort          |
| I. Data Control        |             |
|                        |             |
| X. Exit Program        |e            |
+------------------------+-------------+
|  : Enter Selection     |tion         |
+------------------------+-------------+
```

```
--086803_HTV_OPTIONS_MENU-------------------------------------+
|                                                              |
|   Choose HTV Category from List                              |
|                                                              |
| A. Holding Time Expired in Transit (field to lab).           |
|                                                              |
|   Original Analysis Performed within HT: reprepped or        |
|   reanalyzed outside of holding time because:                |
| B.      Internal QC Out.                                     |
| C.      Matrix QC out.                                       |
| D.      Other.                                               |
| E. PA or Log-In Error.                                       |
| F. Capital Resource / Priority Conflict.                     |
| G. Labor   Resource / Priority Conflict.                     |
| H. Instrument Failed.                                        |
| I. Analysis Error.                                           |
| J. Scheduling Error.                                         |
| K. Other.                                                    |
|                                                              |
| X. EXIT Program                                              |
+--------------------------------------------------------------+
|  : Enter Selection                                           |
+--------------------------------------------------------------+
```

EXAMPLE 7

ANOMALY PROGRAM (continued) EXAMPLE 7

[Selection "A - Holding Time Expired in Transit (field to lab)." is generally used, and requires completion of the following screen.]

PROJECT ID : 086803
PA : GILDERSLEEVE
CLIENT : STONE CONTAINER CORPORATION
AREA DIRECTOR : MORBAMOSKY
AREA SUPERVISOR : JJONES (current supervisor)
REPORTED BY : COUNCAN
ANALYSES : PH-A
DATE SAMPLED : MMYYDD
SAMPLES AFFECTED : ALL
DATE HT EXPIRED : (calculate from COC or LIMS)

DATE PREP/ANALYSES
ACTUALLY PERFORMED:

REASON:

1. Holding time expired in transit (field to lab).

EXPLANATION:

[Record any pertinent circumstances concerning this HTV.]

[Edit screens can be terminated by CTRL Z. Enter "exit" to store/send or "quit" to avoid saving.]

EXAMPLE 8

EXAMPLE 8

Quanterra, Inc. Quanterra Environmental Services - Sacramento
Date: 15 APR 96 RBONALY Sample List Screen Print Report
Page 1

PROJECT (Page 1 of 2)
Client: CALLAB-OUTCAD1 Name: Quanterra, Inc. - Sacramento
Program: CALLAB-003458 Name: Advanced Tech (LR) - General Program
Project: CALLAB-087261 Name: [redacted]ASH TCDDDF 041596
Type: NONE Case: JOB: SAS: Level:
PO #: 122623 Invoice #:
Description: Ash samples: TCDDDF and 8080(PCB).

Composite containers of each sample prior to analysis.
Results by dry weight.

-003: sample containers cracked in transit, volume transferred.

Contact: CALLAB-[redacted] Bettz (414)272-0209
Alternate: - - -
CC: Copy: - - -
JOE Id: CALLAB[redacted] ACCT PAYABLE (414)272-0209
Inv To: CALLAB[redacted] ACCT PAYABLE (414)272-0209
Project Status: ACTIVE Status History: AB QAS#
Team Id: 0040 -CAL High Res Market Segment: AT- Advanced Tech
Results to client computer? N Deliverables:
Phone results to client? N Client Project ID: MACINAW
Other Quanterra lab involved? N Project Duplicated from:
Subcontractor involved? N Sales Exec: DMAXSELL
Calculate holding times from collection dates? Y Primary PA: EVERRUE
Delivery Penalty: NONE Turnaround Class: NORMAL
Last Followup Date: Promised Turnaround Days: 20
Actual Start Date: 15 APR 96 Next Followup Date:
Project Due Date: 05 MAY 96 Expected Start Date: 15 APR 96
Receipt Date: 15 APR 96 Time: 10:00 Storage Location: W1A
Comments: Four incinerator ash samples received under Chain-of-Custody in good condition. Delivered by Express Western Delivery.
Log Released: Invoiced:
Preliminary Data: Reported:
Delivery Accepted: RBONALY 15 APR 96 10:30 Reviewed:
Created: RBONALY 15 APR 96 Modified: RBONALY 15 APR 96

Smp	Grp	Alq	Rec Date	Due Date	Matrix	Status	Client Description	Inst
0001	B	* MB	15 APR 96	02 MAY 96	ASH	LOGGED	Method Blank	0
0001	A	* SA	15 APR 96	02 MAY 96	ASH	LOGGED	01NE	0
0002	A	SA	15 APR 96	02 MAY 96	ASH	LOGGED	02SE	0
0003	A	SA	15 APR 96	02 MAY 96	ASH	LOGGED	03SW	0
0004	A	SA	15 APR 96	02 MAY 96	ASH	LOGGED	04NW	0

EXAMPLE 8 (cont.)

EXAMPLE 8

```
Quanterra, Inc.                Quanterra Environmental Services - Sacramento
Date: 16 APR 96          RBONALY              Sample Detail Screen Print Report
                                                                        Page  1

                              SAMPLE DETAIL
Client: CALLAB-QUECA01  Name: Quanterra, Inc. - Sacramento
Program: CALLAB-003458  Name: Advanced Tech (LR) - General Program
Project: CALLAB-087261  Name: [redacted]ASH TCDDDF 041596
 Sample #: 0001 •  Aliquot: MB      Group Code: B
                                                  Sample Status: LOGGED
                                                 Status History: L
 Client Description: Method Blank
 Calculate holding times from collection dates? N
 Collection Date:            Time:                     Interval: 0.00
    Receipt Date: 15 APR 96 Time: 10:00  Turnaround Class: NORMAL
 Sample Due Date: 02 MAY 96                          Matrix: ASH
Quanterra sample #:          414045   Permit #:                 Well ID:
   Lab Description: Method Blank
       Received By: RBONALY
     Disposal Type: NO-DISP                                Last Instruction #: 0
      Color Before:                Clarity Before:              Artifact:
      Color After :                Clarity After :               Texture:
 Delivery Accepted: RBONALY         15 APR 96     Disposed:
            Logged: RBONALY         15 APR 96    Completed:
           Created: RBONALY         15 APR 96     Modified:

  Smp  Alq Rp   Test Code          Ver Matrix   Priority Status   Due Date  M
  0001 MB  0    TCDDDF-S           3   ASH      NORMAL   PREPWAIT 02 MAY 96
  0001 MB  0    P-DIXAFUR-SOX-L    1   ASH      NORMAL   LOGGED   25 APR 96
```

EXAMPLE 8 (cont.)

EXAMPLE 8

Quanterra, Inc. — Quanterra Environmental Services - Sacramento
Date: 15 APR 96 — RBONALY — Sample Detail Screen Print Report
Page 1

SAMPLE DETAIL

Client: CALLAB-QUECAD1 Name: Quanterra, Inc. - Sacramento
Program: CALLAB-003458 Name: Advanced Tech (LR) - General Program
Project: CALLAB-088261 Name: [redacted] ASH TCDDDF 041596
Sample #: 0001* Aliquot: SA Group Code: A
Sample Status: LOGGED
Status History: L

Client Description: DINE
Calculate holding times from collection dates? Y
Collection Date: 14 APR 96 Time: 16:30 Interval: 0.00
Receipt Date: 15 APR 96 Time: 10:00 Turnaround Class: NORMAL
Sample Due Date: 02 MAY 96 Matrix: ASH
Quanterra sample #: 416046 Permit #: Well ID:
Lab Description: 3-500CGJ
Received By: RBONALY
Disposal Type: DISP-30 Last Instruction #: 0
Color Before: Clarity Before: Artifact:
Color After : Clarity After : Texture:
Delivery Accepted: RBONALY 15 APR 96 Disposed:
Logged: RBONALY 15 APR 96 Completed:
Created: RBONALY 15 APR 96 Modified:

Smp	Alq	Rp	Test Code	Ver	Matrix	Priority	Status	Due Date	M
0001	SA	0	TCDDDF-S	3	ASH	NORMAL	PREPWAIT	02 MAY 96	
0001	SA	0	P-DINFUR-SOX-L	1	ASH	NORMAL	LOGGED	24 APR 96	
0001	SA	0	8080-PCB-S	2	ASH	NORMAL	PREPWAIT	02 MAY 96	
0001	SA	0	P-8080-PCB-S	1	ASH	NORMAL	LOGGED	28 APR 96	

EXAMPLE 8 (cont.)

EXAMPLE 8

Quanterra, Inc.
Date: 15 APR 96 RSCHALT

Quanterra Environmental Services - Sacramento
Group Codes Screen Print Report
Page 1

GROUP CODE

Client: CALLAB-QUECA01 Name: Quanterra, Inc. - Sacramento
Program: CALLAB-003458 Name: Advanced Tech (LR) - General Program
Project: CALLAB-087261 Name: [redacted] ASH TCDDDF 041596

Grp	Samp#	Description	Matrix	Disposal	Inst
A	0	TCDDDF/BDBO(PCB)	ASH	DISP-30	0
B	0	TCDDDF(MB)	ASH	NO-DISP	0

Grp	Description	Matrix	Disposal	# Samples Expect
A	TCDDDF/BDBO(PCB)	ASH	DISP-30	0

Package/Project	Test Code	Vr	Rp	Test Description	Inst
	TCDDDF-S	3	0	2,3,7,8-TCDD/TCDF - by Low	0
	P-DIXFUR-SOX-L	1	0	Prep - Low Res. Soxhlet Ext	0
	BDBO-PCB-S	2	0	PCBs in Soil	0
	P-BDBO-PCB-S	1	0	Prep - PCBs by GC	0

Grp	Description	Matrix	Disposal	# Samples Expect
B	TCDDDF(MB)	ASH	NO-DISP	0

Package/Project	Test Code	Vr	Rp	Test Description	Inst
	TCDDDF-S	3	0	2,3,7,8-TCDD/TCDF - by Low	0
	P-DIXFUR-SOX-L	1	0	Prep - Low Res. Soxhlet Ext	0

EXAMPLE 8 (cont.)

EXAMPLE 8

Ticket Report
Quanterra Environmental Services, Sacramento -
880 Riverside Parkway

West Sacramento, California 95605
(916) 373-5600

CALLAB-087261

Date Received : 15 APR 96 10:00

[redacted]

(414) 272-0209

Project ID,
EPA Case, RWA Lot : [redacted]
ASH TCDDDF 041596
P.O. Number : 122623
Delivered By :
Storage Location : W1A
Logged in by : RMONALY

Four incinerator ash samples received under Chain-of-Custody in good condition. Delivered by Express Western Delivery.

Sample ID	Ensaco ID	Client's label info	Date/Time Samp.	Containers
087261-0001-MB	414045	Method Blank		Method Blank
087261-0001-SA	414046	01NE	14 APR 96 16:30	3-500CEJ
087261-0002-SA	414047	02SE	14 APR 96 17:00	3-500CEJ
087261-0003-SA	414048	03SW	14 APR 96 17:30	3-500CEJ
087261-0004-SA	414049	04NW	14 APR 96 18:00	3-500CEJ

Samples not destroyed in testing are retained a maximum of thirty (30) days unless otherwise requested.

Client Manager: Karen Verrue

EXAMPLE 9

Quanterra Incorporated - West Sacramento
DATA CONTROL INSTRUCTION SHEET

EXAMPLE 9

Project/_____

Resubmittal ☐ Old Project/_____

CC:_____(Name)

_____(LIMS ID)

Send by:_____(Date)

MAIL PROCESS:

☐ Quanterra Courier
☐ DHL
☐ California Courier
☐ Federal Express
☐ Regular Mail
☐ Other:_____

☐ Raw Data
☐ Disk Deliverables
☐ Client Accounts
☐ E-Mail
☐ Tier I
☐ Tier II

INVOICE

☐ LIMS ONLY (Prime invoice from lims only)
☐ PLIN (Prime invoice w/PLIN in lieu of expense)
☐ PLIN & PJ DIST (Prime invoice w/PLIN and Project documents, surcharge is by test)
☐ PROJ DIST (Prime invoice w/project documents, surcharge is by test)
☐ PROJ DIST/SUB (Prime invoice w/project documents and project surcharge)

☐ Mail with report
☐ Mail to accounts payable w/copy sent with report
☐ Other _____

ADDED INSTRUCTIONS

_____
_____
_____
_____
_____
_____
_____
_____
_____
_____
_____
_____
_____

(OVER)

EX#9 MCD

EXAMPLE 9 (cont.)

EXAMPLE 9

ADVANCED TECHNOLOGY
REPORTING INSTRUCTION SHEET

Quanterra Environmental Services

Project #_____

Date Due to Client_____

[ ] Fax Date _____

Report To: _____

Fax #_____

Send Original Report By:
[ ] Fed-X
[ ] Regular Mail
[ ] International Mail
[ ] Quanterra Courier
[ ] Electronic Delivery
[ ] _____

Include: _____ Copy/Copies
[ ] Summary Data Sheets & COC
[ ] Summary Results Only
[ ] Chromatograms
[ ] Initial Calibration
[ ] Continuing Calibration
[ ] LCS/OPR
[ ] Disk (Format) _____
[ ] Paginated
[ ] Wet Weight [ ] Dry Weight
[ ] TEQ's
[ ] Ion Ratio's
[ ] QAPjP, SOW in place
[ ] Separate Dioxin and Furan Results
[ ] _____

CC Report Copy To:

_____
_____
_____

CC Deliverables:
[ ] Same As Above
[ ] Cover Letter Only
[ ] Summary Data Sheets & COC
[ ] _____

Mail Invoice:
[ ] To Accounts Payable with Copy in Report
[ ] With Report
[ ] _____

Cover Letter Comments:

_____
_____
_____

Additional Instructions:

_____
_____
_____

EXAMPLE 10

EXAMPLE 10

Quanterra Incorporated - West Sacramento

PROJECT ID: _____ LOC: _____ IN-HOUSE CHAIN-OF-CUSTODY Page ___

SAMPLE#	CONTAINER	CHECK-OUT: INIT	CHECK-OUT: TEST	DATE	TIME	W	SAMPLE#	CONTAINER	CHECK-IN: INIT	DATE	TIME	STOR LOC	COMMENT

1. Sampled Item (1,2,4-10)
2. Containers (ex 1AG0,2HCCG,3WPL,1HCCG)
3. Initials your initials
4. Test: analysis sample is for
5. Date: date taken/returned
6. Time: time taken/returned
7. W: witness of check-out
8. Stor Loc: location samples returned to
9. Comments: (ex empty,refusing)

QA55 D7

Dt 39

EXAMPLE 11

Quanterra Incorporated - West Sacramento
Project Review Checklist #1

EXAMPLE 11

Project Number_____

Shipment Receipt:

__ Bill(s) of lading received (signature if necessary)
__ COC/Letter of Transmittal signed and dated with time of receipt
__ Condition of samples noted on COC
__ Project Receipt Checklist completed
__ COC generated for undocumented samples (from label info)

Project Folder:

__ Data Control Instruction Sheet included
__ Sample ID(s) and collection date(s)/time(s) match COC or discrepancies explained
__ Sample list screen report stapled to groupcode screen report
__ Groupcode(s) verified
__ Number, matrix, and condition of samples present in comments
__ Means of delivery present in comments
__ Correct receipt date and time recorded
__ Ticket report stapled to copy of COC
__ Ticket report, original COC, and bill(s) of lading stapled to left side of folder
__ Folder color reflects TAT assigned (Red for Rush, Manilla for Normal)
__ Project label is correct
__ E-mail notification sent to Project Manager
__ Billing calculated
__ Wrote project# in log book

__ Watchers notified of SHTT(s) and metals notified of filtering/preservation
__ HTV filed for samples with expired hold times upon receipt (if applicable)
__ Anomaly sent
__ Subbed out analyses changed to SENT-OUT (or @CEL, @WADS, etc.)
__ RUSH e-mail notification sent to appropriate department(s)

In-House COC Folder:

__ In-house COC stapled to right side of folder
__ Ticket report and copy of bottle inventory (if needed) stapled to left side of folder
__ Labels are correct
__ Folder filed

Initial/Date _____

QA235 03/96 MCD

EXAMPLE 12

QUANTERRA INCORPORATED
West Sacramento
Temperature Log

EXAMPLE 12

Thermometer ID: N/A

Location: Sample Receiving

Unit #: WALK-IN

Month/Year: 5/96

Temperature must be within 2 - 6°C.

If temperature readings fall outside this range, inform the QA Department. If the date indicated falls on a weekend, write "WKND" in the space.

Date	Temperature	Initials	Date	Temperature	Initials
1	___	___	17	___	___
2	___	___	18	___	___
3	___	___	19	___	___
4	___	___	20	___	___
5	___	___	21	___	___
6	___	___	22	___	___
7	___	___	23	___	___
8	___	___	24	___	___
9	___	___	25	___	___
10	___	___	26	___	___
11	___	___	27	___	___
12	___	___	28	___	___
13	___	___	29	___	___
14	___	___	30	___	___
15	___	___	31	___	___
16	___	___			

Comments: _____

Please be sure to anomalize any excursions or omissions on this log sheet.

EXAMPLE 13

EPA / CLP SAMPLE LOG-IN SHEET EXAMPLE 13

Lab Name: QUANTERRA Environmental Services, Inc. (West Sacramento)	Project Receipt Logbook #
Shipment Received By (Print Name/Signature):	Logbook Page #
Form Completed By (Print Name/Signature):	Case/SAS #
LIMS Entry Date	SDG #

Remarks		Sample ID	EPA Tag #	Assigned Lab #	Condition of Sample Shipment, etc.
1. Custody Seals	Present / Absent Intact / Broken				Sample Condition:
2. Custody Seal No.	_____				
	_____				
3. Chain-of-Custody Form/ Traffic Report	Present / Absent				
4. Delivery Method(s)	Bill / Driver Present / Absent				
5. Waybill(s) Number	_____				
	_____				Discrepancies:
6. Sample Tags	Present / Absent / NA				
7. Sample Tag Numbers	Listed / Not Listed / NA				
8. Data information on all items agree?	Yes / No				
9. Shipping Container	Intact / Damaged				
10. Temperature(s)	Blank_____				
	Analytical_____				
11. Date Received at lab	_____				
12. Time Received	_____				

Sample Storage and Transfer			
Analysis	Location(s)	By	On
Analysis	Location(s)	By	On

Include this form with full EPA or full CLP projects.
1100 SCD

SA-001

SOP AMENDMENT

SOP Number:	SAC-QA-0003
SOP Title:	SAMPLE RECEIPT AND PROCEDURES
SOP Sections Affected by Change:	All.
Reason for Addition or Change:	An appendix has been added to deal specifically with chemical warfare agents and their degradates. The appendix must be reviewed prior to first-time handling of samples suspected of containing degradate contaminants.
Change Effective From [Date]:	3/13/97
Change or Addition (Specific Section; use additional sheets if necessary.):	See attached Appendix 1 to SAC-QA-0003.

Submitted by: **Date:**

Approved by:*

Technical Reviewer: Michael S. Fleury *Date:* 3/14/97

EH&S Signature: Cheryl Stitt *Date:* 3/14/97

QA Signature: [illegible] *Date:* 3/14/97

Management Signature: [illegible] *Date* 3/14/97

Must be same signature authorities of SOP being revised. d:\\qsacca01\sacqa\sop\amend.dot

D743

Appendix 1

Chemical Warfare Degradates - Potential Hazards in Sample Receipt

Background

Quanterra has begun receiving samples to be analyzed for degradates of chemical warfare agents. These degradates are not particularly toxic, as compared with most of the compounds we deal with every day. The fact that these compounds are degradates of chemical warfare agents does, however, present a **new type of potential hazard** for us. We have developed policies regarding the handling of such samples but as we begin to get real-world samples, we are finding that some of our initial assumptions regarding them are not holding true. The purpose of this document is to discuss these compounds and the potential hazards involved in handling them.

Review of Agent Compounds

Chemical warfare agents fall into a wide variety of categories, ranging from defoliants such as Agent Orange (tear gas) to nerve agents such as Sarin. The two types of agents we are most concerned with are the **nerve agents** and the **blistering agents**, such as sulfur mustard. Other lethal agents have been developed and tested for use in chemical warfare; however, these other compounds are either extremely volatile and reactive (and therefore highly unlikely to be present in an environmental sample) or were never produced in significant quantities in the U.S.

Nerve agents

These compounds are members of the organophosphate class of compounds. These compounds are similar to many common household pesticides such as diazinon. The difference is that the nerve agents are far more toxic to humans. The first nerve agent, Tabun, was discovered by a German pesticide company during the process of screening new compounds for pesticide activity. Once the toxicity of Tabun was determined, various military organizations began screening many related compounds in hopes of finding even more efficient ways of annihilating large numbers of people. Out of this effort have come five established (non-classified, i.e. public domain) agents. --- **Tabun (or GA), Sarin (or GB), GD, GF, and VX.**

All of these compounds are toxic via inhalation, ingestion, skin contact, or just about any other route of entry into the body.

A **characteristic of these compounds is their volatility**. A more volatile nerve agent will disperse in air more effectively than a less volatile one, producing increased numbers of casualties through inhalation.

Less volatile agents, on the other hand, will stick around on soil, vegetation, clothing, etc. and will therefore last longer. Out of these various agents, the only two produced in significant quantities in the U.S. are Sarin and VX. **Sarin is the most volatile** of the above agents while VX is the least volatile. Both are exquisitely toxic - a drop of pure VX barely visible to the naked eye is enough to kill a person through skin contact.

Blistering agents

These were the first of the modern agents developed specifically for the purpose of killing people. Unlike the nerve agents, the blistering agents are basically different mixtures of one compound, sulfur mustard, with other non-toxic chemicals which affect its dispersion characteristics.

The primary hazard with sulfur mustard involves skin contact. The term "mustard gas" is somewhat of a misnomer - the stuff will kill you if you inhale enough of it, but this not likely to occur as it is not particularly volatile under normal conditions. Significant quantities of sulfur mustard (a.k.a. HD or HT) have been produced in the U.S.

Chemical Agents in the Environment

It is unlikely (but never impossible) that we at Quanterra will receive any sample which contains a dangerous concentration of active agent. We **should not** receive samples from any area known to be contaminated with bulk amounts of active chemical agent (areas with buried drums, old munitions, etc.) because the Army policy for such sites is to destroy the agent onsite.

Additionally, most of the areas in which degradate analysis is required are areas in which agent was used decades ago - all active agent is likely to have degraded. Nonetheless, it is imperative that we know as much as possible regarding the compounds and the samples in order to protect ourselves against any potential hazard.

The behavior of these compounds in the environment has been extensively studied. Most of the information we have is from a study titled **Environmental Chemistry and Fate of Chemical Warfare Agents**. This study was prepared for the Army Corps of Engineers by Southwest Research Institute in 1994.

Nerve Agents

All of the compounds we are concerned with (Sarin and VX) undergo **hydrolysis in the presence of water**. This hydrolysis proceeds at different rates, depending upon the compound. At worst (cool temperature, normal pH, no dissolved ions, no microbes), either of these compounds getting into water would be degraded to extremely low levels ($< 1 \times 10^{-6}$ of the original concentration) within a couple of years. It is more likely that this level of degradation would occur much more quickly.

Degradation in soil is a far more complex issue. The rate of hydrolysis will depend upon a variety of factors, including soil moisture, pH, mineral content, microbes, temperature, etc. Most available studies show that these compounds last no more than a few days in the tested soil types.

Sulfur Mustard

In some ways, sulfur mustard behaves in a fashion similar to the nerve agents - i.e. it hydrolyzes rapidly in the presence of water. There is, however, an important difference. Under the right conditions, **the hydrolysis products of mustard can polymerize and form bubbles containing active mustard**. The mustard inside of these bubbles is shielded from further hydrolysis by the hydrolysis products. This has turned out to be a problem in areas where large amounts of mustard were dumped at sea. Fishermen in such areas have been injured due to pulling up nets contaminated with blobs of active mustard. An indication that this may have occurred would be a biphasic sample. This situation can also occur in soil.

Potential Hazards to Quanterra Personnel

Following steps listed in this Appendix and other safety policies will help reduce hazards to the greatest degree possible. However, such policies are no substitute for educated, observant personnel.

As always, you must think about what you are doing when you handle these materials. No policy can account for every potential situation. Staff are expected to follow all sample handling policies identified in the Chemical Hygiene Plan and steps following sample receipt listed in this Appendix.

Soil samples should be screened for agent prior to their shipment to Quanterra. Data should be reviewed at the project management and EH&S staff level to ensure samples are "safe" for staff to handle. If the screening status is unknown (i.e. no data is available), project management personnel (Karla Buechler or Mike Filigenzi) should be consulted. Samples **should not** be handled. If staff are unavailable, the cooler should be left in cold storage until the situation is resolved.

Water samples for degradate analysis are not likely to be screened for active agent prior to our receipt. This was not our original understanding, but the Army's policy appears to be that active agents degrade quickly enough in aqueous systems that no screening is needed. The data provided in the Environmental Chemical Fate and Chemical Warfare agents seems to support this notion. Half-life for many of the agents have been documented and is readily available for review by staff. None-the-less, it is still important to exercise care with such samples.

When in doubt, seek assistance from project management, a TC or EH&S staff.

Sample Receipt Procedures

⇒ Follow SAC-QA-003, Section 4 (Procedure)

⇒ Follow steps 4.2.1 - 4.2.5

⇒ Opening Shipment -- Prioritizing Workload

⇒ Review screen data BEFORE opening the cooler if soil samples.

Note: Double gloves are required when handling chemical warfare degradates samples.

⇒ Following established safety policies, open the cooler, remove any paperwork, and check the interior condition.

⇒ Suspect coolers should be opened in the Hood to reduce any potential for exposure.

⇒ If a sample contains a broken or leaking sample, isolate the cooler in the hood and IMMEDIATELY contact the project manager, TC and/or EH&S staff.

⇒ Any sample which appears to be biphasic in appearance must be isolated. Immediate notification to project management, TC and/or EH&S staff is required.

⇒ When in doubt, get help regarding sample receipt. Worker, health and safety is paramount to sample analysis.

<u>Scope of Work</u>

It is possible that we will receive a large number of samples from the **Aberdeen Proving Grounds (APG)** over the next few years. APG has been the center for chemical warfare training and research in the U.S. since 1918. We have been told by our client (EA Engineering) that the procedure for investigating sites at APG is as follows:

- APG personnel perform a **hazard analysis** to determine whether there is an potential for agent contamination in soil based on historical information.
- If there is reason to believe chemical agents may have been used at the site, soil samples are taken and **screened** for agent.
- If the screening shows no agent contamination, sampling for other parameters may proceed.

We do not have enough experience with these projects to know whether this procedure is unique to APG or a standard Army procedure. We are conducting additional investigation to further verify this.

In any case, results of screening will be made available to Quanterra.

Regardless of screening data, any cooler containing samples for degradate analysis (or any other samples from an area suspected of potential agent contamination) **should be inspected carefully** upon receipt. Anyone inspecting the samples, logging them in, or handling them for any other reason should observe all of Quanterra's regular safety procedure.

In addition, **two pairs of gloves** should be worn in order to minimize any potential for skin contact with toxic compounds. Please note that **skin contact appears to be the most likely potential route of exposure.** This is based on the fact that nerve agents are likely to have degraded leaving sulfur mustard as the most likely potential contaminant and due to the likelihood that the samples will be cold. The temperature in the cooler is important from the standpoint of safety as well as sample integrity - cold samples mean a significantly lower potential for any kind of toxic vapor formation. Coolers containing broken jars or bottles should be placed in a hood immediately and left there until the client has been contacted. EH&S staff and/or the Project managers will give instructions regarding return to client or disposal based on the screen data.

In conclusion, it must be emphasized that these samples must be handled with the appropriate level of care. Observant, educated personnel are our best defense against exposure to any kind of toxic materials found in our samples.

APPENDIX E
NEESA 20.2-047B - SAMPLING AND CHEMICAL QUALITY ASSURANCE REQUIREMENTS FOR THE NAVY INSTALLATION RESTORATION PROGRAM

JUNE 1988

SAMPLING AND CHEMICAL ANALYSIS QUALITY ASSURANCE REQUIREMENTS FOR THE NAVY INSTALLATION RESTORATION PROGRAM

NEESA 20.2-047B

NAVAL ENERGY AND ENVIRONMENTAL SUPPORT ACTIVITY
Port Hueneme, California 93043

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Project Manager: Anthony R. Sturtzer

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Prepared by

Gloria J. Mencer
Mitzi S. Miller
J. W. Zolyniak

Prepared by the
Oak Ridge Gaseous Diffusion Plant
Oak Ridge, Tennessee 37831
operated by
MARTIN MARIETTA ENERGY SYSTEMS, INC.
for the
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LIST OF ACRONYMS

AA	Atomic Absorption
ASTM	American Society for Testing Materials
BNA	Base Neutral Acids
CCC	Calibration Check Compounds
CERCLA	Comprehensive Environmental Response, Compensation and Liability Act
CLP	Contract Laboratory Protocol
COC	Chain of Custody
DQO	Data Quality Objective
EFD	Engineering Field Division (U.S. Navy)
EIC	Engineer in Charge (U.S. Navy)
FEL	Field Equipment Log
GC	Gas Chromatography
HSL	Hazardous Substances List
IRP	Installation Restoration Program
ICP	Inductively Coupled Plasma
LQAC	Laboratory Quality Assurance Coordinator
MPR	Monthly Progress Report
MS	Mass Spectrometry
NCR	NEESA Contract Representative (Martin Marietta Energy Systems, Inc.)
NEESA	Naval Energy and Environmental Support Activity
NPL	National Priorities List
PCB	Polychlorinated Biphenyl
PVC	Polyvinyl Chloride
QA	Quality Assurance
QC	Quality Control
RRF	Relative Response Factor
RSD	Relative Standard Deviation
SOW	Statement of Work
SPCC	System Performance Check Compounds
TCL	Toxic Contaminant List
VOA	Volatile Organic

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1. INTRODUCTION

The Installation Restoration Program (IRP) identifies and evaluates past hazardous material disposal sites in order to control the migration of hazardous contaminants. The program also controls hazards that may result from these past disposal operations. The IRP has the following phases: Preliminary Assessment/Records Search, Site Inspection/Remedial Investigation, Technical Base Development, Feasibility Study, and implementation of selected alternatives for remediation. During any of these phases, analysis of soil, water, and waste samples may be performed. The Navy program for the IRP includes performing field investigations and analysis of samples. The purpose of this document is to specify the requirements for the control of the accuracy, precision, and completeness of the samples, and data from the point of collection through reporting. Because every instance and concern may not be addressed in this document, contractors are encouraged to discuss any questions with the Navy engineer in charge (EIC) or the appropriate Naval Energy and Environmental Support Activity (NEESA) contract representative (NCR).

1.1 SCOPE

Laboratories performing studies in support of the IRP are required to obtain Navy approval prior to beginning field studies or analyses of samples and to maintain that approved status throughout the site characterization. The laboratory approval is specific to a particular study for a given site and Statement of Work (SOW). The Navy Requirements document provides guidance to the laboratories on obtaining and maintaining approval. Should more than one laboratory be involved in the analysis of samples from a single site, each laboratory performing analysis must be approved and must comply with the quality control (QC) requirements. These objectives and requirements conform, in general, with the *U.S. Environmental Protection Agency Federal Register*, November 29, 1983 (p. 53937 or 40 CFR 792), the *Food and Drug Administration Federal Register*, December 22, 1978 (p. 59986 or 21 CFR 58), the *Quality Assurance Program Requirements for Nuclear Facilities*, ANSI/ASME NQA-1, 1986 ed., and the *Interim Guidelines and Preparing Quality Assurance Project Plans* (U.S. EPA, EPA-600/4-83-004, QAMS-005/80).

Each laboratory is required to submit a Laboratory Analysis Quality Assurance (QA) Plan. Each engineering contractor must submit a site work plan as part of the approval process. The laboratory's QA plan and the site work plan are emphasized, since the content of those plans and the laboratory's strict adherence to it are essential for obtaining and maintaining Navy approval. Certain basic requirements are stressed--a laboratory QA coordinator (LQAC), the use of accepted analytical methods, careful documentation of chain of custody (COC), corrective action policy, and use of control charts. The laboratory-approval process and subsequent laboratory reporting requirements provide the mechanism for verifying that a laboratory is adhering to its QA and work plan.

Currently, most IRP studies do not include analysis of air, plant, or tissue samples. Future revisions that will include more discussion as to available methods for biota and air are planned. If questions on these methods arise, the NCR may be consulted at the Martin Marietta Energy Systems, Inc., Analytical Chemistry Department at the Oak Ridge Gaseous Diffusion Plant. Where Environmental Protection Agency (EPA) methods are not available for biota, methods from other agencies and published methods which have undergone method validation by the laboratory requesting approval must be used. On occasion, when methods are required for biota and no EPA method is available, the proposed method must be submitted to the NCR for approval.

1.2 APPROACH

The approach reflected in this document is one of outlining requirements and allowing the laboratories, principally through their QA plans, to detail their approach to meeting these requirements. For example, with the exception of the laboratory control sample program, see Sect. 4.4, the discussion of QC procedures includes a requirement that warning and action limits be set but allows each laboratory to describe its procedures for establishing such limits. The specific organization and presentation of the laboratory plan are left largely to the discretion of the laboratory, although certain areas must be addressed.

In order for the above approach to work, emphasis must be placed on effective communication between the laboratory, the Navy EIC, the NCR, and the engineering subcontractor. All documents must be concise, well organized, and free of jargon that might hinder constructive review and evaluation.

1.3 LEVELS OF QC

Data quality objectives (DQOs) are requirements needed to support decisions relative to the various stages of remedial actions. Throughout the project planning process, DQOs are supplied through qualitative and quantitative statements. They are specified in such documents as sampling plans, work plans and QA plans. Five general levels of analytical options to support data collection are identified by Comprehensive Environmental Response, Compensation and Liability Act (CERCLA). The Navy has adopted three of the analytical levels as QC requirements. They are C, D, and E, which correlate with Levels 3, 4, and 5 described in *Data Quality Objectives for Remedial Response Activities Development Process* by the EPA. These levels are based on the type of site to be investigated, the level of accuracy and precision required and the intended use of the data. The level of QC required at the site will be decided by the Navy EIC. Analytical requirements for the remaining two levels have not been defined. Table 1.1 outlines the basic QC requirements at each level. The laboratory method requirements for each level of QC are outlined in Sect. 7.

Table 1.1. Overall plan for QC based on type of site

DQO Levels[1]	Type of Site	QC Requirements						
3	Major Non-NPL Level C	PE sample	Laboratory[2] audit	QA Plan review	Use EPA-approved method[3]	Monthly review	10% field duplicates	Review of final data
4	NPL Level D	PE sample	Laboratory[2] audit	QA Plan review	Use CLP procedures	Monthly review	10% field duplicates	CLP validation
5	Non-NPL Level E	PE sample	Laboratory[2] audit	QA Plan review	Use EPA-approved methods.[3] Non-EPA methods for tissue and explosives.	Monthly review	5% Field duplicates	Review of final data

[1]QC criteria for DQO Levels 1 and 2 has not been defined.
[2]All laboratory audits will be performed by the NCR.
[3]Includes methods from SW 846, Amercian Society for Testing Materials, and Federal Register.

CLP - Contract laboratory protocol
PE - Performance evaluation samples
DQO - Data quality objective

1.3.1 Level C QC

A site requiring Level C QC would be a site near a populated area, not on the NPL, and not likely to be undergoing litigation. The Level C QC includes review and approval of the laboratory QA and the site work plan. The laboratory must successfully analyze a performance sample, undergo an audit, correct deficiencies found during the audit, and provide MPRs on QA. The laboratory that performs Level C QC must have passed the performance sample furnished by the Superfund CLP in the past year. The laboratory does not need to be receiving CLP bid lots of samples.

Level C allows the use of non-CLP methods but requires that the methods be accepted EPA methods listed in Tables 7.1 through 7.5. All methods used must be EPA methods or be equivalent to EPA methods. Further discussion about these methods is presented in Sect. 7. The laboratory must successfully analyze a performance sample, undergo an audit, correct deficiencies found during the audit, and provide MPRs on QA. These audits will be administered and evaluated by the NCR. The Navy audit and performance sample are required in addition to any specified by the EPA Superfund Program.

1.3.2 Level D QC

Level D QC is to be used for sites that are on or about to be on the National Priorities List (NPL). These sites are typically near populated areas and are likely to undergo litigation. Level D QC includes review and approval of the laboratory QA plan, the site work plan, and the field QA plan. The laboratory must successfully analyze a performance sample, undergo an audit, correct deficiencies found during the audit, and provide monthly progress reports (MPRs) on QA. These activities will be administered and evaluated by the NCR. This audit and the analysis performance sample are in addition to those related to the EPA Superfund Program. The laboratory that performs Level D QC must have passed the performance sample furnished through the Superfund Contract Laboratory Protocol (CLP) and must be able to generate the CLP deliverables. For a Level D site, the CLP methods are used and the CLP data package generated. The Navy audit and performance sample are required in addition to any specified by the EPA Superfund Program.

1.3.3 Level E QC

A site requiring Level E QC will be located away from a populated area, will not be an NPL site, and will have a low probability of litigation. Level E QC includes review and approval of the laboratory QA plan and the site work plan. The laboratory must successfully analyze a performance sample, undergo an audit, correct deficiencies found during the audit, and provide MPRs on QA. For Level E, the laboratory is not required to have passed a CLP performance sample. Level E allows the use of non-CLP methods but requires that the methods be accepted EPA methods listed in Tables 7.1 through 7.5. All methods used must be EPA or equivalent. Further discussion about these methods is presented in

Sect. 7. Level E QC is also appropriate for analysis of the contents of underground storage tanks where the samples are primarily pure product or waste.

1.4 ROLES AND RESPONSIBILITIES

As indicated in Fig. 1.1, the organizations involved are NEESA, the Navy Engineering Field Division (EFD), and the subcontractors. Each organization has multiple tasks and groups that support the project. Fig. 1.1 includes the structure of the organization related to the IRP process. A brief description of the key roles and responsibilities is listed.

1. Navy Energy and Environmental Support Activity

   NEESA is responsible for ensuring that the quality of laboratory analyses performed during the various phases of the IRP is acceptable. NEESA is also responsible for managing the NCR.

2. Engineering Field Division

   The EIC at the EFD provides the site information and history, provides logistical assistance, specifies the site requires investigation and reviews results and recommendations.

3. Engineer in Charge

   The EIC is responsible for coordinating procurement, finance, and reporting; for ensuring that all documents are reviewed by the NCR; for communicating comments from the NCR and other technical reviewers to the subcontractors; and for ensuring that the subcontractors address all the comments submitted and take appropriate corrective actions.

4. NEESA Contract Representative

   The NCR is responsible for ensuring that each project has appropriate overall QA. The NCR reviews laboratory QA plans, work plans, submits performance sample data, provides field and laboratory audits, and reviews data from the site. The questions from subcontractors and the EIC regarding specific field and laboratory QC practices are directed to the NCR. The NCR also provides evaluation of referee samples.

5. Engineering Subcontractor

   Each project has an engineering subcontractor that specializes in setting up the sampling for IRP studies, evaluating the hydrology and geology of a site, assessing risks of contamination, and designing and implementing clean-up techniques. Each engineering firm is required to have a laboratory available to perform sample analysis.

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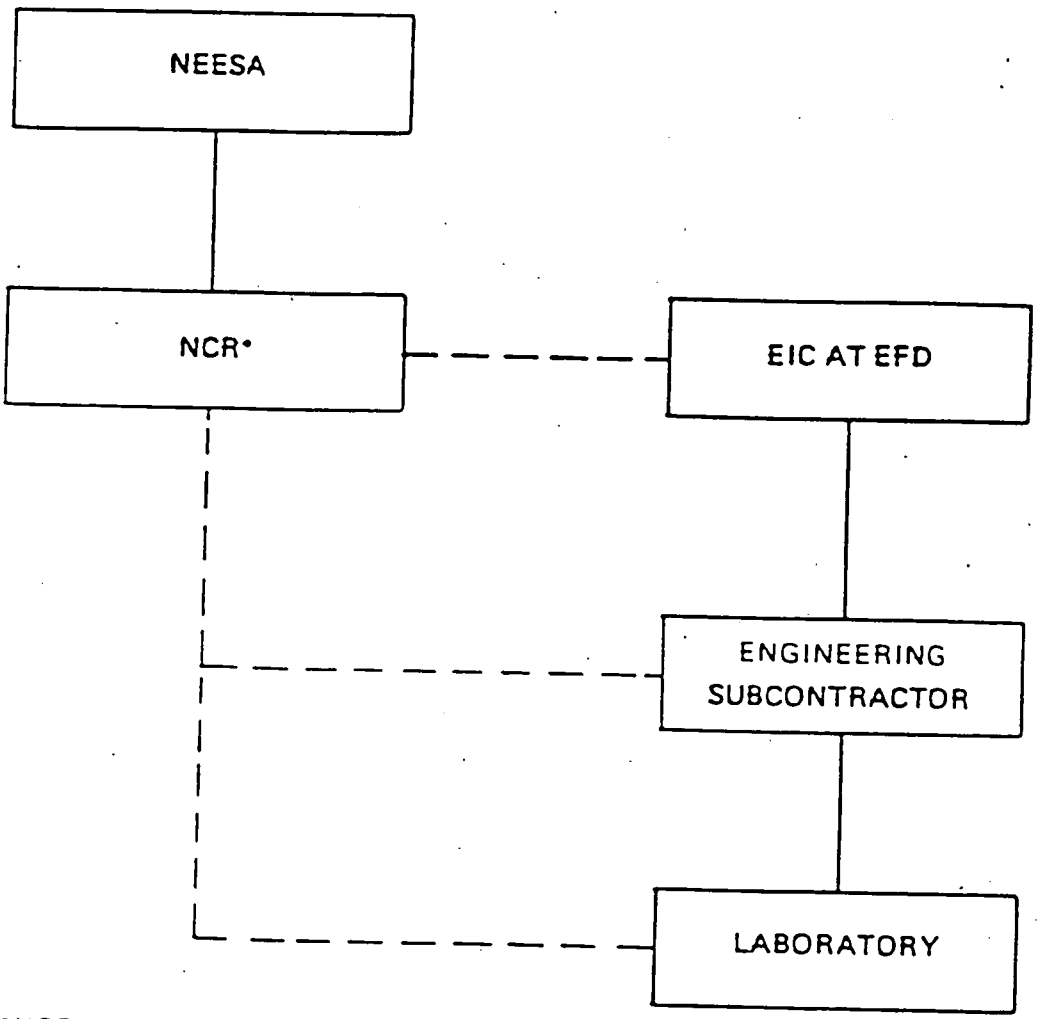

*NCR-MARTIN MARIETTA ENERGY SYSTEMS, INC.

Fig. 1.1. QA Organization

The engineering firm also employs drillers and other personnel to perform IRP tasks. The engineering firm submits a site-specific work plan.

6. Analytical Laboratory

The analytical laboratory is employed by the engineering firm and must adhere to the laboratory requirements in this document. The laboratory is required to prepare and submit a laboratory QA plan, to analyze and submit the results of proficiency testing, to submit to an on-site inspection, and to correct any deficiencies cited during inspection by the NCR. The laboratories are required to identify a LQAC responsible for overall QA. The LQAC must not be responsible for schedule, costs, or personnel other than QA assistants. It is preferred that the LQAC report to the laboratory director. The LQAC must have the authority to stop work on projects if QC problems arise which affect the quality of the data produced.

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2. APPROVAL PROCESS

Prior to beginning any field studies or analysis of samples from the field, contract laboratories will be required to receive Navy approval. This section describes the laboratory approval process in terms of the activities and documentation required of participants in the process.

2.1 OVERVIEW

Laboratory approval is necessary to ensure that contract laboratories meet the minimum requirements for a QC program that facilitates the generation of data of defensible accuracy and precision. Specific objectives of the approval process are as follows:

- to communicate Navy's QC requirements to the laboratories,
- to verify that such requirements are being met by each laboratory prior to analysis of Navy field samples,
- to establish plans for maintaining the QC program while work is being done for the Navy, and
- to ensure that proper communication and planning have been done between the engineering subcontractor and the laboratory prior to the laboratory receiving samples.

The above objectives will be met through an approval process that includes the following elements:

- proficiency testing through analysis of performance samples,
- laboratory inspection and audit,
- reviewing laboratory QA plans,
- reviewing site-specific QC plans, and
- reviewing of sampling plans including QC procedures.

The overall process and the above elements are described in detail in the remainder of this section.

2.1.1 The Laboratory Approval Process

The laboratory approval process, as depicted in Fig. 2.1, begins with the engineering subcontractor awarding a contract to the laboratory. The engineering subcontractor is responsible for supplying a site-specific work plan to the NCR. The laboratory and engineering contractors are required to prepare a site-specific work plan and laboratory QA plan.

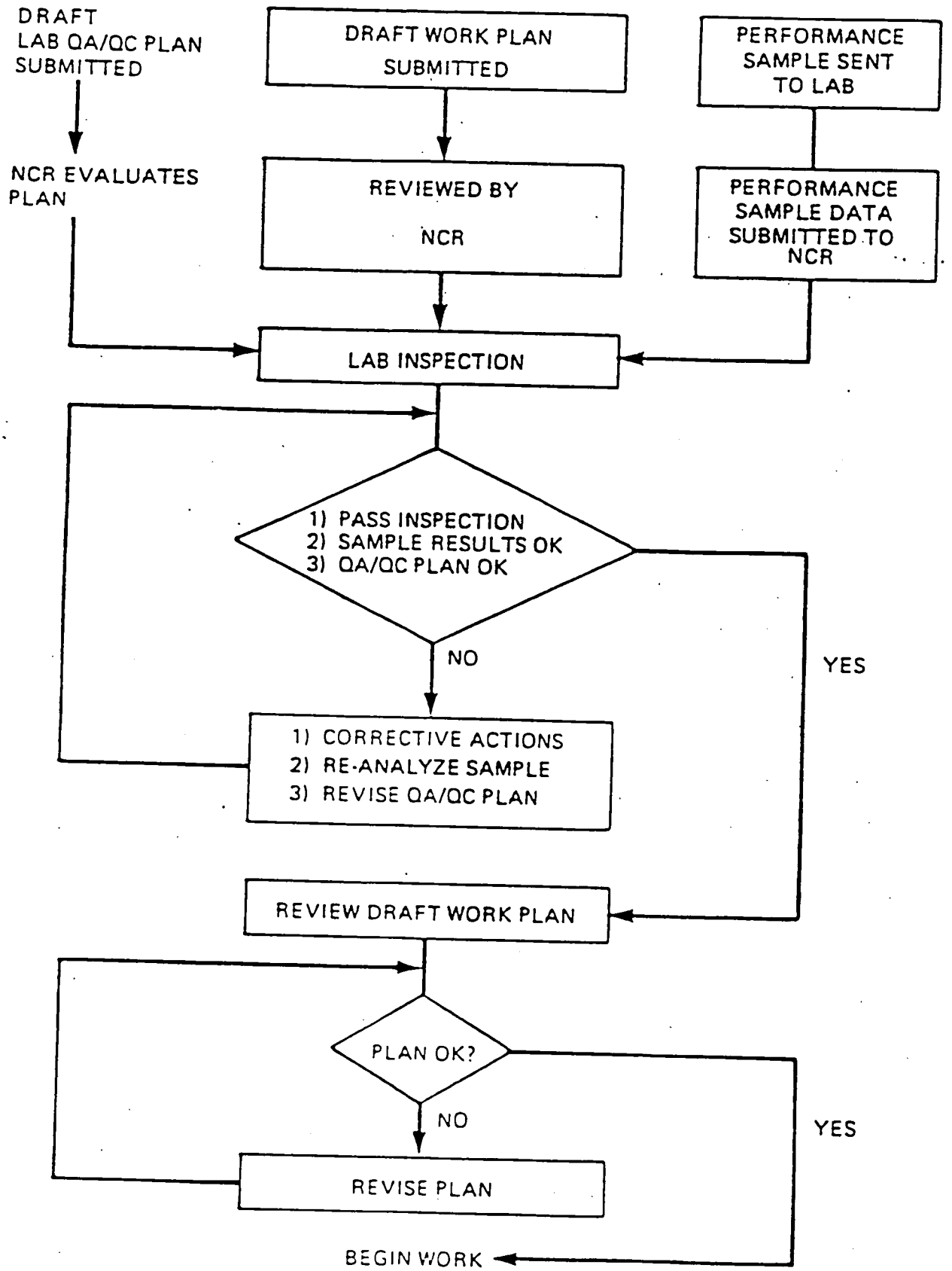

Fig. 2.1. Approval and Review Process

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The site-specific work plan shall include a section on QA. This section shall either outline the field and laboratory QA or shall reference documents which outline the QA procedures. The laboratory shall successfully analyze proficiency samples. The site-specific work plan, QA plans, and the results of the proficiency test are submitted to the NCR who will evaluate this information. The QA plans, the proficiency test results, and a draft work plan shall be received and evaluated by the NCR prior to scheduling a laboratory inspection. Based on the results of these evaluations and the inspection, the laboratory and engineering firm may be required to revise their QA plans, to retest a proficiency sample(s), to revise the work plan, or to prepare and implement a corrective action plan addressing deficiencies cited during the inspection.

Approval to begin work on samples is based on a combination of satisfactory QA plans and a site-specific work plan, satisfactory results of proficiency testing, and acceptable laboratory inspection. Approval may be granted to perform all or part of the methods required for a study.

2.1.2 Laboratory Reapproval

If a laboratory is requested to analyze samples from a second site, the NCR will evaluate the similarity between the analysis from the first and the second sites. The past performance of the laboratory and the time elapsed from previous sample analysis determine the steps the laboratory must follow to be reapproved. If a laboratory has performed well in the past, if the methods in the first and second work plan are similar, and if it has been less than a year since the first approval, the engineering contractor may only need to submit the site work plan, and work may proceed. If the laboratory's past performance was satisfactory but it has been longer than a year since a performance sample was analyzed, the laboratory must successfully analyze a new performance sample.

Any changes in personnel or general laboratory QA must be submitted to the NCR prior to receiving approval to begin the next site.

Figure 2.2 shows a flow diagram of the reapproval process.

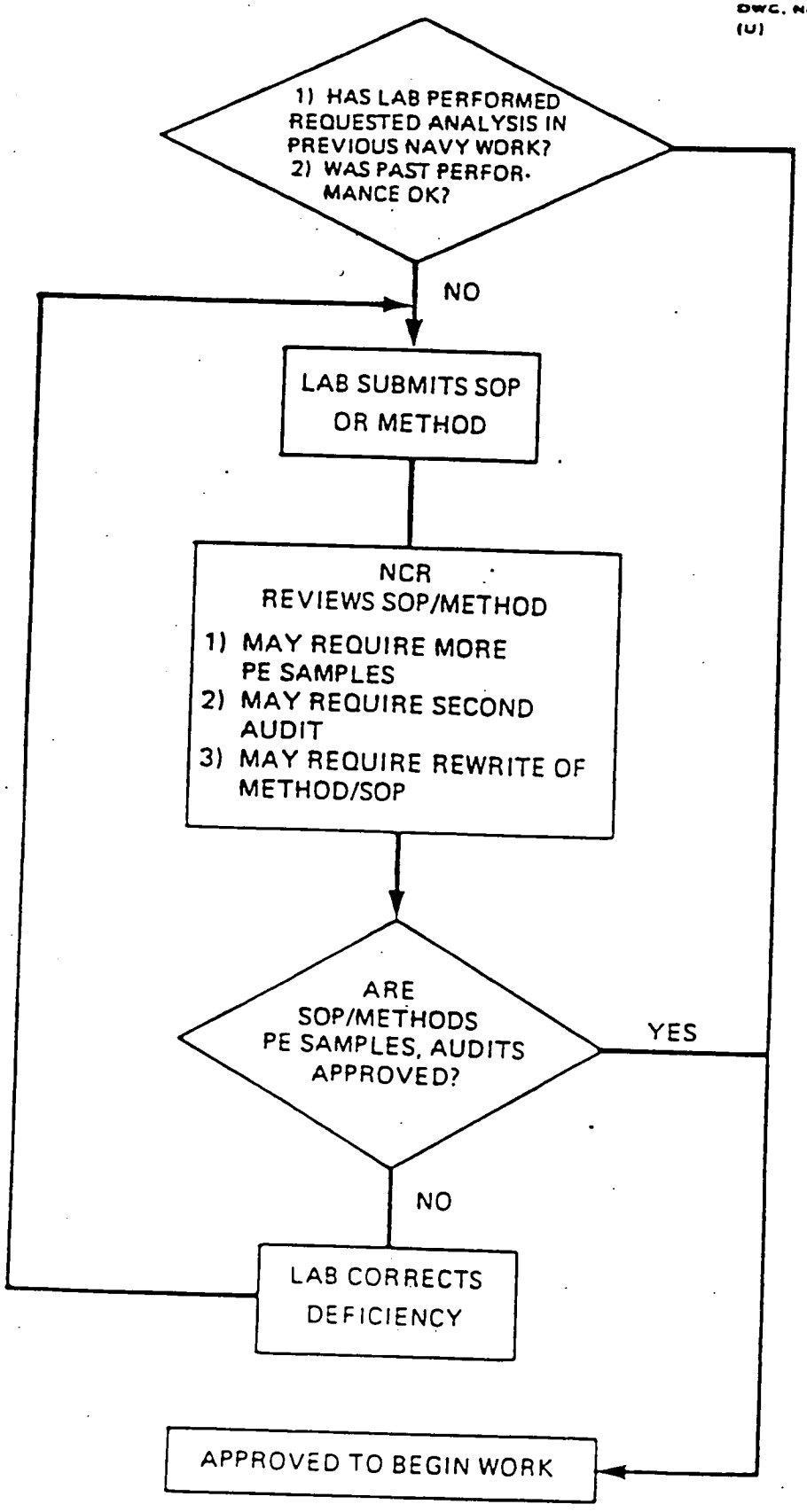

Fig. 2.2. Laboratory Reapproval Process

3. SITE-SPECIFIC QC REQUIREMENTS

The following are the requirements for the site-specific QC section to be included in the site-specific work plan or to be presented as a separate QC document.

3.1 CONTENTS OF SITE-SPECIFIC QC SECTION

1. The laboratory must be identified along with all other subcontractors.

2. Any pertinent state environmental or EPA federal/regional requirements shall be presented. This includes specific procedures or clean-up levels.

3. References must be made to the appropriate corporate or laboratory QA plans which contain pertinent information.

4. A discussion of COC and shipping practices must be provided.

5. Tables of the following shall be included.

   - Analytical methods and numbers of samples of each matrix to be collected at each site.

   - List of analytes to be identified and quantitated.

   - List of holding times, preservatives, amount of sample required, and container requirements.

   - List of the number, type, and matrix of field and laboratory QC samples by site. This includes trip blanks, equipment rinsates, field blanks, field duplicates, laboratory method blanks, laboratory matrix spikes and duplicates.

   - List of sample volume and bottles vs method.

6. All site-specific field sampling procedures which are not included in any corporate QA plan shall be presented.

7. Decontamination procedures for both drilling and sampling equipment shall be described.

8. Data quality objectives shall be discussed. This shall include precision, accuracy, and completeness required for acceptable data.

3.2 STATE AND REGION REQUIREMENTS FOR SITE QC PLAN

In addition to, or in place of, the requirements in this document, those requirements specific to the state or EPA region applicable to the

site shall be considered. Any state- or region-specific requests must be addressed in the site work plan.

3.3 SAMPLING DESIGN

Every site is unique in its own way. To this end, a sampling rationale shall be included with the work plan. The rationale should define and explain thoroughly the sampling statistics, the equipment involved, and anticipating data to be gained by this proposed methodology.

3.4 PRESERVATIVES

After samples have been taken, they shall be sent to the laboratory for analysis within 24 h after collection to ensure that the most reliable and accurate answers will be obtained as a result of the analysis. The holding time begins from the date of collection in the field. Preservatives shall be added in the field. Tables 3.1, 3.2, and 3.3 present the holding times, type of containers, and preservatives to be used. A table corresponding to each of the three different methods such as those from the *Federal Register; SW-846* 3rd ed.; and CLP is presented. The site-specific plan shall outline which preservatives will be used, and it shall be based on these tables. Freezing of samples shall not be permitted.

3.5 SAMPLE CONTAINER CLEANING PROCEDURES

In general, glass bottles with Teflon lids are used for organic samples, while polypropylene is used for metals and other inorganics. The following specifies the bottle cleaning required. If precleaned bottles are purchased, this must be noted in the work or field QA plan and approved by the NCR. If precleaned bottles are used, a certificate indicating that the bottles are analyte free must be provided.

3.5.1 Cleaning Procedure for Glass Bottles

1. Wash glass bottles, Teflon liners, and caps in hot tap water with laboratory-grade nonphosphate detergent.

2. Rinse three times with tap water.

3. Rinse with 1:1 nitric acid (metals-grade), American Society for Testing Materials (ASTM) Type I deionized water.

4. Rinse three times with ASTM Type I deionized water.

5. Rinse with pesticide-grade methylene chloride using 20 mL for 1/2-gal container and 5 mL for 4- and 8-oz containers.

6. Oven dry at 125°C. Allow to cool to room temperature in an enclosed contaminant-free environment.

Table 3.1 Required containers, preservation techniques, and holding times (40 CFR, Part 136, July 1, 1987)

[illegible]	[illegible]	[illegible]	[illegible]
Table IA—[illegible]			
1-4 [illegible]	P, G	Cool, 4°C, 0.008% [illegible]	[illegible]
5 [illegible]	P, G	[illegible]	Do
Table IB—[illegible]			
1 [illegible]	P, G	Cool, 4°C	14 days
2 [illegible]	P, G	[illegible]	Do
4 [illegible]	P, G	Cool, 4°C, [illegible]	28 days
9 [illegible]	P, G	Cool, 4°C	48 hours
11 [illegible]	P, G	[illegible]	28 days
14 [illegible]	P, G	Cool, 4°C	48 hours
15 [illegible]	P, G	Cool, 4°C, [illegible]	28 days
16 [illegible]	P, G	[illegible]	Do
17 [illegible]	P, G	[illegible]	[illegible]
21 [illegible]	P, G	Cool, 4°C	48 hours
23-24 [illegible]	P, G	Cool, 4°C, [illegible] pH > 12, 0.6g [illegible]	14 days
25 [illegible]	P	[illegible]	28 days
27 [illegible]	P, G	[illegible]	6 months
28 [illegible]	P, G	[illegible]	[illegible]
31, 43 [illegible]	P, G	Cool, 4°C, [illegible]	28 days
[illegible]			
18 [illegible] VI	P, G	Cool, 4°C	24 hours
35 [illegible]	P, G	[illegible]	28 days
3, 5-8, 10, 12, 13, 19, 20, 22, 26, 29, 30, 32-34, 36, 37, 45, 47, 51, 52, 58-60, 62, 63, 70-72, 74, 75 [illegible]	P, G	Do	6 months
38 [illegible]	P, G	Cool, 4°C	48 hours
39 [illegible]	P, G	Cool, 4°C [illegible]	28 days
40 [illegible]	P, G	Cool, 4°C	48 hours
41 [illegible]	G	Cool, 4°C, [illegible]	28 days
42 [illegible]	P, G	Cool, 4°C, HCl or [illegible]	28 days
44 [illegible]	P, G	[illegible]	Do
46 [illegible]	G [illegible]	[illegible], Cool 4°C	48 hours
47 [illegible]	[illegible]	[illegible]	[illegible]
48 [illegible]	[illegible]	[illegible]	[illegible]
49 [illegible]	G [illegible]	Cool 4°C [illegible]	28 days
50 [illegible]	G	Cool 4°C	48 hours
53 [illegible]	P, G	Cool 4°C [illegible]	28 days
54 [illegible]	P, G	Cool 4°C	7 days
55 [illegible]	P, G	Do	48 hours
56 [illegible]	P, G	Do	7 days
57 [illegible]	P, G	Do	48 hours
61 [illegible]	P	Do	7 days
64 [illegible]	P, G	Do	28 days
65 [illegible]	P, G	Do	Do
66 [illegible]	P, G	Do	Do
		Cool 4°C [illegible] pH > 9	7 days
67 [illegible]	P, G	[illegible]	[illegible]
68 [illegible]	P, G	Cool 4°C	48 hours
69 [illegible]	P, G	[illegible]	[illegible]
73 [illegible]	P, G	Cool 4°C	48 hours
Table IC—[illegible]			
13, 18-20, 22, 24-28, 34-37, 39-43, 45-47, 56, 66, 88, 89, 92-95, 97 [illegible]	G [illegible]	Cool 4°C 0.008% [illegible]	14 days
6, 57, 90 [illegible]	Do	Cool 4°C 0.008% [illegible]	Do
3, 4 [illegible]	Do	Cool 4°C 0.008% [illegible]	Do
23, 30, 44, 49, 53, 67, 70, 71, 83, 85, 96 [illegible]	G [illegible]	Cool 4°C 0.008% [illegible]	7 days until extraction, 40 days after extraction
7, 38 [illegible]	Do	[illegible]	7 days until extraction
14, 17, 48, 50-52 [illegible]	Do	Cool 4°C	7 days until extraction, 40 days after extraction
72-74 [illegible]	Do	Cool 4°C [illegible] 0.008% [illegible]	Do
76-82 PCBs [illegible]	Do	Cool 4°C	Do
54, 55, 65, 69 [illegible]	Do	Cool 4°C 0.008% [illegible]	Do
1, 2, 5, 8-12, 32, 33, 58, 59, 64, 68, 84, 86 [illegible]	Do	Do	Do
15, 16, 21, 31, 75 [illegible]	Do	Cool 4°C 0.008% [illegible]	Do
29, 35-37, 60-63, 91 [illegible]	Do	Cool 4°C	Do
87 TCDD	Do	Cool 4°C 0.008% [illegible]	Do
Table ID—[illegible]			
1-70 [illegible]	Do	Cool 4°C pH 5-9	Do
Table IE—[illegible]			
1-5 [illegible]	P, G	[illegible]	6 months

[illegible]

Table 7.1. List of approved biological test procedures (40 CFR, Part 136, July 1, 1987)

Parameter and units	Method [1]	Reference (Method number or Page)			
		EPA [2]	Standard Methods 15th Ed	ASTM	USG
Bacteria					
1 Coliform (fecal) number per 100 ml	MPN, 5 tube, 3 dilution, or, membrane filter (MF) [4], single step	p. 132	908C		
2 Coliform (fecal) in presence of chlorine number per 100 ml	MPN, 5 tube, 3 dilution	p. 124	908C		
3 Coliform (total), number per 100 ml	MPN, 5 tube, 3 dilution, or, MF [4] single step or two step	p.132	908C		8-0055-
		p 114	906A		
4 Coliform (total) in presence of chlorine, number per 100 ml	MPN, 5 tube, dilution, or MF [4] with enrichment	p 108	906A		8-0025-
		p 114	906A		
5 Fecal streptococci, number per 100 ml	MPN, 5 tube, 3 dilution, MF [4], or, plate count	p. 111	906 (A-A.5c)		
		p 136	910A		
		p 143	910B	00055-77 [6]	
			910C		

Table 1A notes

[1] The method must be specified when results are reported

[2] "Microbiological Methods for Monitoring the Environment, Water and Wastes, 1978", EPA-600/8-78-017, U.S. Environmental Protection Agency

[3] Greeson, P.E., et al, Methods for Collection and Analysis of Aquatic Biological and Microbiological Samples, "U.S. Geological Survey, Techniques of Water-Resources Book 5, Chapter A4, Laboratory Analysis, 1977.

[4] 0.45 um membrane filter or other pore size certified by the manufacturer to fully retain organisms to be cultivated, and free of extractables which could interfere with their growth environment

[5] Approved only if dissolution of the KF Streptococcus Agar (Section 5.1, USGS Method 8-0055-77) is made in a boiling water bath to avoid scorching of the medium

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Table 3.3 Preservatives and holding times for EPA-document SW-846 (3rd ed.)

Parameter	Container	Preservative	Holding Time Soil	Holding Time Water
Volatiles by GC/MS, and GC	Water - 40-mL glass vial with Teflon-lined septa Soil-glass with Teflon-lined septa	Cool, 4°C	14 days	14 days
PCB/ pesticides	G, Teflon-lined lid	Cool, 4°C	Extract within 7 days, analyze 40 days	Extract within 7 days, analyze 40 days
Extractable organics	G, Teflon-lined lid	Cool, 4°C	Extract within 7 days, analyze 40 days	Extract within 7 days, analyze 40 days
Metals	P, G	HNO_3 to pH<2	6 months	6 months
Mercury	P, G	HNO_3 to pH<2	28 days	28 days
Cyanide	P, G	NaOH to pH>12 Cool 4°C add 0.6 g ascorbic acid if residual chlorine present	14 days	14 days
Chromium	P, G	HNO_3 to pH<2	24 h	24 h

3.5.2. Cleaning Procedure for Bottles Used for Volatile Organics (40-mL Glass)

1. Wash glass vials, Teflon-backed septa, Teflon liners, and caps in hot tap water using laboratory-grade nonphosphate detergent.

2. Rinse three times with tap water.

3. Rinse three times with ASTM Type I deionized water.

4. Oven dry vials, septa, and liners at 125°C.

5. Allow vials, septa, and liners to cool to room temperature in an enclosed contaminant-free environment.

6. Seal 40-mL vials with septa (Teflon side down) and cap.

7. Store in contaminant-free area.

3.5.3. Cleaning Procedure for Polyethylene Bottles

1. Wash polyethylene bottles and caps in hot tap water with laboratory-grad nonphosphate detergent.

2. Rinse with 1:1 nitric acid (metals-grade), ASTM deionized water).

3. Rinse three times with ASTM Type I deionized water.

4. Invert and air dry in contaminant-free environment.

3.5.4. All Bottles Should Be

1. Capped and labeled with sample numbers and packed in cooler or box.

2. Stored in contaminant-free area.

3.6 ORGANIZATION AND PERSONNEL

All management personnel responsible for performing field sampling or analytical work shall be listed along with their job assignment and years of experience in performing this type of work. Any education and training related to the tasks performed for this project shall also be listed.

3.7 FIELD QC SAMPLES

Although the number of QC samples changes, the types of field QC samples remain the same regardless of the level of QC implemented. Table 3.4 lists the percentage of field QC samples per level per sample matrix. A sampling event is considered to be from the time the sampling personnel arrive at the site until these personnel leave for more than a day. An example of two events would occur if sampling personnel went to a site for three weeks, drilled borings, and put groundwater wells in place. During this visit, soil and water samples were collected. The sampling crew left the site for two months, thus concluding the first sampling event. The crew later returned to collect another set of groundwater samples over a three-day period. The second visit would constitute the second sampling event.

Table 3.4. Field QC samples per sampling event

Type of Sample	Level C		Level D		Level E	
	Metal	Organic	Metal	Organic	Metal	Organic
Trip blank (for volatiles only)	NA[1]	1/cooler	NA[1]	1/cooler	NA[1]	1/cooler
Equipment rinsate[2]	1/day	1/day	1/day	1/day	1/day	1/day
Field blank	1/source/event for all levels and all analytes					
Field duplicates[3]	10%	10%	10%	10%	5%	5%
Referee duplicate[3]						

[1]NA - Not applicable.

[2]Samples are collected daily; however, only samples from every other day are analyzed. Other samples are held and analyzed only if evidence of contamination exists.

[3]The duplicate must be taken from the same sample which will become the laboratory matrix/spike duplicate for organics or for the sample used as a duplicate in inorganic analysis.

The following information defines and explains the blanks, duplicates, and referee samples.

1. Trip Blanks

Trip blanks are defined as samples which originate from analyte-free water taken from the laboratory to the sampling site and returned to the laboratory with the volatile organic (VOA) samples. One trip blank should accompany each cooler containing VOAs, should be stored at the laboratory with the samples, and analyzed by the laboratory. Trip blanks are only analyzed for VOAs.

2. Equipment Rinsates

Equipment rinsates are the final analyte-free water rinse from equipment cleaning collected daily during a sampling event. Initially, samples from every other day should be analyzed. If analytes pertinent to the project are found in the rinsate, the remaining samples must be analyzed. The results from the blanks will be used to flag or assess the levels of analytes in the samples. This comparison is made during data validation. The rinsates are analyzed for the same parameters as the related samples.

3. Field Blanks

Field blanks consist of the source water used in decontamination and steam cleaning. At a minimum, one field blank from each event and each source of water must be collected and analyzed for the same parameters as the related samples.

4. Field Duplicates/Splits

Duplicates or splits for soil samples are collected, homogenized, and split. All samples except VOAs are homogenized and split. Volatiles are not mixed, but select segments of soil are taken from the length of the core and placed in 40-mL glass vials. Cores may be sealed and shipped to the laboratory for subsampling if the project deems this appropriate. The duplicates for water samples should be collected simultaneously. Field duplicates should be collected at a frequency of 10% per sample matrix for Levels D and C. For Level E, the duplicates should be analyzed at a frequency of 5%. All the duplicates should be sent to the primary laboratory responsible for analysis. The same samples used for field duplicates shall be split by the laboratory and be used as the laboratory duplicate or matrix spike. This means that for the duplicate sample, there will be analyses of the normal sample, the field duplicate, and the laboratory matrix spike/duplicate.

5. Referee Duplicates

Duplicates/splits shall be sent to the referee QA laboratory if regulators (state or region) collect split samples or if a special problem occurs in sample analysis or collection. These duplicates/splits are collected and analyzed in addition to the field duplicates mentioned in the previous paragraph.

3.8 CHAIN OF CUSTODY

Samples, other than those collected for *in situ* field measurements or analyses, are identified by using a standard sample label which is attached to the sample container. The sample labels are sequentially numbered and are accountable. The following information shall be included on the sample label.

1. Site name.
2. Field identification or sample station number.
3. Date and time of sample collection.
4. Designation of the sample as a grab or composite.
5. Type of sample (matrix) and a brief description of the sampling location.
6. The signature of the sampler.
7. Sample preservation and preservative used.
8. The general types of analyses to be conducted.

If a sample is split with another party, sample labels with identical information shall be attached to each of the sample containers.

The COC record is used to record the custody of samples and shall accompany samples at all times. The following information shall be supplied to complete the COC record.

1. Project name.
2. Signature of samplers.
3. Sampling station number or sample number, date and time of collection, grab or composite sample designation, and a brief description of the type of sample and sampling location.
4. Signatures of individuals involved in sample transfer (i.e., relinquishing and accepting samples). Individuals receiving the samples shall sign, date, and note the time that they received the samples on the form.
5. Matrix.

Sample analysis request sheets serve as official communication to the laboratory of the particular analyses required for each sample and provide further evidence that the COC is complete.

COC records initiated in the field shall be placed in a plastic cover and taped to the inside of the shipping container used for sample transport from the field to the laboratory.

3.9 SHIPPING REQUIREMENTS

Shipping containers shall be secured using nylon strapping tape and custody seals to ensure that samples have not been disturbed during transport. The custody seals shall be placed on the containers so they cannot be opened without breaking the seal.

Samples which must be kept at 4°C shall be shipped in insulated containers with either freezer forms or ice. If ice is used, it shall be placed in a container so that the water will not fill the cooler as the ice melts. The samples shall be shipped within 24 h of collection to allow the laboratory to meet holding times. The Department of Transportation regulations shall be used for packaging, quantities of shipment, and the way samples are sent. Each subcontractor responsible for sampling shall become familiar with the regulations.

Copies of the signed COC forms shall be delivered with the data packages. The originals shall remain on file with the contractor or with the laboratory.

3.10 SAMPLE RECEIPT

Upon receipt, the laboratory shall sign and keep copies of the air bill. The COC shall be signed. The temperature of the cooler shall be

measured and documented. The condition of the samples shall be documented. If any breakage or discrepancy arises between COC, sample labels, and requested analysis, the sample custodian will notify the engineering subcontractor. The pH of incoming samples shall be checked and documented upon receipt. Any discrepancy or improper preservation shall be noted by the laboratory as an out-of-control event and shall be documented on an out-of-control form with the corrective action taken. The out-of-control form shall be signed and dated by the custodian and any other person responsible for corrective action.

4. LABORATORY QA PLAN REQUIREMENTS

An essential step in the sequence of events leading to Navy approval of contract laboratories is the preparation and acceptance of a QA plan for each laboratory.

The contents and format of an acceptable plan are described below. If the laboratory has a general QA plan in place, this should be sent for review. In this case, a site-specific QA plan may not be needed. Any deviations or additions to the normal laboratory QA should be documented in the site-specific work plan.

4.1 PURPOSE AND SCOPE

The QA plan is a statement of the laboratory's approach to ensuring that quality data are generated from the analysis of Navy samples. In the context of laboratory approval, the plan provides a basis for evaluating a laboratory's QC procedures. This evaluation includes a critical review of the plan and verification of the laboratory's adherence to the plan through inspection.

4.2 ORGANIZATION AND CONTENTS OF PLAN

The items listed below may be presented in any order that the laboratory desires; however, the list includes the items that are required in the QA plan.

1. Title Page with Provision for Signatures
2. Table of Contents
3. Organization and Personnel
4. Personnel Training
5. Sample-Handling Practices and COC
6. Material Procurement and Control
7. Facilities and Equipment
8. Equipment Maintenance
9. Analytical Procedures
10. Calibration
11. Limits of Detection
12. Analysis of QC Samples and Documentation
13. Out-of-Control Events and Corrective Action
14. Data Evaluation and Data Reduction
15. Holding Times and Preservatives
16. Internal Laboratory Audits and Approvals from Other Agencies
17. Document Control
18. QA Reports to Management
19. Accuracy, Precision, and Completness

1. Title Page with Provisions for Signature

   A title page with provision of approval signatures and date of revision shall be provided.

2. Table of Contents

   A table of contents shall be provided.

3. Laboratory Organization and Personnel

   This section provides an overview of the laboratory organization as it relates to implementation of the QC program. The roles, responsibilities, and authority of key laboratory personnel are described with emphasis on the authority given the LQAC with regard to QC monitoring, reporting, and corrective action.

   An appendix shall contain a list of all the personnel, their assignments and responsibilities, degrees of education, and the years of applicable experience.

4. Personnel Training

   The plan shall address how personnel are trained in laboratory methods, in QC, and in safety policies.

5. Sample-Handling Practices and COC

   This section shall include tracking of samples through the laboratory, receipt of samples, verification of preservation, login of samples, and COC. Sample storage and disposal shall also be included. Preparation of bottles and glassware washing shall also be included.

6. Material Procurement and Control

   This section shall include a description of procedures for purchasing materials, quality inspection prior to use in sample analysis, chemical and standard inventory, solvent storage policies, and laboratory waste disposal.

7. Facilities and Equipment

   A list of basic types of equipment, year of purchase, and general description of the facility assures that the laboratory is large enough to handle the sample load expected and that the equipment is capable of performing the analysis.

8. Equipment Maintenance

   This section shall include general information as to who performs both major, preventive, and day-to-day maintenance and how it is documented.

9. Analytical Procedures

This section shall contain a list of all procedures that the laboratory offers (by method number and matrix) in the event that future work may require analyses not specified in the SOW.

Any method variances must be reported, and any documentation from EPA for approvals-of-method variances shall be presented to assure that they are known prior to sample analysis.

The laboratory policy and implementation procedures shall emphasize that methods are available to the analyst.

10. Calibration

This section shall include calibration procedures by instrument type, calibration frequency, reference standards used, calibration acceptance criteria, and calibration documentations procedures. Calibration applies to both instruments such as gas and liquid chromatography, GC/MS, inductively coupled plasma (ICP), atomic absorption (AA), infrared and ultraviolet spectroscopy, and wet chemical methods.

The method for assuring that balances, refrigerators, and ovens are accurate and how these pieces of equipment are checked must be outlined. Balances and ovens must be checked prior to use. Balances must also be checked by an outside company annually.

11. Limits of Detection

The laboratory shall indicate what the typical method-detection limits are for water, soil, and any other matrix commonly analyzed by the laboratory, with the understanding that this varies with the sample matrix. The procedures for determining the limits of detection for each type of method and the frequency of detection limit verification shall be outlined.

12. Analysis of QC Samples and Documentation

This section shall summarize the QC procedures and documentation to be used in the day-to-day operation of the laboratory. The discussion shall emphasize the following:

- analysis of field, method, and reagent blanks;
- analysis of duplicates, spiked samples, spiked laboratory blanks, and reference or control standards such as EPA check standards;
- the criteria used to establish warning and action limits for the above types of QC samples;

- documentation and examples of control data and control charts (see Sect. 4.4) for explanation of control charts and their usage;
- the frequency of blanks and other QC samples and controls;
- how the data from the QC samples are reported and reviewed;
- who reviews and makes decisions from the QC data;
- details of how requirements of minimum control program in Sect. 8.2 will be met; and
- verification of calibration.

13. Out-of-Control Events and Corrective Action

This section shall contain a definition as to the types of out-of-control occurrences, how these occurrences are documented, and who is responsible for correction and documentation. It is recognized that several out-of-control events occur. Four examples are given.

- Observations Corrected at the Bench - If the calibration of an instrument is not linear, the analyst may find this and correct it prior to continuing to analyze samples. The laboratory may document this and note that the corrective action was to recalibrate and that no samples were affected, as none were analyzed prior to calibration.
- Corrective Actions Taken by Supervisor - A matrix spike recovery is out of control and the laboratory supervisor finds this after the samples for the day have been analyzed. The supervisor shall document that the laboratory blank spiked with surrogates or standards was in control and that other sample spikes were in control; therefore, no reanalysis of the sample is required.
- Corrective Actions at the Receiving Level - If the sample is broken, the analyst may note this and document whether or not more sample is available. If no more sample is available, the customer shall be notified and the decision documented.
- Statistical Out-of-Control Events - If a control chart is being monitored and the measured parameter exceeds the 99% confidence limit then explanation shall be documented as to when the parameter exceeded statistical limits.
  - Procedures shall be outlined as to what corrective action is taken if an out-of-control event occurs and how it is documented and used to improve laboratory performance. The documentation shall be easily used by all personnel and shall be part of routine laboratory procedure.

- Procedures for assuring that results for samples processed during out-of-control conditions are not reported shall be outlined.

- The conditions necessary to reestablish control and criteria for assuring the system is operating properly.

14. Data Evaluation and Data Reduction

A discussion of data evolution procedures for each analytical method as well as for an entire data set shall be included. The process of certification of reviewed data shall be outlined with an explanation of how suspect data are flagged if they are suspect but still reported.

15. Holding Times and Preservatives

The document shall include laboratory policy for meeting holding times for sample analysis and how it is assured that these are met. The sample storage requirements, holding times, and preservatives specified in Tables 3.1, 3.2 and 3.3 are minimal criteria for Navy approval.

16. Internal Laboratory Audits and Approvals from Other Agencies

A listing of approvals from other agencies and states gives an indication of the general quality and type of laboratory experience the organization has. If the laboratory performs self-audits, the frequency and method of documentation shall be outlined.

17. Document Control

The QA plan shall outline the flow of documents containing COC and data. The plan shall explain how documents are checked, signed, and filed.

18. QA Reports to Management

The plan shall include the frequency and information of management QA reports.

19. Accuracy, Precision, and Completeness

The plan shall include the laboratory's definition and method of evaluating the precision, accuracy, and completeness of measurement parameters and of evaluating data sets.

4.3 CONTROL SAMPLES

Control samples are those samples containing known concentrations of analytes that are introduced into a run of environmental samples to

monitor the performance of the analytical system. Control samples involving duplicates, blanks, analytical standards, reference materials, and spikes can be used in different phases of the overall analysis from sampling through storage, transportation, and preparation to the analytical method itself. The choice of types of controls relates to the analysis phase(s) to be controlled and the information (e.g., precision, accuracy, interferences, recovery) to be developed.

The QA plan describes generally how and where such control mechanisms are used by the laboratory. Control materials may be purchased from commercial sources, the National Bureau of Standards, or the EPA. A brief description of each control sample (or set of samples) used shall be provided in the MPR, subsequent to its introduction, and shall cover the following items.

1. Where the control samples are made.

2. How they are made.

3. How many are made and with what frequency.

4. How they are used.

   - Physically (e.g., placed in the sample tray along with 14 environmental samples just before the samples enter the processing stream).

   - Analytically (e.g., used to determine the recovery factor of the procedures; used to check for interferences).

5. Frequency of analysis of control sample.

4.4 CONTROL CHARTS

Control charts provide a useful tool in assessing QC efforts and improving processes through graphic displays of a parameter(s) and its variability over time. The parameter plotted on the chart is usually related to control sample testing--either directly in terms of concentrations or indirectly in terms of derived information such as means of concentrations, ranges of concentrations, percent recovery of spikes, relative percent differences based on duplicate results, or slopes of least-squares data fits.

The laboratory should include in its QA plan, as required in Sect. 4.2, a brief description of the basic methodology used in control charting, covering such considerations as the following.

1. Verification that the methods are valid and working properly prior to beginning control charts.

2. Number of control samples per run.

3. Number of runs analyzed.

4. Parameters to be plotted against time and the general formulae for developing these parameters.

5. Statistical/mathematical basis for assigning warning and rejection limits on the charts in terms of, for example, standard deviation.

6. Types of shifts, trends, or biases that may typically be revealed by these charts.

4.4.1 Method Blank/Spike Control Program

Controls are required for only the methods and analytes pertinent to the program. The laboratory shall employ a measurement-control program which, as a minimum, consists of monitoring the results of laboratory preparation and analysis of control samples using statistical control charts. The basis of this program is to demonstrate that the laboratory method for sample preparation and analysis is working properly. This minimum program consists of using the laboratory's distilled and/or deionized water and spiking it with known compounds or elements. By plotting the results of the method blank spike on control charts, a true picture of the actual process of sample analysis is obtained with fewer problems from matrix effects and sample nonhomogeneity. This information, used in conjunction with matrix spike recoveries, can aid in determining whether an out-of-control condition is due to laboratory problems or matrix problems. Therefore, one batch of control material is the spiked laboratory blank water. The second batch of control material is a soil or sand. This soil can be pulverized and homogenized. If the soil used is known to contain some of the analytes of interest, then no spiking may be required. Additional spiking may be done to an aliquot of control soil just prior to sample preparation. The method blank/spike water (laboratory water) should be analyzed when water samples are analyzed and the method blank/spike soil analyzed alongside soil or waste samples.

The analytes selected for spiking should be representative of the compound class for the organics. It is suggested that the surrogates used for volatiles and base/neutral/acids (BNAs) analyses be used as control analytes for the GC/MS methods. At least two pesticides should be used when pesticide methods are performed and one polychlorinated biphenyl (PCB) when PCBs are analyzed. For wet chemical methods, a single spike of an appropriate control for each method may be used. As an example for cyanide, a control of sodium cyanide from a source other than that used for calibration may be spiked into water and analyzed alongside the water samples. For the metals, it is suggested that at least three of the metals typically analyzed by ICP be monitored and that each element analyzed by furnace or flame atomic absorption be monitored.

4.4.2 Control Sample Quality

The laboratory QA plan shall describe the steps which will be taken to ensure and verify the quality of the two types of control samples of Sect. 4.4.1. The QA plan shall address the following concerns pertaining to the control batches.

1. How the batch will be selected.
2. Shelf life of control batch.
3. Under what conditions the batch will be stored.
4. How the batch will be homogenized.
5. How and when the individual samples will be taken.
6. How and when the sample will be spiked.
7. How the batch will be replaced as it is depleted.
8. How the control charts will be affected by changes in batches.

The QA plan shall address the following concerns pertaining to the spikes.

1. What compound/element will be used to spike.
2. How the spike material will be selected.
3. Target concentration of spiking compound/element.
4. How long the spike is expected to last.
5. Under what conditions the spike will be stored.
6. How the spike will be homogenized.
7. How and when the individual samples will be taken.
8. How the spike will be replaced as it is depleted.
9. How the control charts will be affected by changes in spikes.

4.4.3 Minimum Statistical Control Charting

As a minimum, the laboratory shall run two control charts for each analyte listed in Table 4.1. These charts shall monitor the laboratory measurements obtained from individually spiked water samples and individually spiked soil samples.

Each control chart shall consist of a center line, two warning limits, and two control limits. The control chart parameters should be calculated according to the formulae provided in Table 4.2. A minimum of 20 points/chart shall be obtained prior to the initial attempt to establish the control chart parameters.

If the laboratory does not have 20 points to use in setting control chart limits, the recommended EPA recoveries for the method will be used until such time as 20 points are attained.

4.4.4 Minimum Criteria for an Out-of-Control Condition

A laboratory process for a particular analyte should be considered out of statistical control whenever, as a minimum, any one of the following conditions is demonstrated by a control chart monitoring that analyte.

1. Any one point is outside of the control limits.
2. Any three consecutive points are outside the warning limits.
3. Any eight consecutive points are on the same side of the centerline.
4. Any six consecutive points are such that each point is larger (smaller) than its immediate predecessor.
5. Any obvious cyclic pattern is seen in the points.

Table 4.1. Typical number of analyses to be monitored through measurement control program

Number	Analyses
10	Metals by AA and ICP
1	Mercury
3	Volatiles
1	Wet chemicals
1	PCB
2	Pesticides
3	Base neutrals
3	Acids

4.4.5 Reactions to Out-of-Statistical-Control Conditions on Control Samples

The laboratory QA plan shall describe the steps which will be taken in the occurrence of an out-of-statistical-control condition from the control charts of Sect. 4.2.3. The steps should be similar to those requested in Sect. 4.6 but shall include those actions related to the quality and stability of the control batches, sampling, spiking, and handling of the control samples.

4.4.6 Administration of the Control Charts

The laboratory QA plan shall address the following aspects of administering the control charts of Sect. 4.4.2.

1. What types of laboratory activities the control charts will monitor.
2. How often control samples will be run.
3. How soon after results are obtained will charts be monitored.
4. Who is responsible for reading the charts.
5. How will changes in people, equipment, processes affect the charts.
6. How often and under what circumstances will limits be updated.

4.4.7 Statistical Quality of the Control Charts

The formulae for the control chart parameters given by Table 4.2 are those commonly accepted and used. They are based on normally distributed measurements and short-term variation. If these bases are inappropriate, the charts will not perform as desired. The charts will either falsely signal out-of-control warnings more frequently than usual, fail to detect existing out-of-control conditions as often as they ordinarily would, or both (for different types of out-of-control states). In order to correct any problems due to improperly fitting control charts, the laboratory may propose alternate methods for setting the control chart parameters for those analytes of Table 4.2. All such proposals

Table 4.2. Control chart formula for Water and Soil Control Batch Program

Definitions

Let X_1, X_2, X_3, ..., X_n ($n \geq 20$) represent the first n time-ordered determinations for an analyte of Table 4.2 from either the water or soil control batch program.

Then, define the following:

$\bar{X}$ = average = $(1/n)(X_1 + X_2 + \ldots + X_n)$,
$Ri = |X_i - X_{(i-1)}|$ $i = 2,3,\ldots,n$
R_2 = average moving range of two successive points,

$$= [1/(n-1)][\,|X_2 - X_1| + |X_3 - X_2| + \ldots + |X_n - X_{(n-1)}|\,] .$$

Control Chart Parameter Estimation

Parameter	Symbol	Formula
Centerline	CL	$\bar{X}$
Upper control limit	UCL	$\bar{X} + 3R_2/d_2$
Lower control limit	LCL	$\bar{X} - 3R_2/d_2$
Upper warning limit	UWL	$\bar{X} + 2R_2/d_2$
Lower Warning limit	LWL	$\bar{X} - 2R_2/d_2$

(d_2 = 1.128, factor from tables for control charting within n = 2, see American Society for Quality Control)

should include data and supportive statistical evidence. Possible alternate statistical approaches can include using nonparametric techniques, medians instead of averages for the centerlines, identifying sources of variation, using long-term variation instead of short-term variation in setting limits, and transformations of the data.

4.4.8 Example of Setting Control Limits

As an example of setting control chart parameters and a very brief introduction to interpretation of the chart, consider the following:

A sample is obtained from the batch of control soil which has been thoroughly mixed and is stored in a special atmospherically controlled location. It is carefully spiked with known amounts of the constituents of Table 4.2 and sent to sample preparation to be processed with a customer's solid waste samples. It is analyzed along with the other samples. It is subjected to the same types of treatment as the other samples in the batch. This scenario is repeated until 20 control samples have been analyzed.

The data are listed in Table 4.3. Also shown are calculations according to the formulae in Table 4.2. Figure 4.1 displays the results of the initial attempt at sizing the data to the control chart parameters. The point falling above the upper control limit was investigated. It was determined that the sample had received a double spiking and, thus, was deleted from the second iteration calculation of the chart parameters. Figure 4.2 shows the second fitting. This fit appears adequate, and the chart is approved by the LQAC authority. Had no explanation for the high result been found, the first calculations would have been used. The chart would have been placed under a probationary condition and its performance monitored with guarded caution.

4.5 OUT-OF-CONTROL EVENTS

The interpretation of control charts can reveal shifts, trends, biases, and conditions where parts of the analytical system are out-of-control. The contract laboratory should specify in the QA plan its criteria of defining an out-of-control condition related to the different zones on a control chart [e.g., data beyond the rejection limits, data in the zone(s) between the rejection and warning limits, and data inside the warning limits] and different patterns within these zones [e.g., number of consecutive data points on one side of the mean, number of consecutive data points in the middle zone number of monotically changing data points, obviously repetitive patterns (Garfield, 1984)].

The laboratory shall identify what actions will be taken when the warning and/or control limits are exceeded. Warning conditions may only require more frequent observations of a piece of equipment, while rejection conditions require shutting down an instrument.

Any incident that delays sample processing for a period of time, affects holding times, or delays work by more than two days should immediately be reported by phone to the NCR. The NCR should be informed as soon as the problem is solved and an explanation given as to the corrective action taken. An example of this type of event would be the breakdown of a GC/MS system used for VOAs which could not be repaired for several days. If the laboratory could not use another instrument in its laboratory, then provisions for another approved laboratory to analyze the samples would need to be made.

Table 4.3. Data and calculations for control chart example

Order I	Result X	Moving range $\lvert X_I - X_{I-1} \rvert$
1	12.25	
2	7.52	4.73
3	12.29	4.78
4	10.04	2.25
5	8.48	1.56
6	10.89	2.40
7	9.57	1.32
8	11.40	1.83
9	9.28	2.12
10	11.66	2.39
11	12.06	0.40
12	8.52	3.54
13	11.14	2.62
14	19.56	8.42
15	10.48	9.08
16	9.12	1.35
17	12.79	3.66
18	10.30	2.49
19	5.54	4.76
20	8.93	3.39
Sum	211.82	63.0

First calculations (Fig. 4.1)

Average = 211.82/20 = 10.591
Average moving range = 63.09/19 = 3.321

Centerline = 10.591
Upper control limit = 10.591 + 3 × 3.321/1.128 = 19.423
Lower control limit = 10.591 − 3 × 3.321/1.128 = 1.758
Upper warning limit = 10.591 + 2 × 3.321/1.128 = 16.479
Lower warning limit = 10.591 − 2 × 3.321/1.128 = 4.703

Second Iteration after Removing Point #14 (Fig. 4.2)

Average = 192.26/19 = 10.119
Average moving range = 46.26/18 = 2.570

Centerline = 10.119
Upper control limit = 10.119 + 3 × 2.570/1.128 = 16.954
Lower control limit = 10.119 − 3 × 2.570/1.128 = 3.284
Upper warning limit = 10.119 + 2 × 2.570/1.128 = 14.676
Lower warning limit = 10.119 − 2 × 2.570/1.128 = 5.562

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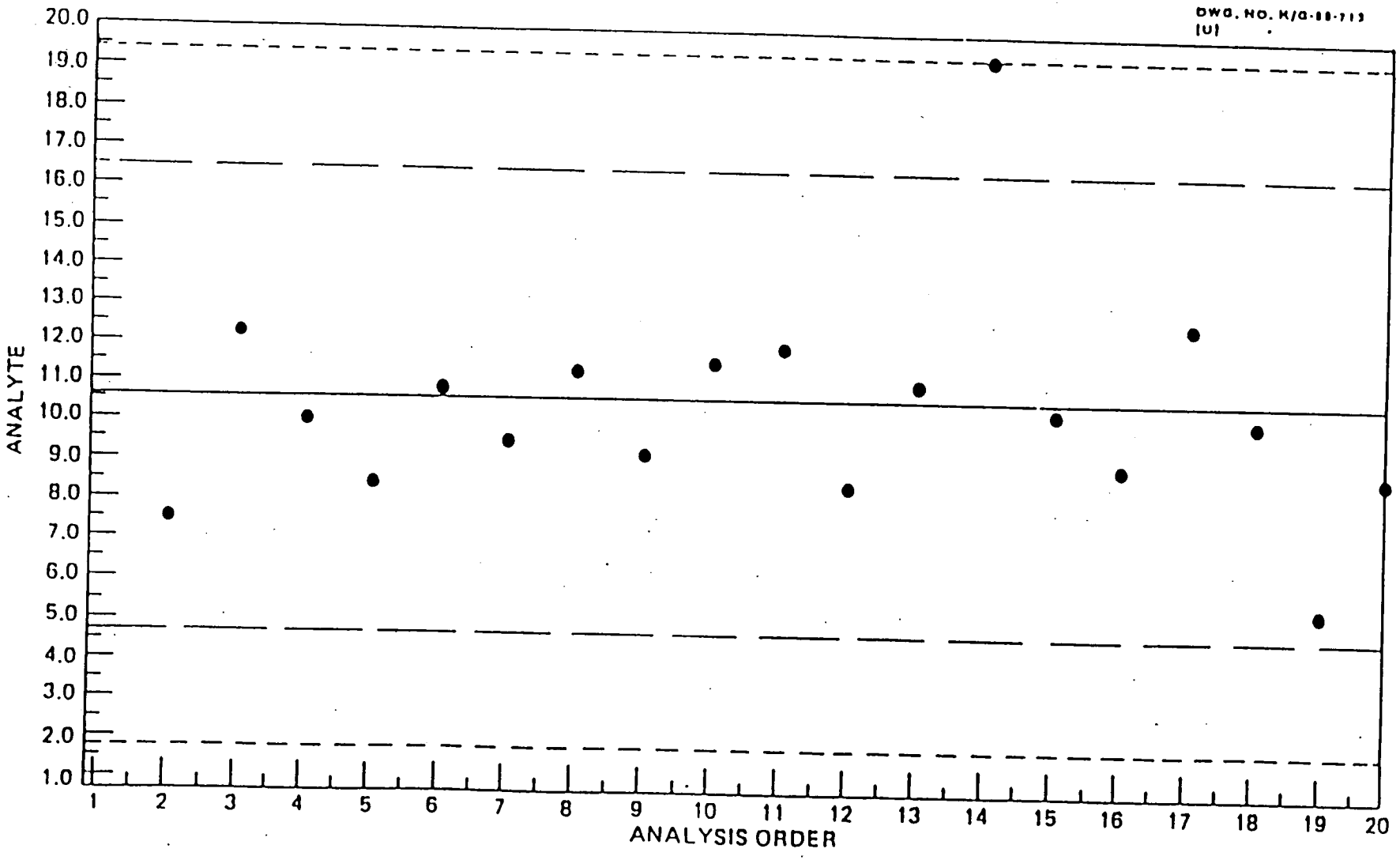

Fig. 4.1. Control Chart Example Data and Control Values of Table 4.3

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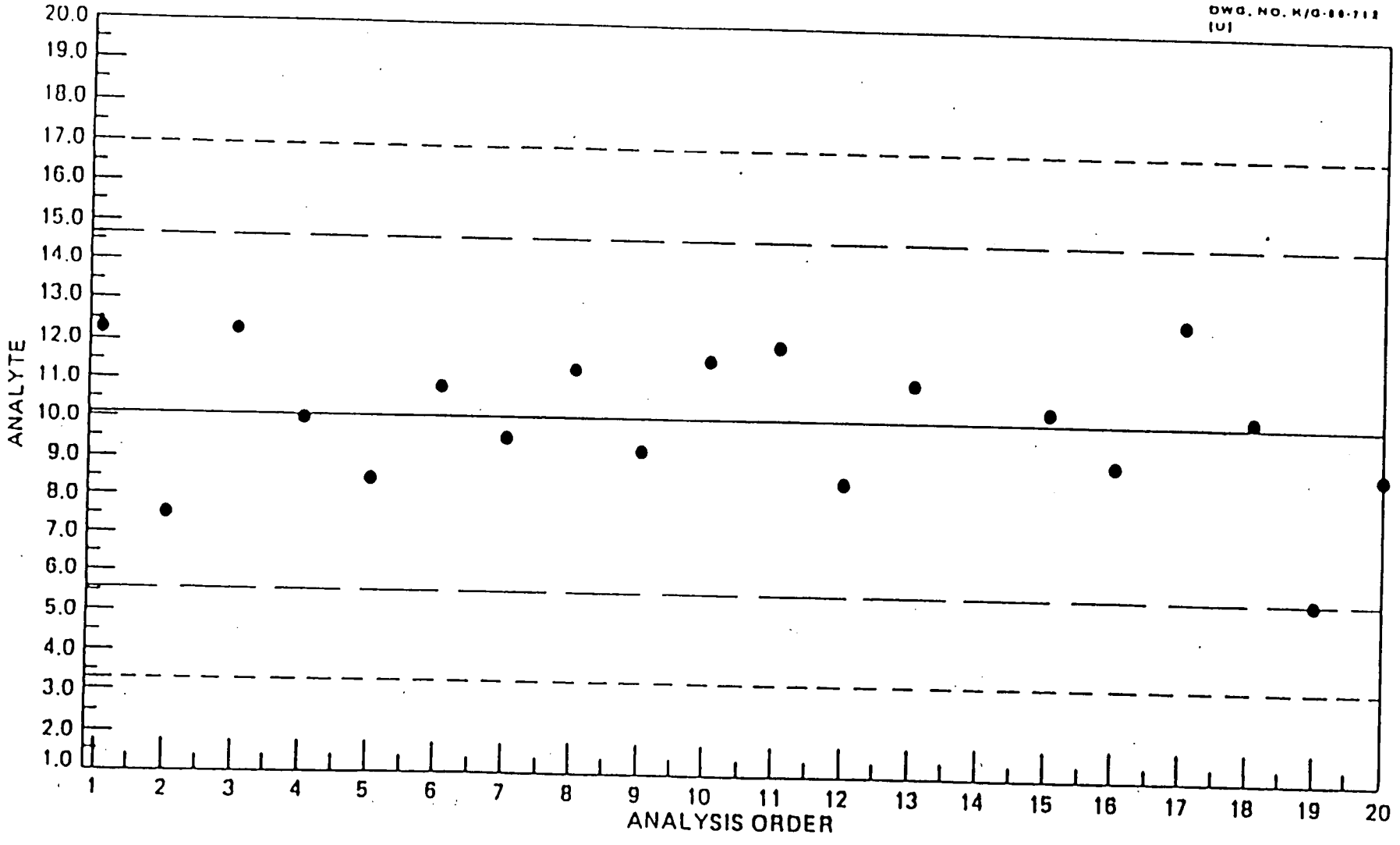

Fig. 4.2. Control Chart Example Data and Second Iteration Control Values of Table 4.3

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Many laboratories use forms to aid in rapidly reporting out-of-control events and corrective actions. One way to expedite the more routine out-of-control occurrences, which are frequently corrected by the analyst prior to running samples, is to list these on a form. For instance, a form for the GC/MS laboratory might list events such as continuing check standard outside limits, tune not met, and peak areas for the internal standard outside criteria. The analyst would check the occurrence, note that the item was corrected prior to sample analysis, initial, and date the form. If forms for out-of-control events are made specific to the group using these, time can be saved in documenting events and corrective actions (see Sect. 4.2 for examples of out-of-control events).

4.6 CORRECTIVE ACTION REPORT

For out-of-control incidents, it is essential to document the nature of the incident and the corrective actions taken to set the system back "in control." A corrective action report, to be signed by the laboratory director and the LQAC, should be reported in the MPR to the NCR and discuss the following topics.

1. Where - did the out-of-control incident occur (laboratory name, address, telephone number, section name)?

2. When - did the incident occur?

   - was it corrected?

3. Who - discovered the out-of-control incident?

   - verified the incident?

   - corrected the problem?

4. What - was the name of the test?

   - was the disposition of the test or control and/or instrument?

   - was the nature of the corrective action?

   - will be done to prevent the reoccurrence of the problem?

5. Why - did the incident happen (if scientific explanation is available)?

A copy of the subject control charts and other data describing the out-of-control conditions should be included in the corrective action report.

All out-of-control incident documentation and copies of the corrective action reports sent to the NCR should be

1. placed in the laboratory archive record for the sample(s) in question,
2. placed in the LQAC's file of incidents documentation, and
3. referenced and briefly described in the MPR.

5. PROFICIENCY TESTING

Prior to beginning analysis of field samples, each laboratory must analyze proficiency samples for chemical substances representative of those anticipated in environmental samples. The purpose of proficiency testing is to gage each laboratory's proficiency with samples which are designed to mimic field samples. A second benefit of performance samples is to provide a known material from a source outside the laboratory which can be used to evaluate the laboratory's performance.

5.1 SUBMITTING THE PROFICIENCY SAMPLES

Proficiency samples will be provided to the LQAC within ten working days of receipt of the site work plan. The samples may be soil or water samples or vials of concentrate. The laboratory will be directed as to any required sample reconstitution and the analytes to be determined. If analyses are to be subcontracted to a second laboratory, appropriate proficiency samples must be sent by the NCR to that laboratory as well.

5.2 RESULTS OF PROFICIENCY SAMPLES

Results of proficiency sample analyses are to be received by the NCR within 20 working days after receipt of the samples. The NCR must have the data at least five working days prior to inspection in order to properly evaluate the data. If performance samples are to be subcontracted to a second laboratory, the data report should be sent directly to the primary laboratory by the subcontract laboratory. The entire performance data package is then submitted to the NCR. QC data such as blanks, spikes, EPA controls, daily calibration check standards, sample chromatograms, mass spectra of identified compounds and raw absorbance data for metals shall be provided.

5.3 EVALUATION OF PROFICIENCY SAMPLE RESULTS

The NCR will compare the laboratory's evaluation of proficiency sample results to peer group proficiency sample results.

Performance will always be acceptable if the laboratory results are not statistically different from the peer group results, at 95% confidence, and no procedural problems are found during the laboratory inspection. For nonacceptable results, the records will be reviewed to determine the cause for the nonconformance. The actual limits for a batch of performance samples will be provided only after the batch is discontinued.

BLANK SHEET

6. LABORATORY INSPECTION

The laboratory inspection will occur within 45 days after the laboratory(s) have provided the first edition of the QA plan, the site-specific work plan, and the performance sample data. The inspection will be performed by an experienced chemist from the NCR. The chemist may be accompanied by the EIC.

6.1 PURPOSE OF INSPECTION

The purpose of laboratory inspection is to verify that the Navy QC requirements are being met as reflected by the laboratory's daily operations in adherence to the QC plan received by the NCR.

6.2 INSPECTION PROCEDURE

The laboratory inspection involves four phases.

1. Overview and Orientation

The inspector meets with the laboratory management, including the laboratory director, the LQAC, and others as the director deems appropriate. The objectives of the visit are reviewed and a schedule established. The inspector discusses the review of the laboratory's QA/QC plan and the results of the proficiency testing.

2. Observation, Examination, and Review

According to the schedule, the inspector does the following.

- Witnesses performance of specified analytical procedures.
- Reviews sample handling and storage procedures. The inspector will follow the trail of the performance samples through the laboratory.
- Examines the QC records including QC manuals, instrument calibration and maintenance records, control charts, instrument run logs, sample preparation logs, notebooks used to document analysis, corrective action reports for out-of-control events, and performance data generated for other programs such as Superfund CLP and state drinking water.

3. Findings

The inspector conducts an exit interview with the laboratory director, the LQAC, and any other laboratory personnel the director deems appropriate. The inspector summarizes the findings of the visit and details specific deficiencies to be addressed by corrective action.

Recommendations regarding corrective action may be provided. A written report summarizing the findings is provided to the LQAC, the Navy EIC and NCR, and engineering contractor within ten working days of the inspection.

4. Corrective Action (if required)

Within ten days of receipt of the findings, the laboratory submits to the NCR and the EIC a plan to correct the deficiencies identified in the inspection. The plan should include for each deficiency, a description of the corrective action and a date indicating when the action is to be implemented or completed.

A repeat inspection may be required in instances where the number of deficiencies requiring corrective actions are complex. Repeat inspections will be scheduled for the earliest possible date after the last corrective action plan is received by the NCR.

The laboratory shall send a follow-up report which supplies information indicating the proof that the plan has been carried out. For example, if no control charts exist, then the plan would state that these would be in place by a specific date, and the follow-up report would contain copies of the control charts.

7. ANALYTICAL METHODS

An analytical method is a series of steps or procedures that must be performed to determine the identity and quantity of analyte in a sample. The methods to be employed by the Navy-approved laboratory fall mainly into two categories--those which have been approved by the EPA and those which have been developed by the Army. The former refer primarily to the methods presented in the *Federal Register* of October 26, 1984 (49 FR 43234), where the EPA has listed ~250 pollutants (pp. 43251-43258) or pollutant categories and the method(s) by which each must (by virtue of final or final interim ruling status of the methods) be tested. The acceptance of methods not under either status will be handled on a case-by-case basis among the concerned parties. Non-standard methods shall be submitted to the NCR who will discuss the method with Navy and EPA personnel prior to use on Navy projects. Other applicable EPA methods include the *SW-846* methods which are applicable to Resource Conservation and Recovery Act sites and the Superfund CLPs which are applicable to the CERCLA sites.

Many of the EPA methods are found in the documentation of other organizations (e.g., U.S. Geological Services, ASTM) and are incorporated by reference into the regulations. Such incorporation involves listing the organization, the specific document and its date, the method number assigned by the other organization, and perhaps a page number in the document of the other organization. Technically speaking, to maintain the applicability of the regulation, no deviations from the given citations are allowed by the EPA, even in cases where an organization (ASTM, for example) may have an updated version of the method. However, there are instances where EPA regional offices have granted exceptions to different laboratories for the testing of various substances. If a laboratory has such a variance, in writing, from the EPA (either to use a different ASTM method, for example, than the one cited in the October 26, 1984 *Federal Register* or to use a somewhat modified method, for example, than the one cited in the October 26, 1984 *Federal Register*), a copy of the variance (sent to the NCR) may be used to seek Navy approval of the different or modified method. It must also be shown that the conditions for which the variance was issued by the EPA are similar to the expected conditions (sampling and handling techniques, environmental matrix, concentration range, interferences, etc.) in the IRP.

It is also recognized that the analyst may have some leeway resulting from the regulations themselves. For instance, in the October 26, 1984 *Federal Register*, several methods are listed involving GC. Typically, in paragraph 8.1.2, these methods allow the analyst "certain options," provided various subsequent QC requirements are met. For example, the EPA allows some flexibility in the procedures (and no written permission is needed from the EPA) once a sample has been extracted and placed into the instrument. On the other hand, changes in operations prior to this instrumental analysis (e.g., preparation, storage) would probably require written documentation.

The *Federal Register* of October 26, 1984 (49 FR 43437) also contains a proposed ruling where additional substances and methods are listed: specifically, some proposed modifications to Tables IC and ID (Tables 7.1 through 7.5 of this guide) of the previously mentioned final rule. In those cases where a substance/method does not appear on one of the earlier tables but does occur on one of the proposed listings, the method in the proposed listing is recommended by the EPA (Medz, 1985) but without any regulatory force.

For the analytical method to be used in the case of munition-related substances, the laboratory should consult the NCR who will forward a copy(s) of the appropriate method developed by the Army Toxic and Hazardous Materials Agency.

For biota and air samples, the methods must be evaluated individually by the NCR to determine whether they may be used for the work in question.

A list of references containing methods, statistics, and sampling information is supplied in the Bibliography of this document.

For Level D QC sites, the current CLP methods and documentation must be followed. For methods not covered by CLP and for sites requiring Level D, the latest edition of *SW-846* or other methods listed in Tables 7.1 through 7.5, may be used. For the Levels C and E sites, CLP methods, *SW-846* methods, or other methods listed in Tables 7.1 through 7.5 shall be used. The exception to the Levels C and E method requirement occurs in the volatile and semivolatile area. In any level of QC and for any site where volatiles and semivolatiles are analyzed by GC/MS, the current CLP methods shall be used.

7.1 QC REQUIREMENTS FOR THE LABORATORY

The following are the minimum QC requirements for the laboratory analyses. For Level D QC, the current CLP QC requirements are specified. For methods not defined in the CLP, the blank, blank/spike, matrix spike, and matrix spike duplicate shall be performed for every 20 samples of similar matrix. The batch size for Level D QC is 20 samples.

In Levels C and E, the optimum batch size is determined by the number of samples of similar matrix which can be processed simultaneously through the entire preparation and analysis process. For example, if 5 samples can be extracted and 20 analyzed by the instrument, the batch size is 5. Once this is determined, it is used with the blank/spike control program in the following manner.

In Levels C and E, a blank/spike control shall be analyzed with each batch and shall be plotted on control charts as described in Sect. 4.4. For metals, anions, and other wet chemical analysis, a method blank shall also be processed with each batch and shall contain less than the method detection limit for compounds of interest. In any method using surrogates spiked into the blank, the blank shall serve as both the method blank

Table 7.1. List of approved biological test procedures
(40 CFR, Part 136, July 1, 1987)

Parameter and units	Method [1]	Reference (Method Number or Page)			
		EPA [2]	Standard Methods 15th Ed	ASTM	USGS
Bacteria					
1 Coliform (fecal) number per 100 ml	MPN, 5 tube, 3 dilution, or, membrane filter (MF) [3], single step	p. 132	908C		
2 Coliform (fecal) in presence of chlorine number per 100 ml	MPN, 5 tube, 3 dilution	p. 124	909C		8-0053-77
3 Coliform (total), number per 100 ml	MPN, 5 tube, 3 dilution, or, MF [3] single step or two step	p.132	908C		
		p. 114	908A		
4 Coliform (total) in presence of chlorine, number per 100 ml	MPN, 5 tube, dilution, or MF [3] with enrichment	p. 108	908A		8-0025-77
		p. 114	908A		
5 Fecal streptococci, number per 100 ml	MPN, 5 tube, 3 dilution, MF [3], or, plate count	p. 111	909 (A – A.Sc).		
		p. 139	910A		
	p. 136	910B		00055-77 [4]	
	p. 143	910C			

Table 1A footers

[1] The method must be specified when results are reported

[2] "Microbiological Methods for Monitoring the Environment, Water and Wastes, 1978", EPA-600/8-78-017, U.S. Environmental Protection Agency

[3] Greeson, P.E., et al., Methods for Collection and Analysis of Aquatic Biological and Microbiological Samples, "U.S. Geological Survey, Techniques of Water-Resources Investigations Book 5, Chapter A4, Laboratory Analysis, 1977.

[4] 0.45 um membrane filter or other pore size certified by the manufacturer to fully retain organisms to be cultured, and free of extractables which could interfere with their growth and development

[5] Approved only if dissolution of the KF Streptococcus Agar (Section 5.1, USGS Method 8-0055-77) is made in a boiling water bath to avoid scorching of the medium

Table 7.2. List of approved inorganic test procedures
(40 CFR, Part 136, July 1, 1987)

Parameter and Units	Method	Reference (Method No. or Page)				
		EPA 1979	Std. Methods 16th Ed.	ASTM	USGS[1]	Other
1. Acidity, as $CaCO_3$ mg/L	Electrometric end point or phenolphthalein end point.	305.1	402(4.a)	D1067-82(E)	—	
2. Alkalinity, as $CaCO_3$ mg/L	Electrometric or colorimetric titration to pH 4.5, manual, or	310.1	403	D1067-82(B)	I-1030-84	33.014[1]
	Automated.	310.2	—	—	I-2030-84	
3 Aluminum—Total[2] mg/L	Digestion[2] followed by					
	AA direct aspiration,	202.1	303C	—	I-3051-84	
	AA furnace,	202.2	304	—	—	
	Inductively coupled plasma, or	—	—	—	—	200.7[4]
	Colorimetric (Eriochrome cyanine R).	—	306B	—	—	
4 Ammonia (as N) mg/L	Manual distillation (at pH 9.5)[3], followed by					
	Nesslerization,	350.2	417A	—	—	33.057[7]
	Titration,	350.2	417B	D1426-79(A)	I-3520-84	33.057[7]
	Electrode,	350.2	417D	—	—	
	Automated phenate or	350.3	417E or F	D1426-79(D)	—	
	Automated electrode.	350.1	417G	D1426-79(C)	I-4523-84	
		—	—	—	—	Note 6
5 Antimony—Total[2], mg/L	Digestion[2] followed by					
	AA direct aspiration,	204.1	303A	—	—	
	AA furnace, or	204.2	304	—	—	
	Inductively coupled plasma	—	—	—	—	200.7[4]
6 Arsenic—Total[2], mg/L	Digestion[2] followed by	206.5	—	—	—	
	AA gaseous hydride,	206.3	303E	D2972-84(B)	I-3062-84	
	AA furnace,	206.2	304	—	—	
	Inductively coupled plasma, or	—	—	—	—	200.7[4]
	Colorimetric (SDDC)	206.4	307B	D2972-84(A)	I-3060-84	
7 Barium—Total[2], mg/L	Digestion[2] followed by					
	AA direct aspiration,	208.1	303C	—	I-3084-84	
	AA furnace, or	208.2	304	—	—	
	Inductively coupled plasma	—	—	—	—	200.7[4]
8 Beryllium—Total[2], mg/L	Digestion[2] followed by					
	AA direct aspiration,	210.1	303C	D3654-84(A)	I-3095-84	
	AA furnace,	210.2	304	—	—	
	Inductively coupled plasma, or	—	—	—	—	200.7[4]
	Colorimetric (aluminon)	—	309B	—	—	
9 Biochemical oxygen demand (BOD$_5$) mg/L	Dissolved Oxygen Depletion	405.1	507	—	I-1578-78[1]	33.019[2], p.17[4]
10 Boron—Total mg/L	Colorimetric (curcumin),	212.3	404A	—	I-3112-84	
	or Inductively Coupled plasma.	—	—	—	—	200.7[4]
11 Bromide mg/L	Titrimetric.	320.1	—	D1246-82(C)	I-1125-84	p.544[1]

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Table 7.2. (continued)

Parameter and Units	Method	Reference (Method No. or Page) EPA 1979	Std. Methods 16th Ed.	ASTM	USGS[1]	Other
12. Cadmium—Total[3], mg/L	Digestion[3] followed by					
	AA direct aspiration,	213.1	303A or B	D3557-84(A or B)	I-3135-84 or I-3136-84	33.069[3], p.37[4]
	AA furnace,	213.2	304	—	—	
	Inductively coupled plasma,	—	—	—	—	200.7[4]
	Voltametry[10], or	—	—	D3557-84(C)	—	
	Colorimetric (Dithizone).	—	3108	—	—	
13. Calcium—Total[3], mg/L	Digestion[3] followed by					
	AA direct aspiration,	215.1	303A	D511-84(B)	I-3152-84	
	Inductively coupled plasma, or	—	—	—	—	200.7[4]
	Titrimetric (EDTA).	215.2	311C	D511-84(A)	—	
14 Carbonaceous biochemical oxygen demand (CBOD$_5$), mg/L[11]	Dissolved Oxygen Depletion with nitrification inhibitor.	—	507(5.a.6)	—	—	
15 Chemical oxygen demand (COD), mg/L	Titrimetric, or,	410.1, 410.2, or 410.3	508A	D1252-83	I-3560-84 or I-3562-84	33.034[1], p.17[4]
	Spectrophotometric, manual or automated.	410.4	—	—	I-3561-84	Notes 12 or 13
16 Chloride, mg/L	Titrimetric (silver nitrate)	—	407A	D512-81(B)	I-1183-84	
	or (Mercuric nitrate), or	325.3	407B	D512-81(A)	I-1184-84	33.067[1]
	Colorimetric, manual or	—	—	D512-81(C)	I-1187-84	
	Automated (ferricyanide).	325.1, or 325.2	407D	—	I-2187-84	
17 Chlorine—Total residual, mg/L	Titrimetric					
	Amperometric direct,	330.1	408C	D1253-76(A)	—	
	Starch end point direct,	330.3	408A	D1253-76(B)	—	
				Part 18.3		
	Back titration either end point[14], or	330.2	408B	—		
	DPD-FAS,	330.4	408D	—	—	
	Spectrophotometric, DPD,	330.5	408E	—	—	
	or Electrode.	—	—	—	—	Note 15
18 Chromium VI dissolved, mg/L	0.45 micron filtration followed by					
	AA chelation-extraction,	218.4	303B	—	I-1232-84	
	or Colorimetric (Diphenylcarbazide)	—	—	—	I-1230-84	3078[14]
19 Chromium—Total[3], mg/L	Digestion[3] followed by					
	AA direct aspiration,	218.1	303A	D1687-84(D)	I-3236-84	33.069[3]
	AA chelation—extraction,	218.3	303B	—	—	
	AA furnace,	218.2	304	—	—	
	Inductively coupled plasma, or	—	—	—	—	200.7[4]
	Colorimetric (Diphenylcarbazide).	—	312B	D1687-84(A)	—	
20 Cobalt—Total[3], mg/L	Digestion[3] followed by					
	AA direct aspiration,	219.1	303A or B	D3558-84(A or B)	I-3239-84 or I-3240-84	p.37[4]
	AA furnace, or	219.2	304	—	—	
	Inductively coupled plasma	—	—	—	—	200.7[4]

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Table 7.2. (continued)

Parameter and Units	Method	Reference (Method No. or Page) EPA 1979	Std. Methods 16th Ed.	ASTM	USGS[1]	Other
21. Color, platinum cobalt units or dominant wavelength, hue, luminance, purity.	Colorimetric (ADMI), or (Platinum cobalt), or Spectrophotometric.	110.1 110.2 110.3	204D 204A 204B	— — —	— I-1250-84 —	Note 17
22. Copper—Total[3], mg/L	Digestion[3] followed by AA direct aspiration.	220.1	303A or B	D1688-84(D or E)	I-3270-84 or I-3271-84	33.089[3], p.37[4]
	AA furnace.	220.2	304	—	—	
	Inductively coupled plasma.	—	—	—	—	200.7[4]
	Colorimetric (Neocuproine), or (Bicinchoninate).	— —	313B —	D1688-84(A) —	— —	Note 18
23. Cyanide—Total, mg/L	Manual distillation with $MgCl_2$ followed by Titrimetric, or	— —	412B 412C	— —	— —	p.22[4]
	Spectrophotometric, manual or	335.2	412D	D2036-82(A)	I-3300-84	
	Automated.[10]	335.3	—	D2036-82(A)	—	
24. Cyanide amenable to chlorination, mg/L	Manual distillation with $MgCl_2$ followed by titrimetric or spectrophotometric	335.1	412F	D2036-82(B)	—	
25. Fluoride—Total, mg/L	Manual distillation[5] followed by	—	413A	—	—	
	Electrode, manual or	340.2	413B	D1179-80(B)	—	
	Automated.	—	—	—	I-4327-84	
	Colorimetric (SPADNS), or Automated complexone.	340.1 340.3	413C 413E	D1179-80(A) —	— —	
26. Gold—Total[3], mg/L	Digestion[3] followed by AA direct aspiration, or	231.1	303A	—	—	
	AA furnace.	231.2	304	—	—	
27. Hardness—Total, as $CaCO_3$, mg/L	Automated colorimetric.	130.1	—	—	—	
	Titrimetric (EDTA), or Ca plus Mg as their carbonates, by inductively coupled plasma or AA direct aspiration. (See Parameters 13 and 33.)	130.2	314B	D1126-80	I-1338-84	33.082[3]
28. Hydrogen ion (pH), pH units	Electrometric measurement, or	150.1	423	D1293-84(A or B)	I-1586-84	33.006[3]
	Automated electrode.	—	—	—	—	Note 20
29. Iron—Total[3], mg/L	Digestion[3] followed by AA direct aspiration, or	235.1	303A	—	—	
	AA furnace.	235.2	304	—	—	
30. Iron—Total[3], mg/L	Digestion[3] followed by AA direct aspiration.	236.1	303A or B	D1068-84(C or D)	I-3381-84	33.089[3]
	AA furnace.	236.2	304	—	—	
	Inductively coupled plasma, or	—	—	—	—	200.7[4]
	Colorimetric (Phenanthroline).	—	315B	D1068-84(A)	—	Note 21

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Table 7.2. (continued)

Parameter and Units	Method	Reference (Method No. or Page) EPA 1979	Std. Methods 16th Ed.	ASTM	USGS[1]	Other
31. Kjeldahl nitrogen—Total, (as N), mg/L	Digestion and distillation followed by					
	Titration,	351.3	420A or B	D3590-84(A)	—	
	Nesslerization,	351.3	417D	D3590-84(A)	—	33.051[1]
	Electrode,	351.3	417B	D3590-84(A)	—	
	Automated phenate,	351.3	417E or F	—	—	
	Semi-automated block digestor, or	351.1	—	—	I-4551-78[1]	
	Potentiometric.	351.2	—	D3590-84(A)	—	
		351.4	—	D3590-84(A)	—	
32. Lead—Total[3], mg/L	Digestion[3] followed by					
	AA direct aspiration,	239.1	303A or B	D3559-85(A or B)	I-3399-84	33.089[2]
	AA furnace,	239.2	304	—	—	
	Inductively coupled plasma,	—	—	—	—	200.7[4]
	Voltametry[16], or	—	—	D3559-85(C)	—	
	Colorimetric (Dithizone).	—	3168	—	—	
33. Magnesium—Total[3], mg/L	Digestion[3] followed by					
	AA direct aspiration,	242.1	303A	D511-84(B)	I-3447-84	33.089[2]
	Inductively coupled plasma, or	—	—	—	—	200.7[4]
	Gravimetric	—	3188	D511-77(A)	—	
34. Manganese—Total[3], mg/L	Digestion[3] followed by					
	AA direct aspiration,	243.1	303A or B	D858-84(B or C)	I-3454-84	33.089[2]
	AA furnace,	243.2	304	—	—	
	Inductively coupled plasma, or	—	—	—	—	200.7[4]
	Colorimetric (Persulfate), or	—	3198	D858-84(A)	—	33.126[2]
	(Periodate)	—	—	—	—	Note 22
35. Mercury—Total[3], mg/L	Cold vapor, manual or	245.1	303F	D3223-80	I-3462-84	33.095[2]
	Automated.	245.2	—	—	—	
36. Molybdenum—Total[3], mg/L	Digestion[3] followed by					
	AA direct aspiration,	246.1	303C	—	I-3490-84	
	AA furnace, or	246.2	304	—	—	
	Inductively coupled plasma.	—	—	—	—	200.7[4]
37. Nickel—Total[3], mg/L	Digestion[3] followed by					
	AA direct aspiration,	249.1	303A or B	D1886-84(C or D)	I-3499-84	
	AA furnace,	249.2	304	—	—	
	Inductively coupled plasma, or	—	—	—	—	200.7[4]
	Colorimetric (Heptoxime).	—	3218	—	—	
38. Nitrate (as N), mg/L	Colorimetric (Brucine sulfate), or	352.1	—	D992-71	—	33.063[1], 419D[14], p.28[6]
	Nitrate-nitrite N minus Nitrite N (See parameters 39 and 40)					
39. Nitrate-nitrite (as N), mg/L	Cadmium reduction,					
	Manual or	353.3	418C	D3867-85(B)	—	
	Automated, or	353.2	418F	D3867-85(A)	I-4545-84	
	Automated hydrazine.	353.1	—	—	—	

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Table 7.2. (continued)

Parameter and Units	Method	Reference (Method No. or Page) EPA 1979	Std. Methods 16th Ed.	ASTM	USGS[1]	Other
40. Nitrate (as N), mg/L	Spectrophotometric,					
	Manual or	354.1	419	D1254-67	—	Note 24
	Automated (Diazotization).	—	—	—	I-4540-84	
41. Oil and grease—Total recoverable, mg/L	Gravimetric (extraction).	413.1	503A	—	—	
42. Organic carbon—Total (TOC), mg/L	Combustion or oxidation.	415.1	505	D2579-85(A or B)	—	33.044[1], p.433
43. Organic nitrogen (as N) mg/L	Total Kjeldahl N (Parameter 31) minus ammonia N (Parameter 4.)					
44. Orthophosphate (as P), mg/L	Ascorbic acid method,					
	Automated or	365.1	424G	—	I-4601-84	33.116[1]
	Manual single reagent,	365.2	424F	D515-82(A)	—	33.111[1]
	or Manual two reagent.	365.3	—	—	—	
45. Osmium—Total[3], mg/L	Digestion[3] followed by					
	AA direct aspiration, or	252.1	303C	—	—	
	AA furnace.	252.2	304	—	—	
46. Oxygen, dissolved, mg/L	Winkler (Azide modification), or	360.2	421B	D888-81(C)	I-1575-78[7]	33.028[1]
	Electrode.	360.1	421F	—	I-1576-78[7]	
47. Palladium—Total[3], mg/L	Digestion[3] followed by					
	AA direct aspiration, or	253.1	—	—	—	p.527[9]
	AA furnace.	253.2	—	—	—	p.528[9]
48. Phenols, mg/L	Manual distillation[11] followed by	420.1	—	D1783-80(A or B)	—	Note 26
	Colorimetric (4AAP) manual, or	420.1	—	—	—	Note 26
	Automated[12].	420.2	—	—	—	
49. Phosphorus (elemental) mg/L	Gas-liquid chromatography.	—	—	—	—	Note 27
50. Phosphorus—Total, mg/L	Persulfate digestion followed by	365.2	424C(III)	—	—	33.111[1]
	Manual or	365.2 or 365.3	424F	D515-82(A)	—	
	Automated ascorbic acid reduction, or	365.1	424G	—	I-4600-84	33.116[1]
	Semi-automated block digestor.	365.4	—	—	—	
51. Platinum—Total[3], mg/L	Digestion[3] followed by					
	AA direct aspiration, or	255.1	303A	—	—	
	AA furnace.	255.2	304	—	—	
52. Potassium—total[3], mg/L	Digestion followed by					
	AA direct aspiration,	258.1	303A	—	I-3630-84	33.103[1]
	Inductively coupled plasma	—	—	—	—	200.7[4]
	Flame photometric, or	—	322B	D1428-82(A)	—	
	Colorimetric (Cobaltinitrite).	—	—	—	—	317B[14]

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Table 7.2. (continued)

Parameter and Units	Method	Reference (Method No. or Page) EPA 1979	Std. Methods 16th Ed.	ASTM	USGS[1]	Other
53. Residue—Total, mg/L	Gravimetric, 103-105°C.	160.3	209A	—	I-3750-84	
54. Residue—filterable, mg/L	Gravimetric, 180°C.	160.1	209B	—	I-1750-84	
55. Residue—nonfilterable, (TSS), mg/L	Gravimetric, 103-105°C post washing of residue.	160.2	209C	—	I-3765-84	
56. Residue—settleable, mg/L	Volumetric (Imhoff cone) or gravimetric.	160.5	209E	—	—	
57. Residue—volatile, mg/L	Gravimetric, 550°C.	160.4	209D	—	I-3753-84	
58. Rhodium—Total[3], mg/L	Digestion[3] followed by					
	AA direct aspiration, or	265.1	303A	—	—	
	AA furnace.	265.2	304	—	—	
59. Ruthenium—Total[3], mg/L	Digestion[3] followed by					
	AA direct aspiration, or	267.1	303A	—	—	
	AA furnace.	267.2	304	—	—	
60. Selenium—Total[3], mg/L	Digestion[3] followed by					
	AA furnace,	270.2	304	—	—	
	Inductively coupled plasma, or	—	—	—	—	200.7[4]
	AA gaseous hydride.	270.3	303E	D3859-84(A)	I-3667-84	
61. Silica—Dissolved, mg/L	0.45 micron filtration followed by					
	Colorimetric, Manual or	370.1	425C	D859-80(B)	I-1700-84	
	Automated (Molybdo-silicate), or	—	—	—	I-2700-84	
	Inductively coupled plasma.	—	—	—	—	200.7[4]
62. Silver—Total[10], mg/L	Digestion[3] followed by					
	AA direct aspiration,	272.1	303A or B	—	I-3720-84	33.069[2], p.37[4]
	AA furnace,	272.2	304	—	—	
	Colorimetric (Dithizone), or	—	—	—	—	3198[11]
	Inductively coupled plasma.	—	—	—	—	200.7[4]
63. Sodium—Total[3], mg/L	Digestion[3] followed by					
	AA direct aspiration,	273.1	303A	—	I-3735-84	33.107[2]
	Inductively coupled plasma, or	—	—	—	—	200.7[4]
	Flame photometric.	—	325B	D1428-82(A)	—	
64. Specific conductance, micromhos/cm at 25°C	Wheatstone bridge.	120.1	205	D1125-82(A)	I-1780-84	33.002[2]
65. Sulfate (as SO_4), mg/L	Automated colorimetric (barium chloranilate)	375.1	—	—	—	
	Gravimetric, or	375.3	426A or B	D516-82(A)	—	33.124[2]
	Turbidimetric.	375.4	—	D516-82(B)	—	426C[12]

Table 7.2. (continued)

Parameter and Units	Method	Reference (Method No. or Page)				
		EPA 1979	Std. Methods 16th Ed.	ASTM	USGS[1]	Other
66. Sulfide (as S), mg/L	Titrimetric (iodine) or	376.1	427D	—	I-3840-84	228A[2]
	Colorimetric (methylene blue).	376.2	427C	—	—	
67. Sulfite (as SO_3), mg/L	Titrimetric (iodine-iodate).	377.1	428A	D1339-84(C)	—	
68. Surfactants, mg/L	Colorimetric (methylene blue).	425.1	512B	D2330-82(A)	—	
69. Temperature, °C.	Thermometric.	170.1	212	—	—	Note 31
70. Thallium—Total[3], mg/L	Digestion[3] followed by					
	AA direct aspiration,	279.1	303A	—	—	
	AA furnace, or	279.2	304	—	—	
	Inductively coupled plasma.	—	—	—	—	200.7[4]
71. Tin—Total[3], mg/L	Digestion[3] followed by					
	AA direct aspiration, or	282.1	303A	—	I-3850-78[5]	
	AA furnace.	282.2	304	—	—	
72. Titanium—Total[3], mg/L	Digestion[3] followed by					
	AA direct aspiration, or	283.1	303C	—	—	
	AA furnace.	283.2	304	—	—	
73. Turbidity, NTU	Nephelometric.	180.1	214A	D1889-81	I-3860-84	
74. Vanadium, Total[3], mg/L	Digestion[3] followed by					
	AA direct aspiration,	286.1	303C	—	—	
	AA furnace,	286.2	304	—	—	
	Inductively coupled plasma, or	—	—	—	—	200.7[4]
	Colorimetric (Gallic acid).	—	327B	D3373-84(A)	—	
75. Zinc—Total[3], mg/L	Digestion[3] followed by					
	AA direct aspiration,	289.1	303A or B	D1691-84(C or D)	I-3900-84	33.089[6], p.37[7]
	AA furnace,	289.2	304	—	—	
	Inductively coupled plasma, or	—	—	—	—	200.7[4]
	Colorimetric (Dithizone) or	—	328C	—	—	
	(Zincon).	—	—	—	—	Note 32

[1] "Methods for Analysis of Inorganic Substances in Water and Fluvial Sediments," U.S. Department of the Interior, U.S. Geological Survey, Open-File Report 85-495, 1986, unless otherwise stated.
[2] "Official Methods of Analysis of the Association of Official Analytical Chemists," methods manual, 14th ed. (1985).
[3] For the determination of total metals the sample is not filtered before processing. A digestion procedure is required to solubilize suspended material and to destroy possible organic-metal complexes. Two digestion procedures are given in "Methods for Chemical Analysis of Water and Wastes, 1979." One (Section 4.1.3) is a vigorous digestion using nitric acid. A less vigorous digestion using nitric and hydrochloric acids (Section 4.1.4) is preferred, however, the analyst should be cautioned that this mild digestion may not suffice for all sample types. Particularly, if a colorimetric procedure is to be employed, it is necessary to ensure that all organo-metallic bonds be broken so that the metal is in a reactive state. In those situations, the vigorous digestion is to be preferred making certain that at no time does the sample go to dryness. Samples containing large amounts of organic materials would also benefit by this vigorous digestion. Use of the graphite furnace technique, inductively coupled plasma, as well as determinations for certain elements such as arsenic, the noble metals, mercury, selenium, and titanium require a modified digestion and in all cases the method write-up should be consulted for specific instruction and/or cautions.

NOTE: If the digestion included in one of the other approved references is different than the above, the EPA procedure must be used.

Dissolved metals are defined as those constituents which will pass through a 0.45 micron membrane filter. Following filtration of the sample, the referenced procedure for total metals must be followed. Sample digestion for dissolved metals may be omitted for AA (direct aspiration or graphite furnace) and ICP analyses provided the sample solution to be analyzed meets the following criteria.

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Table 7.2. (continued)

a. has a low COD (<20)
b. is visibly transparent with a turbidity measurement of 1 NTU or less,
c. is colorless with no perceptable odor, and
d. is of one liquid phase and free of particulate or suspended matter following acidification.

[4]The full text of Method 200.7, "Inductively Coupled Plasma Atomic Emission Spectrometric Method for Trace Element Analysis of Water and Wastes," is given at Appendix C of this Part 136.

[5]Manual distillation is not required if comparability data on representative effluent samples are on company file to show that this preliminary distillation step is not necessary, however, manual distillation will be required to resolve any controversies.

[6]Ammonia, Automated Electrode Method, Industrial Method Number 379-75 WE, dated February 19, 1976, Technicon AutoAnalyzer II, Technicon Industrial Systems, Tarrytown, NY, 10591.

[7]The approved method is that cited in "Methods for Determination of Inorganic Substances in Water and Fluvial Sediments," USGS TWRI, Book 5, Chapter A1 (1979).

[8]American National Standard on Photographic Processing Effluents, Apr. 2, 1975. Available from ANSI, 1430 Broadway, New York, NY 10018.

[9]"Selected Analytical Methods Approved and Cited by the United States Environmental Protection Agency," Supplement to the Fifteenth Edition of *Standard Methods for the Examination of Water and Wastewater* (1981).

[10]The use of normal and differential pulse voltage ramps to increase sensitivity and resolution is acceptable.

[11]Carbonaceous biochemical oxygen demand ($CBOD_5$) must not be confused with the traditional BOD_5 test which measures "total BOD." The addition of the nitrification inhibitor is not a procedural option, but must be included to report the $CBOD_5$ parameter. A discharger whose permit requires reporting the traditional BOD_5 may not use a nitrification inhibitor in the procedure for reporting the results. Only when a discharger's permit specifically states $CBOD_5$ is required, can the permittee report data using the nitrification inhibitor.

[12]DIC Chemical Oxygen Demand Method, Oceanography International Corporation, 512 West Loop, P.O. Box 2980, College Station, TX 77840.

[13]Chemical Oxygen Demand, Method 8000, Hach Handbook of Water Analysis, 1979, Hach Chemical Company, P.O. Box 389, Loveland, CO 80537

[14]The back titration method will be used to resolve controversy.

[15]Orion Research Instruction Manual, Residual Chlorine Electrode Model 97-70, 1977, Orion Research Incorporated, 840 Memorial Drive, Cambridge, MA 02138.

[16]The approved method is that cited in *Standard Methods for the Examination of Water and Wastewater*, 14th Edition, 1976.

[17]National Council of the Paper Industry for Air and Stream Improvement, (Inc.) Technical Bulletin 253, December 1971.

[18]Copper, Bicinchoinate Method, Method 8506, Hach Handbook of Water Analysis, 1979, Hach Chemical Company, P.O. Box 389, Loveland, CO 80537.

[19]After the manual distillation is completed, the autoanalyzer manifolds in EPA Methods 335.3 (cyanide) or 420.2 (phenols) are simplified by connecting the re-sample line directly to the sampler. When using the manifold setup shown in Method 335.3, the buffer 6.2 should be replaced with the buffer 7.6 found in Method 335.2.

[20]Hydrogen Ion (pH) Automated Electrode Method, Industrial Method Number 378-75WA, October 1976, Technicon Auto-Analyzer II, Technicon Industrial Systems, Tarrytown, NY 10591.

[21]Iron, 1,10-Phenanthroline Method, Method 8008, 1980, Hach Chemical Company, P.O. Box 389, Loveland, CO 80537.

[22]Manganese, Periodate Oxidation Method, Method 8034, Hach Handbook of Wastewater Analysis, 1979, pages 2-113 and 2-117, Hach Chemical Company, Loveland, CO 80537.

[23]Goerlitz, D., Brown, E., "Methods for Analysis of Organic Substances in Water," U.S. Geological Survey Techniques of Water-Resources Inv., book 5, ch. A3, page 4 (1972).

[24]Nitrogen, Nitrite, Method 8507, Hach Chemical Company, P.O. Box 389, Loveland, CO 80537.

[25]Just prior to distillation, adjust the sulfuric-acid-preserved sample to pH 4 with 1 + 9 NaOH.

[26]The approved method is that cited in *Standard Methods for the Examination of Water and Wastewater*, 14th Edition. The colorimetric reaction is conducted at a pH of 10.0 ± 0.2. The approved methods are given on pp. 576-81 of the 14th Edition: Method 510A for distillation, Method 510B for the manual colorimetric procedure, or Method 510C for the manual spectrophotometric procedure.

[27]R. F. Addison and R. G. Ackman, "Direct Determination of Elemental Phosphorus by Gas-Liquid Chromatography," *Journal of Chromatography*, vol. 47, No. 3, pp. 421–426, 1970.

[28]Approved methods for the analysis of silver in industrial wastewaters at concentrations of 1 mg/L and above are inadequate where silver exists as an inorganic halide. Silver halides such as the bromide and chloride are relatively insoluble in reagents such as nitric acid but are readily soluble in an aqueous buffer of sodium thiosulfate and sodium hydroxide to a pH of 12. Therefore, for levels of silver above 1 mg/L, 20 mL of sample should be diluted to 100 mL by adding 40 mL each of 2 M $Na_2S_2O_3$ and 2M NaOH. Standards should be prepared in the same manner. For levels of silver below 1 mg/L the approved method is satisfactory.

[29]The approved method is that cited in *Standard Methods for the Examination of Water and Wastewater*, 15th Edition.

[30]The approved method is that cited in *Standard Methods for the Examination of Water and Wastewater*, 13th Edition.

[31]Stevens, H. H., Ficke, J. F., and Smoot, G. F., "Water Temperature—Influential Factors, Field Measurement and Data Presentation," U.S. Geological Survey, Techniques of Water Resources Investigations, Book 1, Chapter D1, 1975.

[32]Zinc, Zincon Method, Method 8009, Hach Handbook of Water Analysis, 1979, pages 2-231 and 2-233, Hach Chemical Company, Loveland, CO 80537.

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Table 7.3. List of approved test procedures for nonpesticide organic compounds (40 CFR, Part 136, July 1, 1987)

Parameter [1]	EPA Method Number [1,1]			Other
	GC	GC/MS	HPLC	
1 Acenaphthene	610	625, 1625	610	
2. Acenaphthylene	610	625, 1625	610	
3. Acrolein	603	*624, 1624		
4 Acrylonitrile	603	*624, 1624		
5 Anthracene	610	625, 1625	610	
6 Benzene	602	624, 1624		
7 Benzidine		*625, 1625	605	Note 3, p. 1;
8 Benzo(a)anthracene	610	625, 1625	610	
9 Benzo(a)pyrene	610	625, 1625	610	
10 Benzo(b)fluoranthene	610	625, 1625	610	
11. Benzo(ghi)perylene	610	625, 1625	610	
12 Benzo(k)fluoranthene	610	625, 1625	610	
13 Benzyl Chloride				Note 3, p. 130; Note 6, p. $102.
14 Benzyl Butyl Phthalate	606	625, 1625		
15 Bis(2-chloroethoxy) methane	611	625, 1625		
16 Bis(2-chloroethyl) ether	611	625, 1625		
17 Bis(2-ethylhexyl) phthalate	606	625, 1625		
18 Bromodichloromethane	601	624, 1624		
19 Bromoform	601	624, 1624		
20. Bromomethane	601	624, 1624		
21 4-Bromophenylphenyl ether	611	625, 1625		
22. Carbon tetrachloride	601	624, 1624		Note 3, p. 130;
23 4-Chloro-3-methylphenol	604	625, 1625		
24 Chlorobenzene	601, 602	624, 1624		Note 3, p. 130;
25 Chloroethane	601	624, 1624		
26 2-Chloroethylvinyl ether	601	624, 1624		
27 Chloroform	601	624, 1624		Note 3, p. 130;
28 Chloromethane	601	624, 1624		
29. 2-Chloronaphthalene	612	625, 1625		
30 2-Chlorophenol	604	625, 1625		
31 4-Chlorophenylphenyl ether	611	625, 1625		
32 Chrysene	610	625, 1625	610	
33 Dibenzo(a,h)anthracene	610	625, 1625	610	
34 Dibromochloromethane	601	624, 1624		
35 1,2-Dichlorobenzene	601, 602, 612	624, 625, 1625		
36 1,3-Dichlorobenzene	601, 602, 612	624, 625, 1625		
37 1,4-Dichlorobenzene	601, 602, 612	625, 1624, 1625		
38 3,3-Dichlorobenzidine		625, 1625	605	
39 Dichlorodifluoromethane	601			
40 1,1-Dichloroethane	601	624, 1624		
41 1,2-Dichloroethane	601	624, 1624		
42 1,1-Dichloroethene	601	624, 1624		
43 trans-1,2-Dichloroethene	601	624, 1624		
44 2,4-Dichlorophenol	604	625, 1625		
45 1,2-Dichloropropane	601	624, 1624		
46 cis-1,3-Dichloropropene	601	624, 1624		
47 trans-1,3-Dichloropropene	601	624, 1624		
48 Diethyl phthalate	606	625, 1625		
49 2,4-Dimethylphenol	606	625, 1625		
50 Dimethyl phthalate	606	625, 1625		
51. Di-n-butyl phthalate	606	625, 1625		
52. Di-n-octyl phthalate	606	625, 1625		
53 2,4-Dinitrophenol	604	625, 1625		
54 2,4-Dinitrotoluene	609	625, 1625		
55 2,6-Dinitrotoluene	609	625, 1625		
56 Epichlorohydrin				Note 3, p. 130, Note 6, p. $102.
57. Ethylbenzene	602	624, 1624		
58. Fluoranthene	610	625, 1625	610	
59 Fluorene	610	625, 1625	610	
60 Hexachlorobenzene	612	625, 1625		
61 Hexachlorobutadiene	612	625, 1625		
62 Hexachlorocyclopentadiene	612	*625, 1625		
63 Hexachloroethane	612	625, 1625		
64 Indeno(1,2,3-cd)pyrene	610	625, 1625	610	
65 Isophorone	609	625, 1625		
66 Methylene Chloride	601	624, 1624		Note 3, p. 130.
67 2-Methyl-4,6-Dinitrophenol	604	625, 1625		
68 Naphthalene	610	625, 1625		
69 Nitrobenzene	609	625, 1625		
70 2-Nitrophenol	604	625, 1625		
71 4-Nitrophenol	604	625, 1625		
72 N-Nitrosodimethylamine	607	625, 1625		
73 N-Nitrosodi-n-propylamine	607	*625, 1625		
74 N-Nitrosodiphenylamine	607	*625, 1625		
75 2,2'-oxybis(1-chloropropane)	611	625, 1625		
76 PCB-1016	608		625,	Note 3, p. 43.
77 PCB-1221	608		625,	Note 3, p. 43.
78 PCB-1232	608		625,	Note 3, p. 43.
79 PCB-1242	608		625,	Note 3, p. 43.
80 PCB-1248	608		625,	Note 3, p. 43.
81 PCB-1254	608		625,	Note 3, p. 43.
82 PCB-1260	608		625,	Note 3, p. 43.
83 Pentachlorophenol	604	625, 1625		Note 3, p. 140.

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Table 7.3. (continued)

Parameter [1]	EPA Method Number [2,3]			Other
	GC	GC/MS	HPLC	
84. Phenanthrene				
85. Phenol	610	625, 1625	610	
86. Pyrene	604	625, 1625		
87. 2,3,7,8-Tetrachlorodibenzo-p-dioxin	610	625, 1625	610	
88. 1,1,2,2-Tetrachloroethane		[4] 613		
89. Tetrachloroethene	601	624, 1624		
90. Toluene	601	624, 1624		Note 3, p. 130;
91. 1,2,4-Trichlorobenzene	602	624, 1624		Note 3, p. 130;
92. 1,1,1-Trichloroethane	612	625, 1625		
93. 1,1,2-Trichloroethane	601	624, 1624		Note 3, p. 130;
94. Trichloroethene	601	624, 1624		
95. Trichlorofluoromethane	601	624, 1624		Note 3, p. 130;
96. 2,4,6-Trichlorophenol	601	624,		
97. Vinyl Chloride	604	625, 1625		
	601	624, 1624		

Table IC Notes

[1] All parameters are expressed in micrograms per liter (µg/L).

[2] The full text of Methods 601–613, 624, 625, 1624, and 1625, are given at Appendix A, "Test Procedures for Analysis of Organic Pollutants," of this Part 136. The standardized test procedure to be used to determine the method detection limit (MDL) for these test procedures is given at Appendix B, "Definition and Procedure for the Determination of the Method Detection Limit," of this Part 136.

[3] "Methods for Benzidine, Chlorinated Organic Compounds, Pentachlorophenol and Pesticides in Water and Wastewater," U.S. Environmental Protection Agency, September, 1971.

[4] Method 624 may be extended to screen samples for Acrolein and Acrylonitrile. However, when they are known to be present, the preferred method for these two compounds is Method 603 or Method 1624.

[5] Method 625 may be extended to include benzidine, hexachlorocyclopentadiene, N-nitrosodimethylamine, and N-nitrosodiphenylamine. However, when they are known to be present, Methods 605, 607, and 612, or Method 1625, are preferred methods for these compounds.

[6] 625, Screening only.

[7] "Selected Analytical Methods Approved and Cited by the United States Environmental Protection Agency," Supplement to the Fifteenth Edition of *Standard Methods for the Examination of Water and Wastewater* (1981).

[8] Each analyst must make an initial, one-time, demonstration of their ability to generate acceptable precision and accuracy with Methods 601–613, 624, 625, 1624, and 1625 (See Appendix A of this Part 136) in accordance with procedures each in section 8.2 of each of these Methods. Additionally, each laboratory, on an ongoing basis must spike and analyze 10% (5% for Methods 624 and 625 and 100% for methods 1624, and 1625) of all samples to monitor and evaluate laboratory data quality in accordance with sections 8.3 and 8.4 of these Methods. When the recovery of any parameter falls outside the warning limits, the analytical results for that parameter in the unspiked sample are suspect and cannot be reported to demonstrate regulatory compliance.

Note.—These warning limits are promulgated as an "interim final action with a request for comments."

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Table 7.4. List of approved test procedures for pesticides[1]
(40 CFR, Part 136, July 1, 1987)

Parameter (µg/L)	Method	EPA[2,3]	Standard Methods 15th Ed	ASTM	Other
1. Aldrin	GC	608	509A	D3086	Note 3, p 7; Note 4, p 30.
	GC/MS	625			
2. Amitrole	GC				Note 3, p 83, Note 6, p S66
3. Aminocarb	TLC				Note 3, p 94; Note 6, p S16.
4. Alanton	GC				Note 3, p 83, Note 6, p S66.
5. Atrazine	GC				Note 3, p 83, Note 6, p S66
6. Azinphos methyl	GC				Note 3, p 25; Note 6, p S51.
7. Barban	TLC				Note 3, p 104; Note 6, p S64.
8. α-BHC	GC	608	509A	D3086	Note 3, p 7.
	GC/MS	625			
9. β-BHC	GC	608		D3086	
	GC/MS	625			
10. δ-BHC	GC	608		D3086	
	GC/MS	625			
11. γ-BHC (Lindane)	GC	608	509A	D3086	Note 3, p 7; Note 4, p 30
	GC/MS	625			
12. Captan	GC		509A		Note 3, p 7.
13. Carbaryl	TLC				Note 3, p 94; Note 6, p S60
14. Carbophenothion	GC				Note 4, p 30; Note 6, p S73.
15. Chlordane	GC	608	509A	D3086	Note 3, p 7
	GC/MS	625			
16. Chlorobenzilate	TLC				Note 3, p 104, Note 6, p S64
17. 2,4-D	GC		509B		Note 3, p 115, Note 4, p 35.
18. 4,4'-DDD	GC	608	509A	D3086	Note 3, p 7; Note 4, p 30
	GC/MS	625			
19. 4,4'-DDE	GC	608	509A	D3086	Note 3, p 7; Note 4, p 30
	GC/MS	625			
20. 4,4'-DDT	GC	608	509A	D3086	Note 3, p 7; Note 4, p 30.
	GC/MS	625			
21. Demeton-O	GC				Note 3, p 25, Note 6, p S51
22. Demeton-S	GC				Note 3, p 25, Note 6, p S51
23. Diazinon	GC				Note 3, p 25, Note 4, p 30, Note 6, p S51
24. Dicamba	GC				Note 3, p 115
25. Dichlofenthion	GC				Note 4, p 30, Note 6, p S73
26. Dichloran	GC		509A		Note 3, p 7
27. Dicofol	GC			D3086	
28. Dieldrin	GC	608	509A		Note 3, p 7, Note 4, p 30
	GC/MS	625			
29. Dioxathion	GC				Note 4, p 30, Note 6, p S73
30. Diquat	GC				Note 3, p , Note 6, p S51
31. Diuron	TLC				Note 3, p 104, Note 6, p S64
32. Endosulfan I	GC	608	509A	D3086	Note 3, p 7
	GC/MS	625			
33. Endosulfan II	GC	608	509A	D3086	Note 3, p 7
	GC/MS	625			
34. Endosulfan sulfate	GC	608			
	GC/MS	625			
35. Endrin	GC	608	509A	D3086	Note 3, p 7, Note 4, p 30
	GC/MS	625			
36. Endrin aldehyde	GC	608			
	GC/MS	625			
37. Ethion	GC				Note 4, p 30, Note 6, p S73
38. Fenuron	TLC				Note 3, p 104, Note 6, p S64
39. Fenuron TCA	TLC				Note 3, p 104, Note 6, p S64
40. Heptachlor	GC	608	509A	D3086	Note 3, p 7, Note 4, p 30
	GC/MS	625			
41. Heptachlor epoxide	GC	608	509A	D3086	Note 3, p 7, Note 4, p 30, Note 6, p S73.
42. Isodrin	GC/MS	625			
43. Linuron	GC				Note 4, p 30, Note 6, p S73
44. Malathion	TLC				Note 3, p 104, Note 6, p S64
45. Methoxychlor	GC		509A		Note 3, p 25, Note 4, p 30 Note 6, p S51
46. Methomyl	TLC				Note 3, p 94 Note 6 p S60
47. Metribuzin	GC		509A	D3086	Note 3, p 7, Note 4, p 30
48. Mirex	TLC				Note 3, p 94, Note 6, p S60
49. Monuron	GC		509A		Note 3, p 7
50. Monuron TCA	TLC				Note 3, p 104, Note 6, p S64
51. Neburon	TLC				Note 3, p 104 Note 6 p S64
52. Parathion methyl	TLC				Note 3, p 104 Note 6, p S64
53. Parathion ethyl	GC		509A		Note 3, p 25, Note 4, p 30
54. PCNB	GC		509A		Note 3, p 25
55. Perthane	GC		509A		Note 3, p 7
56. Prometon	GC			D3086	
57. Prometryn	GC				Note 3, p 83 Note 6, p S66
58. Propazine	GC				Note 3, p 83, Note 6, p S66
59. Propham	GC				Note 3, p 83 Note 6, p S66
60. Propoxur	TLC				Note 3, p 104, Note 6, p S64
61. Secbumeton	TLC				Note 3, p 94, Note 6 p S60
62. Simazine	TLC				Note 3, p 83 Note 6, p S66
63. Simetryn	GC				Note 3, p 104 Note 6, p S64
64. Swoden	GC		509A		Note 3, p 83 Note 6 p S66
					Note 3, p 7

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Table 7.4. (continued)

Parameter µg/L	Method	EPA[1,2]	Standard Methods 15th Ed	ASTM	Other
65 Simazine	TLC				Note 3, p 104, Note 6, p 564
66 2,4,5-T	GC		509B		Note 3, p 115, Note 4, p 35
67 2,4,5-TP (Silvex)	GC		509B		Note 3, p 115[7]
68 Terbuthylazine	GC				Note 3, p. 83, Note 6, p 564
69 Toxaphene	GC	608	509A	D3086	Note 3, p 7, Note 4, p 30
	GC/MS	625			
70 Trifluralin	GC		509A		Note 3, p 7

Table 7.4 Notes

[1] Pesticides are listed in this table by common name for the convenience of the reader. Additional pesticides may be found under Table 1C, where entries are listed by chemical na

[2] The full text of methods 608 and 625 are given at Appendix A, "Test Procedures for Analysis of Organic Pollutants," of this Part 136. The standardized test procedure to be used determine the method detection limit (MDL) for these test procedures is given at Appendix B, "Definition and Procedure for the Determination of the Method Detection Limit", of this Part

[3] "Methods for Benzidine, Chlorinated Organic Compounds, Pentachlorophenol and Pesticides in Water and Wastewater," U.S. Environmental Protection Agency, September, 1978 EPA publication includes thin-layer chromatography (TLC) methods

[4] "Methods for Analysis of Organic Substances in Water," U.S. Geological Survey, Techniques of Water-Resources Investigations, Book 5, Chapter A3 (1972)

[5] The method may be extended to include α-BHC, δ-BHC, endosulfan I, endosulfan II, and endrin. However, when they are known to exist, Method 608 is the preferred meth

[6] "Selected Analytical Methods Approved and Cited by the United States Environmental Protection Agency," Supplement to the Fifteenth Edition of *Standard Methods for the Examina of Water and Wastewater* (1981)

[7] Each analyst must make an initial, one-time, demonstration of their ability to generate acceptable precision and accuracy with Methods 608 and 625 (See Appendix A of this Part 136 accordance with procedures given in Section 8.2 of each of these methods. Additionally, each laboratory, on an ongoing basis, must spike and analyze 10% of all samples analyzed Method 608 or 5% of all samples analyzed with Method 625 to monitor and evaluate laboratory data quality in accordance with Sections 8.3 and 8.4 of these methods. When the recoven any parameter falls outside the warning limits, the analytical results for that parameter in the unspiked sample are suspect and cannot be reported to demonstrate regulatory compliar

Note.—These warning limits are promulgated as an "interim final action with a request for comments."

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Table 7.5. List of approved radiological test procedures
(40 CFR, Part 136, July 1, 1987)

Parameter and units	Methods	EPA [1]	Reference (method No. or page)		
			Standard Methods 15th Ed	ASTM	USGS [3]
1 Alpha-Total, pCi/l per liter	Proportional or scintillation counter	900.0	703	D1943-66	pp 75 and 78 [4]
2 Alpha-Counting error, pCi/l per liter	Proportional or scintillation counter	Appendix B	703	D1943-66	p. 79
3 Alpha-Counting error, pCi/l per liter	Proportional counter	900.0	703	D1890-66	pp 75 and 78 [4]
4 Beta-Counting error, pCi/l per liter	Proportional counter	Appendix B	703	D1890-66	p 79
5 (a) Radium-Total, pCi/l per liter	Proportional counter	903.0	705	D2460-70	
(b) ^{226}Ra, pCi/l per liter	Scintillation counter	903.1	706	D3454-79	p 81

Table IE Notes

[1] "Prescribed Procedures for Measurement of Radioactivity in Drinking Water," EPA-600/4-80-032 (1980 updated) U.S. Environmental Protection Agency, August 1980

[2] Fishman, M.J. and Brown, Eugene, "Selected Methods of the U.S. Geological Survey of Analysis of Wastewaters," U.S. Geological Survey Open-File Report 76-177 (1976)

[4] The method found on p 75 measures only the dissolved portion while the method on p 78 measures only the suspended portion. Therefore, the two results must be added to obtain the "total."

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and blank/spike control. In methods not using surrogates such as metals, anions, and wet chemical analysis, a blank and a blank/spike (laboratory control sample) shall be analyzed. For pesticide/PCB methods, surrogates are often used. However, problems have been noted in surrogate recovery for the dibutyl chlorinate typically used. For pesticide/PCB analysis, a blank and a blank/spike shall be analyzed with each batch as separate samples. A pesticide or a PCB shall be used as the spiking compound.

In Level C, when performing analyses for petroleum hydrocarbons; oil and grease; anions such as nitrates, sulfates and chloride; and other wet chemical methods, a matrix spike and matrix spike duplicate are required for every 20 samples of similar matrix. Similar matrix is defined as either soil or water from the same military base.

All methods specified require calibration. In keeping with the method calibration requirements, the following requirements are presented. For all semivolatile and volatile analysis by GC/MS, the current CLP calibration method shall be used. The current CLP criteria shall be used for frequency of calibration, for the system performance check compounds (SPCCs), and for the calibration check compounds (CCCs).

For other methods, a minimum of three different concentration standards for each analyte shall be analyzed for initial calibration. Calibration shall be checked every 12 h of operation and prior to sample analysis. The laboratory shall use the calibration check acceptance criteria specified by the method. The daily calibration acceptance criteria to be used for each method shall be documented in the laboratory QA plan or in the site-specific QA plan. The initial calibration curve shall be plotted and the correlation coefficient and response factors evaluated. The laboratory shall indicate in the laboratory QA plan or in the site-specific QA plan the acceptance criteria to be used for the initial calibration curve. The calibration shall include one standard at a concentration at the method detection limits. The calibration curve shall bracket all samples in the concentration range. If the samples are not within the calibration range, appropriate dilution shall be performed to bring the samples into the calibration range. The aforementioned calibration requirements shall be used for Levels C and E.

In Level C, a matrix spike and matrix spike duplicate are required for volatiles, semivolatiles, and all GC analysis for every 20 samples of similar matrix. For metals analysis, a duplicate and a matrix spike are required for every 20 samples of similar matrix.

For all GC methods used in level C QC, second column confirmation shall be used for all positive responses for the analytes of interest. In Level E, second column confirmation is not required.

In Level E, no matrix spikes or duplicates are required; only the initial and continuing calibration, method blank, and blank/spike are required.

7.2 DELIVERABLES

For Level D QC, a CLP data package shall be delivered. This shall include the summary package and the remainder of the package, which includes initial and continuing calibration, matrix spikes, matrix spike duplicates, blanks, duplicates, surrogate recoveries, chromatograms, mass spectra, and absorbance data. For methods which are not defined by CLP, the calibration information, method blanks, blank/spikes, the chromatograms, absorbance, matrix spikes, and matrix spike duplicates shall be reported. The control charts plotted per Sect. 4 associated with the blank/spikes shall be presented with the data.

For Level C QC, the method blanks, blank/spike, surrogates, matrix spikes, matrix spike duplicates, duplicates, and initial and continuing calibration data shall be reported. Table 7.6 lists the required deliverables. The forms referred to in Table 7.6 are from the current CLP for organics and metals/cyanide. The form numbers will be upgraded as new revisions occur in the CLP, which require changes in form content or numbering.

In Level E, the only information to be submitted is the sample data, method blank data, and the control chart from the blank/spike.

The deliverables shall be presented to the NCR. The forms shall be used when reporting any data in the MPR and in submitting the final data package prior to its inclusion in the appendix and summary tables of the final report. The final data deliverables shall be presented to the NCR at least three weeks prior to issuing the draft of the final report.

7.3 DATA VALIDATION

7.3.1 Level D Validation

At a minimum, the data generated from Level D will be validated per the CLP criteria as outlined in the following documents.

EPA, Hazardous Site Control Division, *Laboratory Data Validation Functional Guidelines for Evaluating Pesticides/PCB's Analyses*, R-582-5-5-01, May 28, 1985.

EPA, Hazardous Site Control Division, *Laboratory Data Validation Functional Guidelines for Evaluating Organics Analyses*, R-582-5-5-01, May 28, 1985.

EPA, Office of Emergency and Remedial Response, *Laboratory Data Validation Functional Guidelines for Evaluating Inorganics Analyses*, 1985.

Table 7.6. Data set deliverables for Level C QA

	Method requirements	Deliverables
Organics	- Method blank spikes with results and control charts. Run with each batch of samples processed.	Control chart
	- Results to be reported on CLP Form 1 or spreadsheet per Sect. 9. Sample results using CLP data flags.	Form 1 or Sect. 9 1/Sample chromatograms/and mass spectra
	- Surrogate recovery from samples reported on CLP Form 2. Surrogates to be used in volatiles, semivolatiles, pesticides/PCB. For volatiles by GC, the names of surrogates should be changed to reflect the surrogate used.	Form 2
	- Matrix spike/spike duplicate 1 spike and spike duplicate per 20 samples of similar matrix reported on Form 3.	Form 3
	- Method blank reported on CLP Form 4.	Form 4 or Sect. 9
	For volatiles by GC, a similar format will be used as CLP Form 4 for blanks.	
	- GC/MS tuning for volatiles/semivolatiles. Report results on Form 5.	Form 5
	- Initial calibration data reported on Form 6.	Form 6
	For volatiles by GC, the initial calibration data with response factors must be reported.	No Form
	For pesticide/PCB data Form 9 must be used for calibration data.	Form 9
	- Continuing calibration GC/MS data reported on Form 7.	Form 7
	For volatiles, GC data, the response factors and their percent differences from the initial must be reported.	No Form
	Internal Standard Area for Volatiles and Semivolatiles.	Form 8

Table 7.6. (continued)

	Method requirements	Deliverables
Organics (cont'd)	- For pesticides/PCB data, the CLP Form 9 must be presented.	Form 9
	No chromatograms or mass spectra are presented for calibration. These data should be filed in the laboratory and available if problems arise in reviewing/validating the data. The calibration information should be available for checking during on-site audits.	
	- Internal standard area for GC/MS analyses CLP Form VIII shall be supplied.	
	- Second column confirmation shall be done for all GC work when compounds are detected above reporting limits. Chromatograms of confirmation must be provided.	Chromatograms
Metals	- Level C, requirements	Deliverables
	- Sample results with CLP flagging system	CLP Form 1 or Sect. 9
	- Initial and continuing calibration	CLP Form 2, Part 1 only
	- Blanks 10% frequency	Form 3
	- Method blank taken through digestion (1/20 samples of same matrix)	Form 3 or Sect. 9
	- ICP interference check sample	Form 4
	- Matrix spike recovery (1 per 20 samples of similar matrix)	Form 5, Part 1
	- Postdigestion spike sample recovery for ICP metals. Only done if predigest spike recovery exceed CLP limits.	Form 5, Part 2 (never used for GFAA work)
	- Postdigest spike for GFAA	Recovery will be noted on raw data
	- Duplicates (1 per 20 samples will be split and digested as separate	Form 6 samples

Table 7.6. (continued)

	Method requirements	Deliverables
Metals (cont'd)	- Method blank spike information will be plotted on control chart, one per batch of samples processed.	Control chart
	- Standard addition. The decision process outlined in CLP page E-3 will be used to determine when standard additions are required.	Form 8
	Holding times	Form 10
Wet Chemistry	Level C	
	- Blank spike 1/batch	Control chart
	- Method Blank 1/batch	Report result No format
	- Sample results	Report result No format
	- Matrix spike/spike duplicate or calibration information	Report result if applicable
	- Calibration check report percent RSD or percent difference from initial calibration	Report percent or percent difference
		No format

7.3.2 Level C Data Validation Guidelines

Listed below are the validation criteria which will be utilized in evaluating the analytical data for a Level C QC site. For methods not listed here, a similar procedure will be submitted by the prime contractor and the laboratory which outlines validation of the holding times, initial calibration, continuing calibration, and blank-vs-sample results. The validation procedure will be approved by the NCR.

1. For Petroleum Hydrocarbons (418.1/SW-3540, EPA 418.1)

   Holding Times - Holding times are 28 days for water samples which are preserved and refrigerated. No holding times are cited for soils.

Calibration - Ensure that a three-to-five point curve bracketing the sample concentration is performed daily.

Blanks - A blank should be run with each batch. If the blank concentration exceeds the reporting limit, the reporting limit shall be raised and the data flagged as estimated (UJ).

2. Target Compound List (TCL) for VOAs (CLP Methods)

Holding Times - Samples must be analyzed within the holding times specified in Sect. 3 or the data should be marked as estimated (J).

GC/MS Tuning - Check that bromofluorobenzene tune is completed each 12-h shift of operation. Check that it meets the CLP criteria. Assure that each sample is associated with a tune.

Initial Calibration - The maximum relative standard deviation [(RSD) percent RSD] shall not be >30% for indicted CLP CCC. The maximum mean relative response factor (RRF) for SPCC shall be >0.300 (0.250 for bromoform). The SPCCs are chloromethane, 1,1-dichloroethane, bromoform, 1,1,2,2-tetrachloroethane, and chlorobenzene. The CCC compounds are vinyl chloride, 1,1-dichloroethene, chloroform, 1,2-dichloropropane, toluene, and ethylbenzene.

Continuing Calibration - The minimum response factor for the SPCC components for VOAs analyses shall not be <0.300 (0.250 for bromoform). The maximum response factor percent deviation for indicated CLP CCC components from the mean initial calibration response factor shall not exceed 25%. If these criteria are exceeded, a new calibration for the compound shall be employed.

Blank/Spike Control Samples - Any control sample which exceeds the internal QC limits set by the laboratory for a given sample matrix shall require all data from the associated batch of samples to be closely inspected. If no analytical problems are found, the data analyzed with the out-of-control point shall be discussed in the QC section of the MPR and final report. If problems are found in the analytical data, the samples associated with the batch shall be reanalyzed and the data from reanalysis reported. If holding times are exceeded in the reanalysis, both sets of data shall be presented.

If the blank/spike results are outside the internal laboratory limits and if the matrix spike results are outside the CLP limits, the laboratory will either reanalyze the samples within the holding times or the data will be flagged with an "R," and the data are not usable.

Surrogates - If surrogates exceed the CLP limits, the data shall be flagged that the surrogates exceeded limits.

Method Blanks - A method blank should be run each day following the Continuing Calibration Standard. Common laboratory solvents should not be found in the blank at levels over five times the detection

limits. Other compounds should not be found in the blank at levels exceeding the detection limits. If common contaminant compounds are detected in samples at a concentration of <10 times the concentration found in the blank, or other compounds at <5 times the concentration in the blank, report those compounds as not detected. Adjust the sample quantitation limit to the value reported in the samples and flag the limit as estimated (UJ).

Matrix Spike/Spike Duplicate - Ensure that 1 out of 20 samples has been spiked in duplicate. The recoveries shall meet the CLP criteria. If the recoveries do not meet the criteria, examine the blank spike data. If the blank spike data exceed the limits and the matrix spikes exceed limits, the data shall be flagged as unusable (R). If the blank spike data from the batch are satisfactory, the data is usable, and the low recovery is discussed in the final report QA/QC and in the QC report sent to the NCR.

Field Trip and Equipment Blanks - If contaminant analytes are detected in samples at concentrations of <5 times the concentration found in the highest associated blank, the results are considered suspect and are reported as estimated.

3. TCL Semivolatile Organics (CLP Methods)

Holding Times - Samples must be extracted within 7 days of collection and analyzed within 40 days of extraction. Any samples which do not meet these requirements must be flagged as estimated.

GC/MS Tune - Make certain that a decafluorotriphenylphosphine tune is completed every 12 h of sample analysis, that each sample is associated with a tune, and that each tune meets CLP requirements. Data are not reported if the instrument does not meet tune.

Initial Calibration - Ensure that a 5-point curve has been completed. The RRF of the BNA compounds shall be a minimum of 0.050 for the SPCC listed in the current revision of the CLP. The maximum RSD for the CCC listed in the CLP procedure is 30.0%. The minimum RRF for the SPCC is 0.050, and the maximum percent difference for the CCC is 25%. If these limits are exceeded, a new calibration curve shall be generated.

Continuing Calibration - The continuing calibration check will be performed once every 12 h during operation. The minimum RRF for the SPCC is 0.05, and the maximum percent difference from the initial calibration shall not exceed 25% for the CCC. If these limits are exceeded, a new calibration curve shall be generated.

Blank/Spike Control Samples - Any control sample which exceeds the internal QC limits set by the laboratory for a given sample matrix shall require all data from the associated batch of samples to be closely inspected. If no analytical problems are found, the data and the out-of-control point shall be discussed in the QC section of

the report. If problems are found in the analytical data, the samples associated with the batch shall be reanalyzed and the data from reanalysis reported. If holding times are exceeded in the reanalysis, both sets of data shall be presented.

If the blank/spike results are outside the internal laboratory limits and if the matrix spike results are outside the CLP limits, the laboratory will either reanalyze the samples or the data will be flagged with an "R," and the data is not usable.

Surrogates - If surrogates exceed the CLP limits, the data shall be flagged that the surrogates exceeded limits.

Blanks - A method blank should be run each day following the Continuing Calibration Standard. Phthalate should not be found in the blank at levels over five times the detection limits. Other compounds should not be found in the blank at levels exceeding the detection limits. If common contaminant compounds are detected in samples at a concentration of <10 times the concentration found in the blank, or other compounds at <5 times the concentration in the blank, report those compounds as not detected. Adjust the sample quantitation limit to the value reported in the samples and flag the limit as estimated (UJ).

Matrix Spike/Spike Duplicate - Ensure that 1 out of 20 samples has been spiked in duplicate. The recoveries should meet the CLP criteria. If the recoveries do not meet the criteria, examine the blank spike data. If the blank spike data exceed the limits and the matrix spikes exceed limits, the data shall be flagged as unusable (R). If the blanks spike data from the batch is satisfactory, the data are usable, and the low recovery is discussed in the final QC report sent to the Analytical Environmental Support Section.

4. Metals

Holding Times - Samples must be analyzed within six months, except mercury shall be analyzed in 28 days from sample collection.

ICP Initial Calibration - A calibration blank and at least one standard must be analyzed daily. An initial calibration verification standard must be within 90 to 110% recovery or the samples should be reanalyzed. If it is not possible to perform reanalysis, the data are rejected and flagged with an "R."

AA Calibration - Calibration blank and at least three standards shall be used in establishing the curve prior to sample analysis. A curve shall be analyzed each day prior to sample analysis.

Calibration Verification - Verification using a standard obtained from a source other than that of the initial calibration shall be used and the result shall be within 90 to 110% of the true value for

both ICP and AA work. Calibration verification shall be done at a minimum frequency of 10% or every 2 h, whichever is more frequent, and shall be done at the end of the analytical run.

Method Blanks - At least one preparation blank shall be prepared with each batch of samples. The blanks shall contain less than the detection limit for all analytes. If the concentration of the associated blanks is above the detection limit and if the lowest analyte concentration is <10 times the blank, reanalysis of the sample must occur. If reanalysis is not done, the data shall be reported and flagged as estimated. The blank shall never be subtracted from the sample.

Field and Equipment Blanks - If contaminant analytes are detected in samples at concentrations of <5 times the concentration found in the highest associated blank, the results are considered suspect and are reported as estimated.

Blank/Spike Laboratory Control Samples - Any laboratory control sample which exceeds the internal QC limits set by the laboratory for a given sample matrix shall require all data from the associated batch of samples to be closely inspected. If no analytical problems are found, the data and out-of-control point shall be discussed in the QC section of the report. If problems are found in the analytical data, the samples associated with the batch shall be reanalyzed and the data from reanalysis reported. If holding times are exceeded in the reanalysis, both sets of data shall be presented. A discussion of data reported when the blank/spike laboratory control sample is out of control shall be presented in the QC section of both the final report and the MPR.

If the blank/spike results are outside the internal laboratory limits and if the matrix spike results are outside the CLP limits, the laboratory will either reanalyze the samples or the data will be flagged with an "R," and the data are not usable.

BLANK SHEET

8. MAINTAINING LABORATORY APPROVAL

Once a laboratory has received Navy approval to begin analysis of samples, maintaining that approval requires adherence to the QA plan and reporting of QA-related information. The performance and reporting requirements outlined below are essential to ensuring that data of known and defensible quality are being generated throughout the course of a site investigation. Topics covered include control samples, control charts, out-of-control events, corrective action reports, significant changes in the QA plan, and other reporting requirements.

8.1 MONTHLY PROGRESS REPORT

The primary means of communication from the laboratories to the NCR will be the MPR to be submitted by the laboratories to the NCR on the 15th of each month in which work for the Navy is performed. The following information is to be included in the MPR.

1. Site name and contract number.

2. Numbers, types and locations of samples collected and analyzed for Navy project only.

3. Data for blanks, spikes, laboratory duplicates and controls related to Navy samples.

4. New methods used for analysis and changes in old methods.

5. Copies of all control charts pertinent to Navy samples and to which results have been added over the reporting period.
6. Summaries of out-of-control incidents during the reporting period, including references to documentation and corrective action reports.

7. Descriptions of and justifications for significant changes in the QA.

8. Changes in LQAC personnel and other key technical personnel; resumes of new personnel must be submitted.

9. Completed sample data.

Much of the information presented in an MPR is incremental in nature and relates to changes and findings since the previous MPR.

1. Control charts from the minimizing control charts program and any additional control charts from monitoring matrix spikes, duplicates, or other QC parameters.

2. Personnel changes relating to QA responsibilities.

3. Method changes (e.g., a minor modification with an attached EPA variance).

4. Procedural changes in establishing control limits and/or the preparation and use of control charts.

Since the first such report for each laboratory has no precedent, more explanation and detail may be necessary; subsequent MPRs will likely not require as much detail in some areas.

8.2 FINAL REPORT

A draft of the final report shall be reviewed by the NCR prior to its release. This report is the final deliverable from the engineering subcontractor. An outline for a typical report is as follows.

1. Site name and Navy contract number.

2. Foreword--signed by those with major responsibilities for the QA program and by project management.

3. Executive Summary--brief review of the report.

4. Table of Contents--with specificity at approximately the same level as the Table of Contents in this Navy document.

5. Introduction--summarize the Navy field sites of interest, when the study occurred (dates of sampling, dates of analysis) and the objectives of the QA plan as they relate to the study.

6. Data Summary--summarize the results on a site-by-site basis.

7. Other Information--present any other information requested in the statement of work such as risk assessment, recommendation to perform more site characterization, or recommend site closure. This information was specified prior to beginning work and is directed by the Navy EIC.

8. The final report shall present the findings from the analytical, geological, and hydrogeological studies. The summary of analytical data will exclude non-detected compounds. No subtraction of blanks is allowed. Data will be flagged if blank contamination occurs. All data flags will follow the result in the summary.

9. QC Summary--the QC summary section will include a discussion of are data which flagged. Flagged data defined as data for which trip, field, or laboratory blanks were contaminated, matrix spike/spike duplicates exceed limits, calibration criteria are not met, and laboratory controls exceed limits. The QC summary will also discuss the results of laboratory blanks, matrix spikes/spike duplicates,

duplicates, control charts, surrogate holding times, field blanks, trip blanks, rinsates, and field duplicates. This section will also discuss precision, accuracy, and completeness.

10. Appendices--the appendices of the report shall include all field and analytical data. One appendix shall contain field logs and forms. A second appendix shall contain the laboratory data of each sample. These data shall be presented in a spreadsheet similar to the Format Section of this report. All trip, field, and laboratory blanks shall be marked so that each sample can be associated with the appropriate blanks.

    A third appendix shall include the method blank spike control charts, surrogate recoveries, matrix spike and duplicate, field, and laboratory duplicates for all spike samples.

8.3 FINAL QC DATA REPORT

A QC data report shall be sent to the NCR. This report shall contain the following.

For Level D QC, the contractor shall submit a subset of data from the CLP data packages. For 20% of the water and 20% of the soil samples, the subcontractor shall submit the full CLP package.

For Level C QC, the deliverables listed in Table 7.7 will be presented.

For Level E QC, the initial and continuing calibration forms, method blank, and blank spike control chart are required.

The report shall indicate the duration and location of storage for the data. The stored data consists of all raw data, QC charts, corrective action, logs, sample lists, COC information, notebooks, work sheets, automated data processing system output, and calibration.

The report shall be delivered to the NCR three weeks prior to the final report.

BLANK SHEET

9. DATA FORMAT

The data format refers to the format in the final report. The contractor may use its own format in the body of the report. However, in the appendices (which contain sample and blank data) a spreadsheet type of format may be used, or the CLP forms for reporting samples and equipment, trip, field, and method blanks may be used. The spreadsheet format allows for more samples per page and for more information on blanks and their association with samples. The spreadsheet format is not meant to be a rigid form. The information listed in Fig. 9.1 must be present. The contractor may add other information which will assist it in review. For calibration, tuning, spikes, surrogates, and duplicates, the current CLP forms are required for data presentation. If any other format is to be used, this shall be discussed with the NCR.

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Sample Number	J25019	JS5020
Date Sampled	03-18-87	03-18-87
Sample Prep..Date	11-25-87	11-25-87
Sample Analysis Date	11-26-87	11-26-87
Sample Numbers of Associated Analytes, Field, Trip, and Equipment Blanks	J4455667	L4455667

	Detection limit	Sample	Results
VOLATILE ORGANIC COMPOUNDS (µg/kg)			
TETRACHLOROETHANE	5	50	50
CHLOROBENZENE	5		
SEMIVOLATILE ORGANIC COMPOUNDS (µg/kg)			
BIS(2-ETHYLHEXYL) PHTHALATE	330		750
2-METHYLNAPTHALENE	330		2500
INORGANIC COMPOUNDS (mg/kg)			
LEAD	10	360	25
HYDROCARBONS (2)			
PETROLEUM HYDROCARBON	1	0.611	0.268
OIL AND GREASE	1		

Sample Results

Analyte

Note: Petroleum hydrocarbon, oil, and grease results recorded in percent.

Fig. 9.1. Example of data format for final report.

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GLOSSARY

Accuracy - The nearness of a result or the mean of a set of results to the true or accepted value.

Analyte - A chemical component of a sample to be determined or measured.

Analytical Method - Defines the samples preparation and instrumentation procedures or steps that must be performed to estimate the quantity of analyte in a sample.

Analytical Spike - The furnace postdigestion spike. The addition of a known amount of standard after digestion.

Background Correction - A technique to compensate for variable background contribution to the instrument signal and the determination of trace metals.

Calibration - The establishment of an analytical curve based on the absorbance, emission intensity, or other measured characteristic of known standards. The calibration standards must be prepared using the same type of acid or concentration of acids as used in the sample preparation.

Calibration Blank - A volume of acidified deionized/distilled water.

Comparability - is a qualitative parameter expressing the confidence with which one data set can be compared with another. Sample data should be comparable with other measurement data for similar samples and sample conditions.

Completeness - Completeness is defined as the percentage of measurements made which are judged to be valid measurements. The completeness goal is to generate sufficient amount of valid data based on project needs.

Continuing Calibration - Analytical standard run every ten analytical samples or every 2 h, whichever is more frequent, to verify the calibration of the analytical system.

Control Limits - A range within which specified measurement results must fall to be compliant. Control limits may be mandatory, requiring corrective action if exceeded, or advisory, requiring that noncompliant data be flagged.

Correlation Coefficient - A number (r) which indicates the degree of dependence between two variables (concentration - absorbance). The more dependent they are, the closer the value to one. Determined on the basis of the least squares line.

Data Quality Objectives - are qualitative and quantitative statements which specify the quality of the data required to support decision during

GLOSSARY (continued)

remedial response activities. Data quality objectives are determined based on the end uses of the data to be collected.

Detection Limit - The minimum concentrations which must be accurately and precisely measured by the laboratory and/or specified in the quality assurance plan.

Dissolved Metals - Analyte elements which have not been digested prior to analysis and which will pass through a 0.45-μm filter.

Duplicates - Identical splits of individual samples which are analyzed by the laboratory to test for method reproducibility. In this case, samples are split in the laboratory.

Equipment Rinsates - The final analyte-free water rinse from equipment cleaning collected daily during a sampling event.

Field Blanks - Blanks are collected and analyzed to determine the level of contamination introduced into the sample due to sampling technique. They may consist of the source water used in decontamination and steam cleaning. At minimum, one sample from each event and each source of water must be collected and analyzed.

Field Duplicates/Splits - Samples that have been divided into two or more portions while in the field. Each portion is then carried through the remaining steps in the measurement process. A sample may be replicated in the field or at different points in the analytical process. For field replicated samples, precision information would be gained on homogeneity, handling, shipping, storage, preparation, and analysis.

Replicate samples divided into two portions and sent to different laboratories and subjected to the same environmental conditions and steps in the measurement process as the split samples.

Instrument Detection Limit - is defined in several ways. For example, (1) that concentration of analyte which produces an output signal twice the root mean square of the background noise may be determined under ideal conditions or (2) determined by multiplying by 3 the standard deviation obtained for the analysis of a standard solution (each analyte in reagent water) at a concentration of 3x-5x instrument detection limit on three nonconsecutive days with seven consecutive measurements per day.

Internal Standards - Compounds added to every standard, blank, matrix spike, matrix spike duplicate, sample (for volatile), and sample extract (for semivolatile) at a known concentration prior to analysis. Internal standards are used as the basis for quantitation of the target compounds.

GLOSSARY (continued)

Laboratory Control Sample - A control sample of known composition. Aqueous and solid laboratory control samples are analyzed using the same sample preparation, reagents, and analytical methods employed for samples received.

Laboratory Quality Assurance Coordinator - An employee of a laboratory with no analysis or production responsibilities and who implements QA and QC. This person is responsible for ensuring all quality problems are resolved.

Matrix - The predominant material comprising the sample to be analyzed. The most common matrices are water, soil/sediment, and sludge.

Matrix Spike - An aliquot of a matrix (water or soil) spiked with known quantities of compounds and subjected to the entire analytical procedure in order to indicate the appropriateness of the method for the matrix by measuring recovery.

Matrix Spike Duplicate - A second aliquot of the same matrix as the matrix spike that is spiked in order to determine the precision of the method.

Method Blank - A blank sample run to ensure reported analytical results are not the results of laboratory contamination.

Method Blank/Spike - Is the distilled and/or deionized water for soil or sand spiked with known compounds or elements. The method blank as defined by Contract Laboratory Protocol for organics and the laboratory control sample as defined by Contract Laboratory Protocol maybe use as the method blank/spike in the Navy Installation Restoration Program.

Method Detection Limits - Minimum concentrations of a substance that can be measured and reported with 99% confidence that the value is above zero. The sample is carried through the entire method under ideal conditions.

Method of Standard Additions - The addition of three increments of a standard solution (spikes) to sample aliquots of the same size. Measurements are made on the original and after each addition. The slope, x-intercept, and y-intercept are determined by least-squares analysis. The analyte concentration is determined by the absolute value of the x-intercept. Ideally, the spike volume is low relative to the sample volume (~10% of the volume). Standard addition may counteract matrix effects; it will not counteract spectral effects. It is also referred to as standard addition.

Out of Control - One or more of several conditions relating to the plotting of control data and indicating unacceptable results.

GLOSSARY (continued)

Percent Solids - The proportion of solid in a soil sample determined by drying an aliquot of the sample.

Precision - Measure of the reproducibility of a set of replicate results among themselves or the agreement among repeat observations made under the same conditions.

Preparation Blank (Reagent Blank, Method Blank) - An analytical control that contains distilled, deionized water and reagents, which is carried through the entire analytical procedure (digested and analyzed). An aqueous method blank is treated with the same reagents as a sample with a water matrix; a solid method blank is treated with the same reagents as a soil sample.

Purge and Trap - An analytical technique used to isolate volatile (purgable) organics by stripping the compounds from water or soil by a stream of inert gas, trapping the compounds on a porous polymer trap, and thermally desorbing the trapped compounds onto the gas chromatographic column.

Quality Assurance - A planned system of activities (program) whose purpose is to provide assurance of the reliability and defensibility of the data.

Quality Control - A routine application of procedures for controlling the monitoring process. QC is the responsibility of all those performing the hands-on operations in the field and in the laboratory.

Reagent Water - Water in which an analyte is not observed at or above the minimum quantitation limit of the parameters of interest.

Recovery - Usually expressed as a percent. The numerical ratio of the amount of analyte measured by the laboratory method divided by the known amount of analyte added to the matrix (i.e., spiked sample) to be analyzed.

Reporting Detection Limits - The same as method detection limits with consideration given for practical limitation such as sample size, matrix interferences, and dilutions.

Representativeness - Expresses the degree to which sample data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, or an environmental condition. Representativeness is a qualitative parameter which is most concerned with the proper design of the sampling program.

Sample Holding Times - Times used to ascertain the validity of results based on the holding time of the sample from time of collection to time of analysis or sample preparation. Holding times may vary depending on the analysis, EPA regional preference, etc.

GLOSSARY (continued)

Semivolatile Compounds - Compounds amenable to analysis by extraction of the sample with an organic solvent. Used synonymously with base neutral acid or extractable compounds.

Serial Dilution - The dilution of a sample by a known factor. When corrected by the dilution factor, the diluted sample must agree with the original undiluted sample within specified limits. Serial dilution may reflect the influence of interferents.

Spikes - Known amounts of specific chemical constituents added by the laboratory to selected samples to test the appropriateness and recover efficiencies of specific analytical methods within the actual sample matrices.

Standard Deviation - The square root of the variance of a set of values.

Surrogates - Compounds added to every blank, sample, matrix spike, matrix spike duplicate, and standard and used to evaluate analytical efficiency of the method by measuring recovery. Surrogates are brominated, fluorinated, or isotopically labelled compounds not expected to be detected in environmental media. These are used typically in organic methods.

Tentative Identified Compounds - Compounds detected in samples that are not target compounds, internal standards or surrogate standards. Up to 30 peaks (those greater than 10% of peak areas or heights of nearest internal standards) are subjected to mass spectral library searches for tentative identification.

Total Metals - Analyte elements which have been digested prior to analysis.

Variance - The sum of the squares of the difference between the individual values of a set and the arithmetic mean of the set, divided by one less than the number of values.

Volatile Compounds - Compounds amendable to analysis by the purge and trap techniques. Used synonymously with purgable compounds.

Data Qualifiers' Definitions as defined by the Contract Laboratory Protocol for Organic Analysis

U - Indicates compound was analyzed for but not detected. The sample quantitation limit must be corrected for dilution and for percent moisture.

J - Indicates an estimated value. This flag is used either when estimating a concentration for tentatively identified compounds where a 1:1 response is assumed, or when the mass spectral data indicate the presence of a compound that meets the identification criteria but the result is less than the sample quantitation limit but greater than zero.

GLOSSARY (continued)

C - This flag applies to pesticide results where the identification has been confirmed by gas chromatography/mass spectrometry. Single component pesticides ≥10 ng/ul in the final extract shall be confirmed by gas chromatography/mass spectrometry.

B - This flag is used when the analyte is found in the associated blank as well as in the sample. It indicates possible/probable blank contamination and warns the data user to take appropriate action. This flag must be used for a TIC as well as for a positively identified TCL compound.

E - This flag identifies compounds whose concentrations exceed the calibration range of the gas chromatography/mass spectrometry instrument for that specific analysis. This flag will not apply to pesticides/PCBs analyzed by GC/EC methods. If one or more compounds have a response greater than full scale, the sample or extract must be diluted and reanalyzed. If the dilution of the extract causes any compounds identified in the first analysis to be below the calibration range in the second analysis, then the results of both analyses shall be reported.

D - This flag identifies all compounds identified in an analysis at a secondary dilution factor. If a sample or extract is reanalyzed at a higher dilution factor, as in the "E" flag above, the "DL" suffix is appended to the sample number on the Form I for the diluted sample, and all concentration values reported on that Form I are flagged with the "D" flag.

A - This flag indicates that a TIC is a suspected aldol-condensation product.

X - Other specific flags and footnotes may be required to properly define the results. If used, they must be fully described and such description attached to the Sample Data Summary Package and the Case Narrative. If more than one is required, use "Y" and "Z," as needed. If more than five qualifiers are required for a sample result, use the "X" flag to combine several flags, as needed. For instance, the "X" flag might combine the "A," "B," and "D" flags for some sample.

R - Quality control indicates that data are not usable (compound may or may not be present). Resampling and reanalysis are necessary for verification.

Q - No analytical result.

GLOSSARY (continued)

Inorganic Data Qualifiers

E - The reported value is estimated because of the presence of interference. An explanatory note must be included under Comments on the cover page (if the problem applies to all samples) or on the specific FORM I-IN (if it is an isolated problem).

M - Duplicate injection precision not met.

N - Spiked sample recovery not within control limits.

S - The reported value was determined by the Method of Standard Additions.

W - Postdisgestion spike for Furnace Atomic Absorption analysis is out of control limits (85-115%), while sample absorbance is less than 50% of spike absorbance.

* - Duplicate analysis not within control limits.

+ - Correlation coefficient for the Method of Standard Addition is less than 0.995.

M (Method) Qualifier

"P" for ICP

"A" for Flame AA

"F" for Furnace AA

"CV" for Manual Cold Vapor AA

"AV" for Automated Cold Vapor AA

"AS" for Semiautomated Spectrophotometric

"C" for Manual Spectrophotometric

"T" for Titrimetric

"NR" if the analyte is not required to be analyzed

BLANK SHEET

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KEY CONTACTS FOR THE NAVY INSTALLATION RESTORATION PROGRAM

Mailing Address for Premium Transport:

Martin Marietta Energy Systems, Inc.
Highway 58, Building K-1004A, Drop A20
Oak Ridge, TN 37830

Mailing Address for Regular Mail:

Martin Marietta Energy Systems, Inc.
P.O. Box 2003
Oak Ridge, TN 37831-7440

Mitzi S. Miller, Program Manager (615) 576-2361

Duties: Organization and assignment of projects to staff, laboratory audits, QA/QC review, work plan review, etc., for all branches of the military.

Henry H. Beiro, Project Manager (615) 576-1568

Duties: Sampling plan review, work plan review, field audits, etc., for all branches of the military.

Ahmed A. Halouma, Project Manager (615) 574-7251)

Duties: Laboratory audits, QA/QC review, work plan review, etc., for Army and Corps of Engineers.

Nile A. Luedtke, Project Manager (615) 574-8752

Duties: Laboratory audits, QA/QC review, work plan review, etc., for the Southern Division of the Navy and asbestos site survey projects, plus backup for other project managers, as necessary.

Gloria J. Mencer, Navy Project Manager (615) 576-1570

Duties: Laboratory auidts, QA/QC review, work plan review, etc., for the Navy.

Margaret P. Ogawa, Secretary (574-0400)

Richard D. Westmoreland, Project Manager (615) 574-8072

Duties: Laboratory audits, QA/QC review, work plan review, etc., for the Air Force and Air National Guard; particularly those projects handled by HAZWRAP.

Dennis L. Forsberg (615) 574-8515

Duties: Sampling plan review, work plan review, field audits, etc., for all branches of the military.

NESSA PROGRAM CONTACTS

Anthony R. Sturtzer (805) 982-3449

Navy Energy and Environmental Support Activity
Code 112E
Building 835
Port Hueneme, California 93043-5014

Duties: Program management of the Installation Restoration Program. Serves as the key contact for NEESA, EFD, and NCR.

Jerry Lionelli

Duties: Assist in the program management of the Installation Restoration Program.

Stephen Eikenberry, Manager

Duties: Manager of the Installation Restoration Program.

APPENDIX F
EXAMPLE DATA DELIVERABLE PACKAGE (EXPLOSIVES)

APPENDIX F
EXAMPLE DATA DELIVERABLE PACKAGE (EXPLOSIVES)

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3F

SOIL MATRIX SPIKE/MATRIX SPIKE DUPLICATE RECOVERY FORM

Name: SWL-TULSA Contract:

Code: SWOK Case No: SDG No:

Client Sample ID:

COMPOUND	SPIKE ADDED (ug/Kg)	SAMPLE CONCENTRATION (ug/Kg)	MS CONCENTRATION (ug/Kg)	MS % REC #	QC LIMITS REC.
HMX	16000	0.0	14600	91	54-128
RDX	6250	0.0	5430	87	66-109
TNB	5750	0.0	5340	93	57-130
TETRYL	3825	0.0	3270	85	32-119
DNB	4875	0.0	4450	91	74-122
TNT	5000	0.0	4580	92	44-124
NB	3500	0.0	3390	97	70-135
26DNT	3875	0.0	3630	94	58-125
24DNT	2400	0.0	2350	98	59-120
2NT	7250	0.0	6810	94	79-124
4NT	4750	0.0	4420	93	74-128
3NT	5000	0.0	4640	93	79-121
2ADNT	17200	0 0	16800	98	ND
4ADNT	18800	0.0	18300	97	ND

f MS analyte recoveries out of QC Limits = 0

COMPOUND	SPIKE ADDED (ug/Kg)	MSD CONCENTRATION (ug/Kg)	MSD % REC #	% RPD#	QC LIMITS RPD	QC LIMITS REC.
HMX	16000	14600	91	0	30	54-128
RDX	6250	5420	87	0	30	66-109
TNB	5750	5310	92	.6	30	57-130
TETRYL	3825	3470	91	6	45	32-119
DNB	4875	4480	92	1	30	74-122
TNT	5000	4610	92	1	35	44-124
NB	3500	3380	97	0	30	70-135
26DNT	3875	3690	95	2	35	58-125
24DNT	2400	2450	102	4	35	59-120
2NT	7250	6770	93	1	35	79-124
4NT	4750	4440	93	0	35	74-128
3NT	5000	4640	93	0	35	79-121
2ADNT	17200	17000	99	1.2	ND	ND
4ADNT	18800	18600	99	1.6	ND	ND
		0				

No of MSD analyte recoveries out of QC Limits = 0

No of RPD's out of QC Limits = 0

3F

SOIL MATRIX SPIKE/MATRIX SPIKE DUPLICATE RECOVERY FORM

Lab Name: SWL-TULSA Contract:

Lab Code: SWOK Case No: SDG No:

Client Sample ID:

COMPOUND	SPIKE ADDED (ug/Kg)	SAMPLE CONCENTRATION (ug/Kg)	MS CONCENTRATION (ug/Kg)	MS % REC #	QC LIMITS REC.
HMX	16000	0.0	14500	91	54-128
RDX	6250	0.0	5370	86	66-109
TNB	5750	0.0	5350	93	57-130
TETRYL	3825	0.0	3280	86	32-119
DNB	4875	0.0	4460	91	74-122
TNT	5000	0.0	4610	92	44-124
NB	3500	0.0	3380	97	70-135
26DNT	3875	0.0	3800	98	58-125
24DNT	2400	0.0	2490	104	59-120
2NT	7250	0.0	6720	93	79-124
4NT	4750	0.0	4270	90	74-128
3NT	5000	0.0	4620	92	79-121
2ADNT	17200	0.0	17000	99	ND
4ADNT	18800	0.0	18600	99	ND

Number of MS analyte recoveries out of QC Limits = 0

COMPOUND	SPIKE ADDED (ug/Kg)	MSD CONCENTRATION (ug/Kg)	MSD % REC #	% RPD#	QC LIMITS RPD	QC LIMITS REC.
HMX	16000	14700	92	1	30	54-128
RDX	6250	5540	89	3	30	66-109
TNB	5750	5430	94	1.5	30	57-130
TETRYL	3825	3260	85	1	45	32-119
DNB	4875	4520	93	1	30	74-122
TNT	5000	4650	93	1	35	44-124
NB	3500	3430	98	1	30	70-135
26DNT	3875	3710	96	2	35	58-125
24DNT	2400	2440	102	2	35	59-120
2NT	7250	6770	93	1	35	79-124
4NT	4750	4240	89	1	35	74-128
3NT	5000	4660	93	1	35	79-121
2ADNT	17200	17200	100	1.2	ND	ND
4ADNT	18800	18800	100	1.1	ND	ND
		0				

No of MSD analyte recoveries out of QC Limits = 0

No of RPD's out of QC Limits = 0

3G

EXPLOSIVES SOIL LABORATORY CONTROL SPIKE RECOVERY

Name: SWL-TULSA Contract:

Code: SWOK Case No: SDG No:

Client Sample ID: LD0701SB A&B

COMPOUND	SPIKE ADDED (ug/Kg)	SAMPLE CONCENTRATION (ug/Kg)	LCS CONCENTRATION (ug/Kg)	LCS % REC #	QC LIMITS REC.
HMX	16000	0.0	14700	92	57-137
RDX	6250	0.0	5730	92	77-113
TNB	5750	0.0	5290	92	64-125
TETRYL	3825	0.0	3030	79	23-124
DNB	4875	0.0	4440	91	82-118
TNT	5000	0.0	4530	91	75-118
NB	3500	0.0	3380	97	69-143
26DNT	3875	0.0	3610	93	78-117
24DNT	2400	0.0	2330	97	74-126
2NT	7250	0.0	6710	93	78-123
4NT	4750	0.0	4370	92	79-125
3NT	5000	0.0	4640	93	78-121
2ADNT	17200	0.0	16400	95	63-142
4ADNT	18800	0.0	18000	96	68-157

of Analyte recoveries out of QC Advisory Limits= 0

FORM III

3G

EXPLOSIVES SOIL LABORATORY CONTROL SPIKE RECOVERY

Lab Name: SWL-TULSA Contract:

Lab Code: SWOK Case No: SDG No:

Client Sample ID: LC0701SB A&B

COMPOUND	SPIKE ADDED (ug/Kg)	SAMPLE CONCENTRATION (ug/Kg)	LCS CONCENTRATION (ug/Kg)	LCS % REC	#	QC LIMITS REC.
HMX	16000	0.0	14600	91		57-137
RDX	6250	0.0	5780	92		77-113
TNB	5750	0.0	4880	85		64-125
TETRYL	3825	0.0	357	9	*	23-124
DNB	4875	0.0	4470	92		82-118
TNT	5000	0.0	5100	102		75-118
NB	3500	0.0	3590	103		69-143
26DNT	3875	0.0	3610	93		78-117
24DNT	2400	0.0	2350	98		74-126
2NT	7250	0.0	6740	93		78-123
4NT	4750	0.0	4400	93		79-125
3NT	5000	0.0	4640	93		78-121
2ADNT	17200	0.0	16800	98		63-142
4ADNT	18800	0.0	18500	98		68-157

# of Analyte recoveries out of QC Advisory Limits= 1

FORM III

8E

ANALYTICAL SEQYUENCE SUMMARY

Name: SOUTHWEST LABORATORY OF OKLAHOMA

Lab Code: SWOK
Instrument ID: IN13

Case No:
GC Column ID: ODS-10

SDG:

Dates of Analyses: 07/11/96 to 07/11/96

CLIENT SAMPLE NO.	LAB SAMPLE ID	DATE ANALYZED	TIME ANALYZED
EXPLOSIVE L1A STANDARD	15-211-5	07/11/96	1709
EXPLOSIVE L2A STANDARD	15-211-5	07/11/96	1737
EXPLOSIVE L3A STANDARD	15-211-5	07/11/96	1806
EXPLOSIVE L4A STANDARD	15-211-5	07/11/96	1835
EXPLOSIVE L5A STANDARD	15-211-5	07/11/96	1904
EXPLOSIVE L1B STANDARD	15-218-5:IA1	07/11/96	1933
EXPLOSIVE L2B STANDARD	15-217-5:IA2	07/11/96	2001
EXPLOSIVE L3B STANDARD	15-216-5:IA3	07/11/96	2030
EXPLOSIVE L4B STANDARD	15-215-5:IA4	07/11/96	2059
EXPLOSIVE L5B STANDARD	15-214-5:IA5	07/11/96	2127
BL0701SA	BL0701SA	07/11/96	2156
LC0701SA	LC0701SA	07/11/96	2253
LD0701SB	LD0701SB	07/12/96	0020
1001-DP01-9-12	26181.01	07/12/96	0048
1001-DP01-9-12MSA	26181.02MSA	07/12/96	0117
EXPLOSIVE L3A STD	15-209-5	07/12/96	0146
EXPLOSIVE L3B STD	15-216-5:CA1	07/12/96	0214
1001-DP01-9-12MSB	26181.02MSB	07/12/96	0243
1001-DP01-9-12MSDA	26181.03MSDA	07/12/96	0312
1001-DP01-9-12MSDB	26181.03MSDB	07/12/96	0341
1001-DP01-15-18	26181.04	07/12/96	0409
1001-DP01-18-21	26181.05	07/12/96	0438
1001-DP01-9-12	26181.07	07/12/96	1003
1001-DP02-15-18	26181.08	07/12/96	1032
1001-DP02-18-21	26181.09	07/12/96	1100
EXPLOSIVE L3A STD	15-209-5	07/12/96	1129
EXPLOSIVE L3B STD	15-216-5:CA2	07/12/96	1158
1001-DP03-9-12	26181.11	07/12/96	1226
1001-DP03-15-18	26181.12	07/12/96	1255
1001-DP03-18-21	26181.13	07/12/96	1324
1001-DP04-9-12	26181.15	07/12/96	1352
1001-DP04-15-18	26181.16	07/12/96	1421
1001-DP04-24-27	26181.17	07/12/96	1450
1001-DP05-9-12	26181.19	07/12/96	1519
1001-DP05-12-15	26181.20	07/12/96	1547

FORM VIII

8E
ANALYTICAL SEQYUENCE SUMMARY

Name: SOUTHWEST LABORATORY OF OKLAHOMA

Lab Code: SWOK
Instrument ID: IN13

Case No:
GC Column ID: ODS-10

SDG:

Dates of Analyses: 07/11/96 to 07/11/96

CLIENT SAMPLE NO.	LAB SAMPLE ID	DATE ANALYZED	TIME ANALYZED
EXPLOSIVE L3A STD	15-209-5	07/12/96	1616
EXPLOSIVE L3B STD	15-216-5:CA3	07/12/96	1645
1001-DP05-15-18	26181.21	07/12/96	1714
1001-DP06-6-9	26181.23	07/12/96	1742
1001-DP05-12-15	26181.24	07/12/96	1811
1001-DP06-18-21	26181.25	07/12/96	1840
EXPLOSIVE L3A STD	15-209-5	07/12/96	1909
EXPLOSIVE L3B STD	15-216-5:CA4	07/12/96	1942
BL0701SB	BL0701SB	07/12/96	2011
LC0701SBA	LC0701SBA	07/12/96	2108
LD0701SBB	LD0701SBB	07/12/96	2234
1001-DP07-9-12	26181.27	07/12/96	2303
1001-DP07-15-18	26181.28	07/12/96	2331
EXPLOSIVE L3A STD	15-209-5	07/13/96	0000
EXPLOSIVE L3B STD	15-216-5:CA5	07/13/96	0029
1001-DP07-18-21	26181.29	07/13/96	0058
1001-DP08-9-12	26181.32	07/13/96	0126
1001-DP08-15-18	26181.33	07/13/96	0155
1001-DP08-21-24	26181.34	07/13/96	0234
1001-DP08-21-24MSA	26181.35MSA	07/13/96	0252
1001-DP08-21-24MSB	26181.35MSB	07/13/96	0321
1001-DP08-21-24MSDA	26181.36MSDA	07/13/96	0350
1001-DP08-21-24MSDB	26181.36MSDB	07/13/96	0419
EXPLOSIVE L3A STD	15-209-5	07/13/96	0448
EXPLOSIVE L3B STD	15-216-5:CA6	07/13/96	0516

FORM VIII

CALIBRATION FACTOR DATA
Form 9A-1

'LOSIVE CALIBRATION DATA SW846-8330

strument ID : IN13
olumn ID : ODS-10

	Inj 1	Inj 2	Inj 3
Injection File Name Level 1 :	1EX0711,4		
Injection File Name Level 2 :	1EX0711,5		
Injection File Name Level 3 :	1EX0711,6		
Injection File Name Level 4 :	1EX0711,7		
Injection File Name Level 5 :	1EX0711,8		

Calibration Date : 07-11-96
Number of Calibration Levels: 5
Line forced thru zero : Yes
Calculation Method : Area

COMPONENT NAME	Area Level 1	CAL FACTOR Level 1	Area Level 2	CAL FACTOR Level 2	Area Level 3	CAL FACTOR Level 3	Area Level 4	CAL FACTOR Level 4	Area Level 5	CAL FACTOR Level 5
HMX	3440	261	84248	255	185491	281	272487	275	321086	243
RDX	2024	368	45275	329	97861	355	142551	346	170612	310
TNB	2858	725	71437	726	155215	788	223977	757	271687	690
---RYL	2138	442	52036	430	112142	463	160952	442	191368	395
	2245	1059	56564	1067	123093	1166	178805	1125	217613	1026
	2770	787	62495	710	134593	765	195961	742	236636	672
	3421	796	82904	770	180367	843	261386	812	318654	741
	2641	522	63850	505	139359	549	198749	523	243958	482
.T	3041	987	76048	988	165673	1076	236530	1020	289611	658
24DNT	2137	486	51452	468	113102	514	164272	498	200211	455
2NT	1861	423	46710	425	102853	468	149318	452	182225	414
4NT	2200	526	54973	527	120305	573	174104	554	212118	507
3NT										

CALIBRATION AND LINEARITY DATA
Form 9B

EXPLOSIVE CALIBRATION DATA - SW846-8330

Instrument ID : IN13
Column ID : ODS-10

	Inj 1	Inj 2	Inj 3
Injection File Name Level 1 :	1EX0711,4		
Injection File Name Level 2 :	1EX0711,5		
Injection File Name Level 3 :	1EX0711,6		
Injection File Name Level 4 :	1EX0711,7		
Injection File Name Level 5 :	1EX0711,8		

Calibration Date : 07-11-96
Number of Calibration Levels: 5
Line forced thru zero : Yes
Calculation Method : Area

Component Name	Mean Cal Factor	% RSD	Corr. Coeff.	Slope area/amt	Intercept
HMX	263	5.80	0.99425	259	0.000
RDX	342	6.59	0.99543	329	0.000
TNB	737	5.02	0.99614	726	0.000
TETRYL	435	5.75	0.99451	421	0.000
DNB	1089	5.12	0.99636	1079	0.000
26DNT	735	6.15	0.99605	709	0.000
24DNT	792	4.91	0.99633	779	0.000
2NT	516	4.76	0.99667	505	0.000
4NT	946	17.42	0.94747	810	0.000
3NT	484	4.85	0.99652	477	0.000
	436	5.18	0.99658	434	0.000
	538	4.82	0.99662	532	0.000

F-8

CONTINUING CALIBRATION DATA
Form 9C

itial Calibration Date: 07-11-96
inuing Calibration #: 1
inuing Cal Date : 07-12-96
tinuing Cal Level : 3
Instrument ID : IN13
Column ID : ODS-10
Injection File Name : 1EX0711,22
Calculation Mode : Area

Component Name	Response Cont Std	Cont CF	Init CF Mean	%D	Init CF Slope	%D
HMX	182738	277	263	5.24	259	6.76
RDX	97680	354	342	3.62	329	7.70
TNB	154339	783	737	6.29	726	7.89
TETRYL	110409	456	435	4.99	421	8.26
DNB	123303	1168	1089	7.26	1079	8.26
TNT	133752	760	735	3.36	709	7.20
NB	179649	839	792	5.95	779	7.79
26DNT	138156	544	516	5.37	505	7.63
24DNT	165155	1072	946	13.40	810	32.42
2NT	112821	513	484	5.94	477	7.43
4NT	103159	469	436	7.46	434	8.00
3NT	120025	572	538	6.33	532	7.37

F-9

CONTINUING CALIBRATION DATA
Form 9C

Initial Calibration Date: 07-11-96
Continuing Calibration #: 2
Continuing Cal Date : 07-12-96
Continuing Cal Level : 3
Instrument ID : IN13
Column ID : ODS-10
Injection File Name : 1EX0711,32
Calculation Mode : Area

Component Name	Response Cont Std	Cont CF	Init CF Mean	%D	Init CF Slope	%D
HMX	190335	288	263	9.62	259	11.19
RDX	100789	365	342	6.91	329	11.13
TNB	160084	813	737	10.24	726	11.91
TETRYL	115270	476	435	9.61	421	13.03
DNB	126906	1202	1089	10.40	1079	11.42
TNT	138713	788	735	7.19	709	11.17
NB	184281	861	792	8.68	779	10.57
26DNT	141946	559	516	8.27	505	10.59
24DNT	165037	1072	946	13.32	810	32.32
2NT	115872	527	484	8.80	477	10.33
4NT	106304	483	436	10.74	434	11.29
3NT	124003	590	538	9.85	532	10.93

200

F-10

CONTINUING CALIBRATION DATA
Form 9C

Initial Calibration Date: 07-11-96
Continuing Calibration #: 5
Continuing Cal Date : 07-12-96
Continuing Cal Level : 3
Instrument ID : IN13
Column ID : ODS-10
Injection File Name : 1EX0711,42
Calculation Mode : Area

Component Name	Response Cont Std	Cont CF	Init CF Mean	%D	Init CF Slope	%D
HMX	184958	280	263	6.52	259	8.05
RDX	98470	357	342	4.45	329	8.57
TNB	156215	793	737	7.58	726	9.21
TETRYL	111411	460	435	5.94	421	9.25
DNB	123023	1165	1089	7.02	1079	8.01
TNT	134913	767	735	4.25	709	8.13
NB	180993	846	792	6.74	779	8.60
26DNT	139403	549	516	6.33	505	8.60
24DNT	166019	1078	946	13.99	810	33.11
2NT	114182	519	484	7.22	477	8.72
4NT	104846	477	436	9.22	434	9.76
3NT	122670	584	538	8.67	532	9.74

CONTINUING CALIBRATION DATA
Form 9C

Initial Calibration Date: 07-11-96
C inuing Calibration #: 4
inuing Cal Date : 07-12-96
tinuing Cal Level : 3
Instrument ID : IN13
Column ID : ODS-10
Injection File Name : 1EX0711,48
Calculation Mode : Area

Component Name	Response Cont Std	Cont CF	Init CF Mean	%D	Init CF Slope	%D
HMX	185974	282	263	7.10	259	8.65
RDX	98231	356	342	4.20	329	8.31
TNB	155220	788	737	6.89	726	8.51
TETRYL	110906	458	435	5.46	421	8.75
DNB	123368	1168	1089	7.32	1079	8.31
TNT	134051	762	735	3.59	709	7.44
NB	181276	847	792	6.91	779	8.77
26DNT	139183	548	516	6.16	505	8.43
24DNT	165487	1075	946	13.63	810	32.68
2NT	113583	516	484	6.66	477	8.15
4NT	104630	476	436	8.99	434	9.54
3NT	121849	580	538	7.94	532	9.00

CONTINUING CALIBRATION DATA
Form 9C

itial Calibration Date: 07-1.-96
inuing Calibration #: 5
inuing Cal Date : 07-13-96
tinuing Cal Level : 3
Instrument ID : IN13
Column ID : ODS-10
Injection File Name : 1EX0711A,9
Calculation Mode : Area

Component Name	Response Cont Std	Cont CF	Init CF Mean	%D	Init CF Slope	%D
HMX	189743	287	263	9.28	259	10.85
RDX	100034	362	342	6.11	329	10.30
TNB	158273	803	737	9.00	726	10.64
TETRYL	110765	458	435	5.33	421	8.61
DNB	125035	1184	1089	8.77	1079	9.78
TNT	136498	776	735	5.48	709	9.40
NB	184742	863	792	8.95	779	10.85
26DNT	141891	559	516	8.22	505	10.54
24DNT	167240	1086	946	14.83	810	34.09
2NT	117239	533	484	10.09	477	11.64
4NT	107738	490	436	12.23	434	12.79
3NT	124776	594	538	10.54	532	11.62

CONTINUING CALIBRATION DATA
Form 9C

Initial Calibration Date: 07-11-96
Continuing Calibration #: 6
Continuing Cal Date : 07-13-96
Continuing Cal Level : 3
Instrument ID : IN13
Column ID : ODS-10
Injection File Name : 1EX0711A,19
Calculation Mode : Area

Component Name	Response Cont Std	Cont CF	Init CF Mean	%D	Init CF Slope	%D
HMX	183896	279	263	5.91	259	7.43
RDX	97779	354	342	3.72	329	7.81
TNB	153220	778	737	5.52	726	7.11
TETRYL	107643	445	435	2.36	421	5.55
DNB	122605	1161	1089	6.66	1079	7.64
TNT	133206	757	735	2.93	709	6.76
NB	179838	840	792	6.06	779	7.91
26DNT	136899	539	516	4.42	505	6.65
24DNT	162797	1057	946	11.78	810	30.53
2NT	112146	510	484	5.31	477	6.79
4NT	102522	466	436	6.80	434	7.33
3NT	119065	567	538	5.48	532	6.51

CALIBRATION STANDARDS DATA TABLE
Form 9D

PLOSIVE CALIBRATION DATA SW846-8330

ection volume = 200 ul

Amount Units = ng

COMPONENT NAME	AMT ON COL Level 1	AMT ON COL Level 2	AMT ON COL Level 3	AMT ON COL Level 4	AMT ON COL Level 5
HMX	13.200	330.000	660.000	990.000	1320.000
RDX	5.500	137.600	276.000	412.000	550.000
TNB	3.940	98.400	197.000	296.000	394.000
TETRYL	4.840	121.000	242.000	364.000	484.000
DNB	2.120	53.000	105.600	159.000	212.000
TNT	3.520	88.000	176.000	264.000	352.000
NB	4.300	107.600	214.000	322.000	430.000
26DNT	5.060	126.400	254.000	380.000	506.000
24DNT	3.080	77.000	154.000	232.000	440.000
2NT	4.400	110.000	220.000	330.000	440.000
4NT	4.400	110.000	220.000	330.000	440.000
3NT	4.180	104.400	210.000	314.000	418.000

RETENTION TIME DATA TABLE
Form 9E

EXPLOSIVE CALIBRATION DAT - SW846-8330

ıstrument ID : IN13
ɔlumn ID : ODS-10

Calibration Date : 07-11-96
Number of Calibration Levels : 5
Number of Injections per level: 1

COMPONENT NAME	RT Level 1	RT Level 2	RT Level 3	RT Level 4	RT Level 5
HMX	3.55	3.55	3.54	3.54	3.52
RDX	4.84	4.84	4.84	4.84	4.82
TNB	5.97	5.97	5.96	5.96	5.94
TETRYL	6.72	6.72	6.71	6.71	6.69
DNB	7.37	7.36	7.35	7.35	7.33
TNT	8.13	8.12	8.11	8.11	8.09
NB	8.45	8.44	8.43	8.43	8.41
26DNT	9.53	9.52	9.51	9.51	9.49
24DNT	10.14	10.13	10.12	10.12	10.10
2NT	11.81	11.81	11.80	11.80	11.77
4NT	12.58	12.57	12.55	12.56	12.52
3NT	13.32	13.33	13.31	13.32	13.28

COMPONENT NAME	RT Cont CA1	RT Cont CA2	RT Cont CA3	RT Cont CA4	RT Cont CA5	RT Cont CA6	RT Cont CA7	RT Cont CA8
X	3.54	3.55	3.54	3.54	3.54	3.54	3.52	3.51
TNB	4.83	4.84	4.82	4.82	4.83	4.83	4.80	4.79
TETRYL	5.95	5.95	5.93	5.93	5.94	5.94	5.92	5.90
DNB	6.71	6.69	6.67	6.66	6.68	6.68	6.65	6.63
TNT	7.34	7.32	7.30	7.28	7.31	7.31	7.28	7.26
NB	8.10	8.08	8.05	8.04	8.06	8.06	8.04	8.01
26DNT	8.42	8.40	8.36	8.35	8.38	8.37	8.35	8.32
24DNT	9.50	9.46	9.42	9.40	9.4	9.44	9.41	9.38
2NT	10.10	10.06	10.01	10.00	10.04	10.04	10.00	9.97
4NT	11.77	11.72	11.65	11.64	11.69	11.68	11.65	11.62
3NT	12.53	12.46	12.40	12.38	12.44	12.44	12.40	12.36
	13.28	13.21	13.14	13.12	13.19	13.18	13.14	13.11

COMPONENT NAME	MEAN_RT	STD_DEV	3X SD
HMX	3.54	0.012	0.037
RDX	4.83	0.016	0.048
TNB	5.94	0.020	0.060
TETRYL	6.69	0.028	0.085
DNB	7.32	0.034	0.102
TNT	8.08	0.036	0.109
NB	8.39	0.041	0.123
26DNT	9.46	0.051	0.152
24DNT	10.06	0.058	0.173
T	11.72	0.072	0.216
	12.48	0.079	0.236
3NT	13.23	0.084	0.252

CALIBRATION FACTOR DATA
Form 9A-1

PA CALIBRATION - METHOD

Instrument ID : IN13
Column ID : ODS-10

	Inj 1	Inj 2	Inj 3
Injection File Name Level 1 :	1EX0711,9		
Injection File Name Level 2 :	1EX0711,10		
Injection File Name Level 3 :	1EX0711,11		
Injection File Name Level 4 :	1EX0711,12		
Injection File Name Level 5 :	1EX0711,13		

Calibration Date : 07-11-96
Number of Calibration Levels: 5
Line forced thru zero : Yes
Calculation Method : Area

COMPONENT NAME	Area Level 1	CAL FACTOR Level 1	Area Level 2	CAL FACTOR Level 2	Area Level 3	CAL FACTOR Level 3	Area Level 4	CAL FACTOR Level 4	Area Level 5	CAL FACTOR Level 5
4ADNT	1123	488	5360	466	55663	484	109164	475	213444	464
2ADNT	1921	768	8764	701	90991	728	179981	720	352342	705

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CALIBRATION AND LINEARITY DATA
Form 9B

DF: CALIBRATION - METHOD

Instrument ID : IN13
Column ID : ODS-10

	Inj 1	Inj 2	Inj 3
Injection File Name Level 1 :	1EX0711,9		
Injection File Name Level 2 :	1EX0711,10		
Injection File Name Level 3 :	1EX0711,11		
Injection File Name Level 4 :	1EX0711,12		
Injection File Name Level 5 :	1EX0711,13		

Calibration Date : 07-11-96
Number of Calibration Levels: 5
Line forced thru zero : Yes
Calculation Method : Area

Component Name	Mean Cal Factor	% RSD	Corr. Coeff.	Slope area/amt	Intercept
4ADNT	475	2.25	0.99989	467	0.000
2ADNT	724	3.72	0.99992	709	0.000

CONTINUING CALIBRATION DATA
Form 9C

Initial Calibration Date: 07-11-96
Continuing Calibration #: 1
Continuing Cal Date : 07-12-96
Continuing Cal Level : 3
Instrument ID : IN13
Column ID : ODS-10
Injection File Name : 1EX0711,23
Calculation Mode : Area

Component Name	Response Cont Std	Cont CF	Init CF Mean	%D	Init CF Slope	%D
4ADNT	56333	490	475	3.04	467	4.88
2ADNT	92115	737	724	1.73	709	3.97

CONTINUING CALIBRATION DATA
Form 9C

Initial Calibration Date: [illegible]-1[illegible]-96
Continuing Calibration #: 2
Continuing Cal Date : 07-12-96
Continuing Cal Level : 3
Instrument ID : IN13
Column ID : ODS-10
Injection File Name : 1EX0711,33
Calculation Mode : Area

Component Name	Response Cont Std	Cont CF	Init CF Mean	%D	Init CF Slope	%D
4ADNT	54642	475	475	0.05	467	1.73
2ADNT	89287	714	724	1.40	709	0.78

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CONTINUING CALIBRATION DATA
Form 9C

Initial Calibration Date: 07-11-96
Continuing Calibration #: 3
Continuing Cal Date : 07-12-96
Continuing Cal Level : 3
Instrument ID : IN13
Column ID : ODS-10
Injection File Name : 1EX0711,43
Calculation Mode : Area

Component Name	Response Cont Std	Cont CF	Init CF Mean	%D	Init CF Slope	%D
4ADNT	52561	457	475	3.86	467	2.14
2ADNT	86298	690	724	4.70	709	2.59

CONTINUING CALIBRATION DATA
Form 9C

Initial Calibration Date: 07-11-96
Continuing Calibration #: 4
Continuing Cal Date : 07-12-96
Continuing Cal Level : 3
Instrument ID : IN13
Column ID : ODS-10
Injection File Name : 1EX0711,49
Calculation Mode : Area

Component Name	Response Cont Std	Cont CF	Init CF Mean	%D	Init CF Slope	%D
4ADNT	55010	478	475	0.62	467	2.42
2ADNT	90359	723	724	0.21	709	1.99

CONTINUING CALIBRATION DATA
Form 9C

itial Calibration Date: -/-11-96
ntinuing Calibration #: 5
ntinuing Cal Date : 07-13-96
ontinuing Cal Level : 3
Instrument ID : IN13
Column ID : ODS-10
Injection File Name : 1EX0711A,10
Calculation Mode : Area

Component Name	Response Cont Std	Cont CF	Init CF Mean	%D	Init CF Slope	%D
4ADNT	56665	493	475	3.65	467	5.50
2ADNT	93782	750	724	3.57	709	5.85

CONTINUING CALIBRATION DATA
Form 9C

Initial Calibration Date: 07-11-96
Continuing Calibration #: 6
Continuing Cal Date : 07-13-96
Continuing Cal Level : 3
Instrument ID : IN13
Column ID : ODS-10
Injection File Name : 1EX0711A,20
Calculation Mode : Area

Component Name	Response Cont Std	Cont CF	Init CF Mean	%D	Init CF Slope	%D
4ADNT	54540	474	475	0.24	467	1.54
2ADNT	90240	722	724	0.34	709	1.86

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CALIBRATION STANDARDS DATA TABLE
Form 9D

CALIBRATION - METHOD

jection volume = 200 ul

Amount Units = ng

COMPONENT NAME	AMT ON COL Level 1	AMT ON COL Level 2	AMT ON COL Level 3	AMT ON COL Level 4	AMT ON COL Level 5
4ADNT	2.300	11.500	115.000	230.000	460.000
2ADNT	2.500	12.500	125.000	250.000	500.000

RETENTION TIME DATA TABLE
Form 9E

DPA CALIBRATION - METHOD

Instrument ID : IN13
Column ID : ODS-10

Calibration Date : 07-11-96
Number of Calibration Levels : 5
Number of Injections per level: 1

COMPONENT NAME	RT Level 1	RT Level 1	RT Level 1	RT	RT
4ADNT	7.89	7.88	7.88	7.88	7.88
2ADNT	8.26	8.26	8.26	8.26	8.26

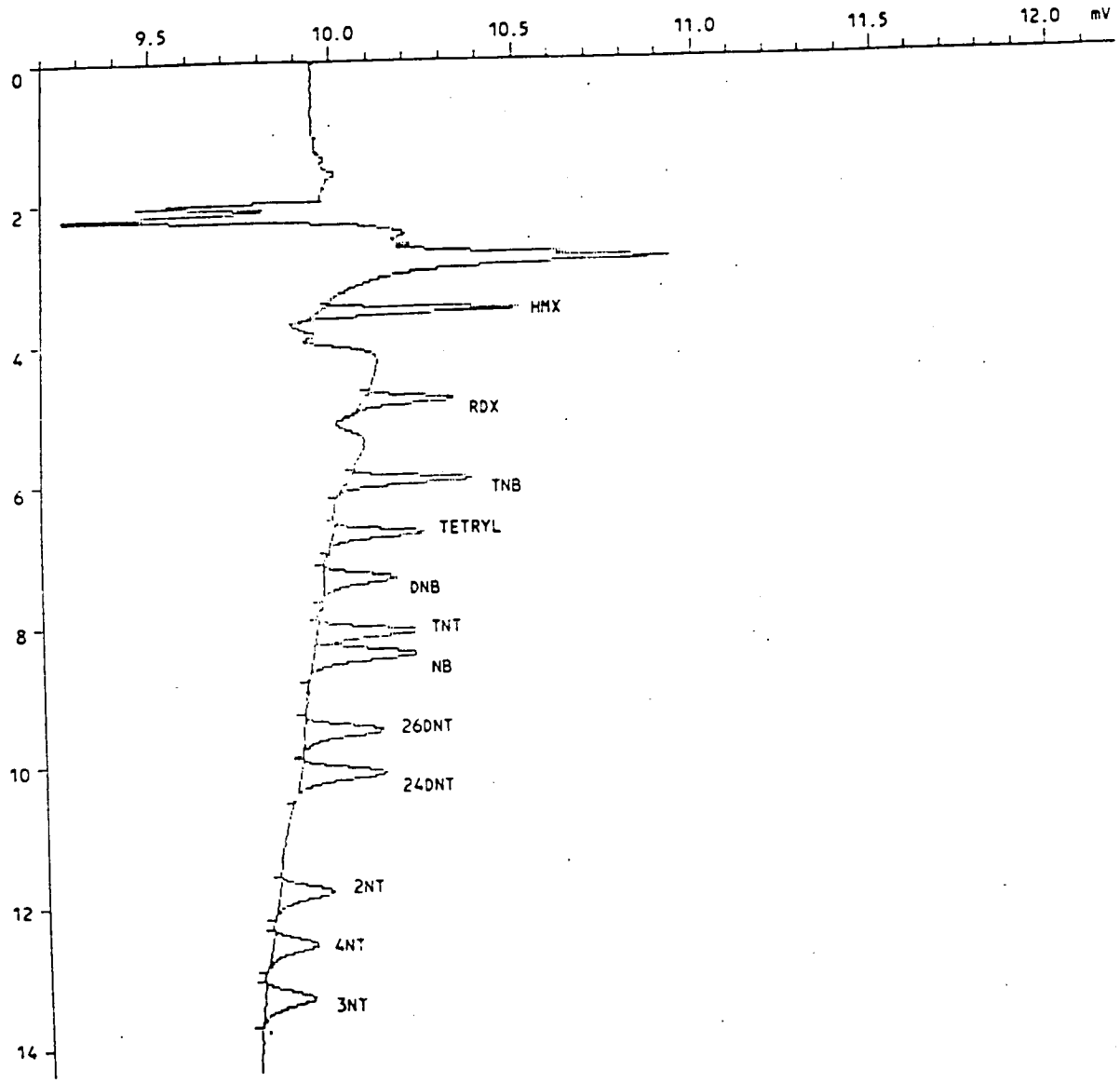
COMPONENT NAME	RT Cont CA1	RT Cont CA2	RT Cont CA3	RT Cont CA4	RT Cont CA5	RT Cont CA6	RT Cont CA7	RT Cont CA8
4ADNT	7.88	7.86	7.81	7.80	7.83	7.84	7.82	7.79
2ADNT	8.26	8.24	8.20	8.18	8.21	8.22	8.20	8.17

COMPONENT NAME	MEAN_RT	STD_DEV	3X SD
4ADNT	7.85	0.035	0.106
2ADNT	8.23	0.034	0.102

INJECTION REPORT

Injection F: <MC3> 1 1EX0711,4,1

Acquired on 11-Jul-96 at 17:09:14
Modified on 13-Jul-80 at 14:29:02
Reported on 13-Jul-96 at 14:38:00
INST ID: IN13, COL. TYPE: ODS-II, ID: ODS-10, INJ:200,COL#:1



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INJECTION REPORT

Injection F: <MC3> 1 1EX0711,4,1

Acquired on 11-Jul-96 at 17:09:14
Modified on 13-Jul-80 at 14:29:02
Reported on 13-Jul-96 at 14:30:09

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: PV
Comment.....: INST. ID: IN13, COL. TYPE: ODS-II, ID: ODS-10,INJ:200,COL#:1
Number of samples.: 49
Calibration file..: 1EX0711 Last modified on 13-Jul-80 at 14:29:02
Method file.....: EXPLOS Last modified on 13-Jul-80 at 14:30:06
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: EXPLOSIVE L1A STANDARD
Sample ID.....: 15-211-5:IA1
Sample type.....: Standard
Sample amount.....: 1.0000
Number of injections.....: 1
Bottle Number.....: 1

Retention time update.....: No update
Calibration level identifier.....: 1
Response factor update.....: Initialise

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000
PH.....: 0.000

User factors:
Volume (mL).....: 1.000
Dilution.....: 1.000
Amount (g or mL).....: 1.000

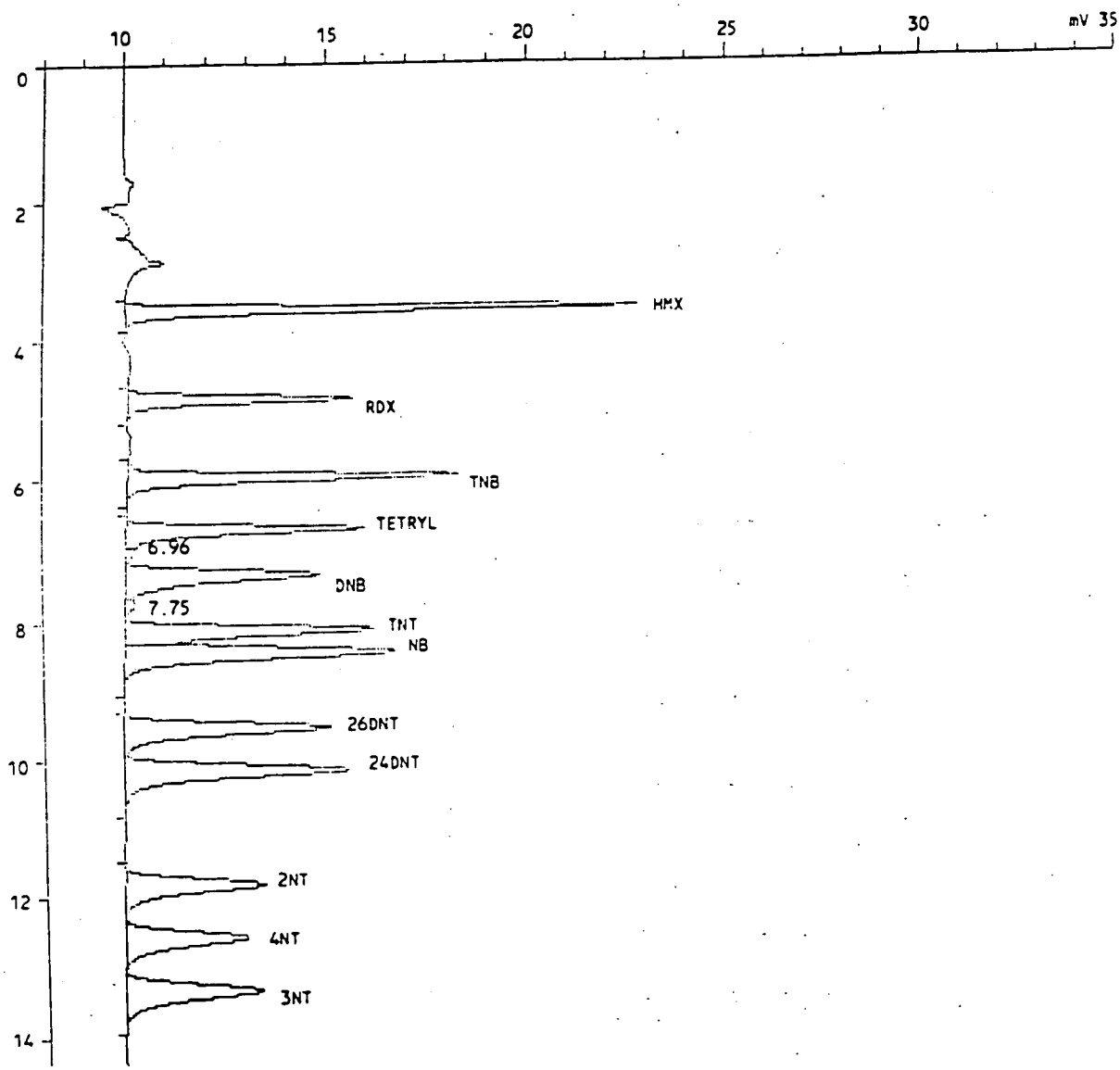
PEAK INFORMATION

Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
1	3.551	558	3440	0.067	HMX
2	4.844	252	2024	0.031	RDX
3	5.973	347	2858	0.020	TNB
4	6.724	255	2138	0.025	TETRYL
5	7.369	203	2245	0.010	DNB
6	8.129	279	2770	0.020	TNT
7	8.453	285	3421	0.022	NB
8	9.529	222	2641	0.026	26DNT
9	10.142	237	3041	0.015	24DNT

INJECTION REPORT

Injection F: <MC3> 1 1EX0711,5,1

Acquired on 11-Jul-96 at 17:37:55
Modified on 13-Jul-80 at 14:28:36
Reported on 13-Jul-96 at 14:37:14
INST ID: IN13, COL. TYPE: ODS-II, ID: ODS-10, INJ:200,COL#:1



INJECTION REPORT

Injection F: <MC3> 1 1EX0711,5,1

Acquired on 11-Jul-96 at 17:37:55
Modified on 13-Jul-80 at 14:28:36
Reported on 13-Jul-96 at 14:30:29

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: PV
Comment.....: INST. ID: IN13, COL. TYPE: ODS-II, ID: ODS-10,INJ:200,COL#:1
Number of samples.: 49
Calibration file..: 1EX0711 Last modified on 13-Jul-80 at 14:29:02
Method file.....: EXPLOS Last modified on 13-Jul-80 at 14:30:06
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: EXPLOSIVE L2A STANDARD
Sample ID.....: 15-210-5:IA2
Sample type.....: Standard
Sample amount.....: 1.0000
Number of injections.....: 1
Bottle Number.....: 1

Retention time update.....: No update
Calibration level identifier.....: 2
Response factor update.....: Initialise

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000
PH.....: 0.000

User factors:
Volume (mL).....: 1.000
Dilution.....: 1.000
Amount (g or mL).....: 1.000

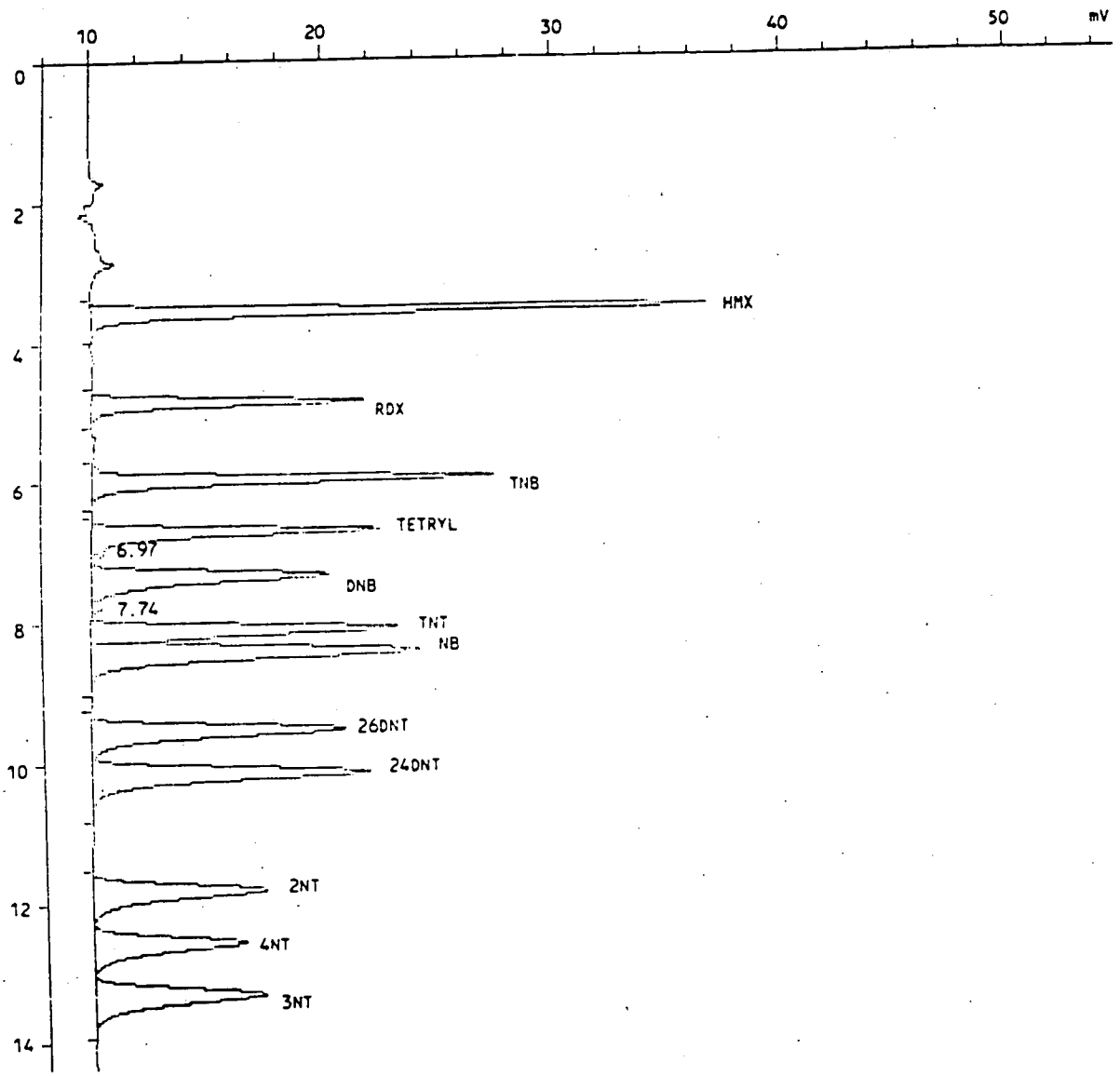
PEAK INFORMATION

Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
1	3.547	12816	84240	1.631	HMX
2	4.840	5627	45275	0.691	RDX
3	5.969	8250	71437	0.493	TNB
4	6.720	5956	52036	0.620	TETRYL
6	7.356	4818	56564	0.263	DNB
8	8.120	6238	62495	0.442	TNT
9	8.440	6761	82904	0.534	NB
10	9.520	5144	63850	0.633	26DNT
11	10.133	5635	76048	0.387	24DNT

INJECTION REPORT

Injection F: <MC3> 1 1EX0711,6,1

Acquired on 11-Jul-96 at 18:06:36
Modified on 13-Jul-80 at 14:28:04
Reported on 13-Jul-96 at 14:36:15
INST ID: IN13, COL. TYPE: ODS-II, ID: ODS-10, INJ:200,COL#:1



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INJECTION REPORT

Injection F: <MC3> 1 1EX0711,6,1

Acquired on 11-Jul-96 at 18:06:36
Modified on 13-Jul-80 at 14:28:04
Reported on 13-Jul-96 at 14:30:51

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: PV
Comment.....: INST. ID: IN13, COL. TYPE: ODS-II, ID: ODS-10,INJ:200,COL#:1
Number of samples.: 49
Calibration file..: 1EX0711 Last modified on 13-Jul-80 at 14:29:02
Method file.....: EXPLOS Last modified on 13-Jul-80 at 14:30:06
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: EXPLOSIVE L3A STANDARD
Sample ID.....: 15-209-5:IA3
Sample type.....: Standard
Sample amount.....: 1.0000
Number of injections.....: 1
Bottle Number.....: 1

Retention time update.....: No update
Calibration level identifier.....: 3
Response factor update.....: Initialise

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000
PH..... .....: 0.000

User factors:
Volume (mL).....: 1.000
Dilution.....: 1.000
Amount (g or mL).....: 1.000

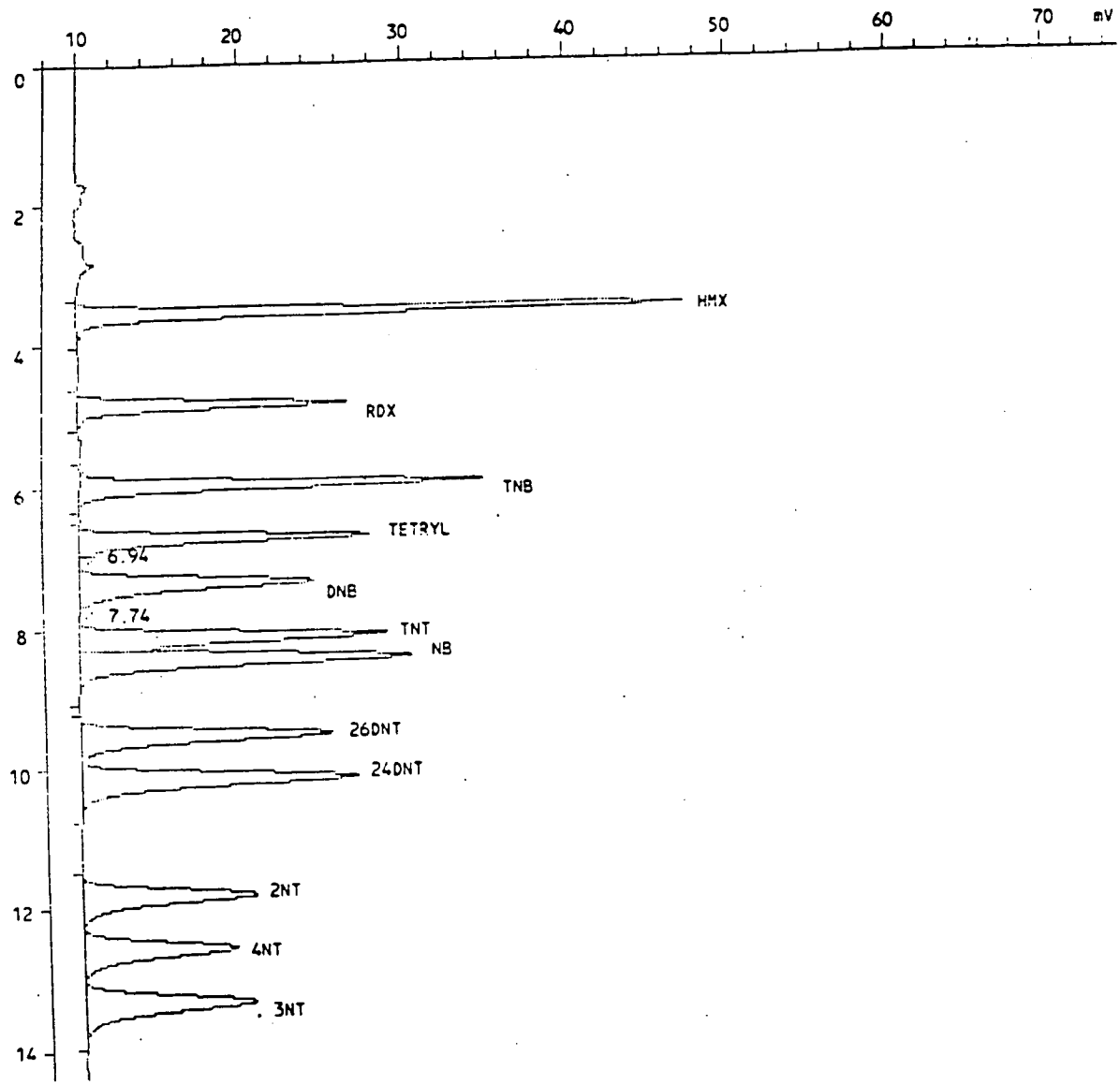
PEAK INFORMATION

Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
1	3.542	26810	185491	3.591	HMX
2	4.836	11853	97861	1.494	RDX
3	5.960	17529	155215	1.072	TNB
4	6.711	12540	112142	1.336	TETRYL
6	7.347	10251	123093	0.572	DNB
8	8.111	13298	134593	0.952	TNT
9	8.427	14391	180367	1.161	NB
10	9.507	11026	139359	1.382	26DNT
11	10.120	12072	165673	0.842	24DNT

INJECTION REPORT

Injection F: <MC3> 1 1EX0711,7,1

Acquired on 11-Jul-96 at 18:35:18
Modified on 13-Jul-80 at 14:26:04
Reported on 13-Jul-96 at 14:35:19
INST ID: IN13, COL. TYPE: ODS-II, ID: ODS-10, INJ:200,COL#:1



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INJECTION REPORT

Injection F: <MC3> 1 1EX0711,7,1

Acquired on 11-Jul-96 at 18:35:18
Modified on 13-Jul-80 at 14:26:04
Reported on 13-Jul-96 at 14:31:13

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: PV
Comment.....: INST. ID: IN13, COL. TYPE: ODS-II, ID: ODS-10,INJ:200,COL#:1
Number of samples.: 49
Calibration file..: 1EX0711 Last modified on 13-Jul-80 at 14:29:02
Method file.....: EXPLOS Last modified on 13-Jul-80 at 14:30:06
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: EXPLOSIVE L4A STANDARD
Sample ID.....: 15-208-5:IA4
Sample type.....: Standard
Sample amount.....: 1.0000
Number of injections.....: 1
Bottle Number.....: 1

Retention time update.....: No update
Calibration level identifier.....: 4
Response factor update.....: Initialise

Dilution factor amounts:
PERCENT MOISTURE..... : 0.000
PH.....: 0.000

User factors:
Volume (mL).....: 1.000
Dilution.....: 1.000
Amount (g or mL).....: 1.000

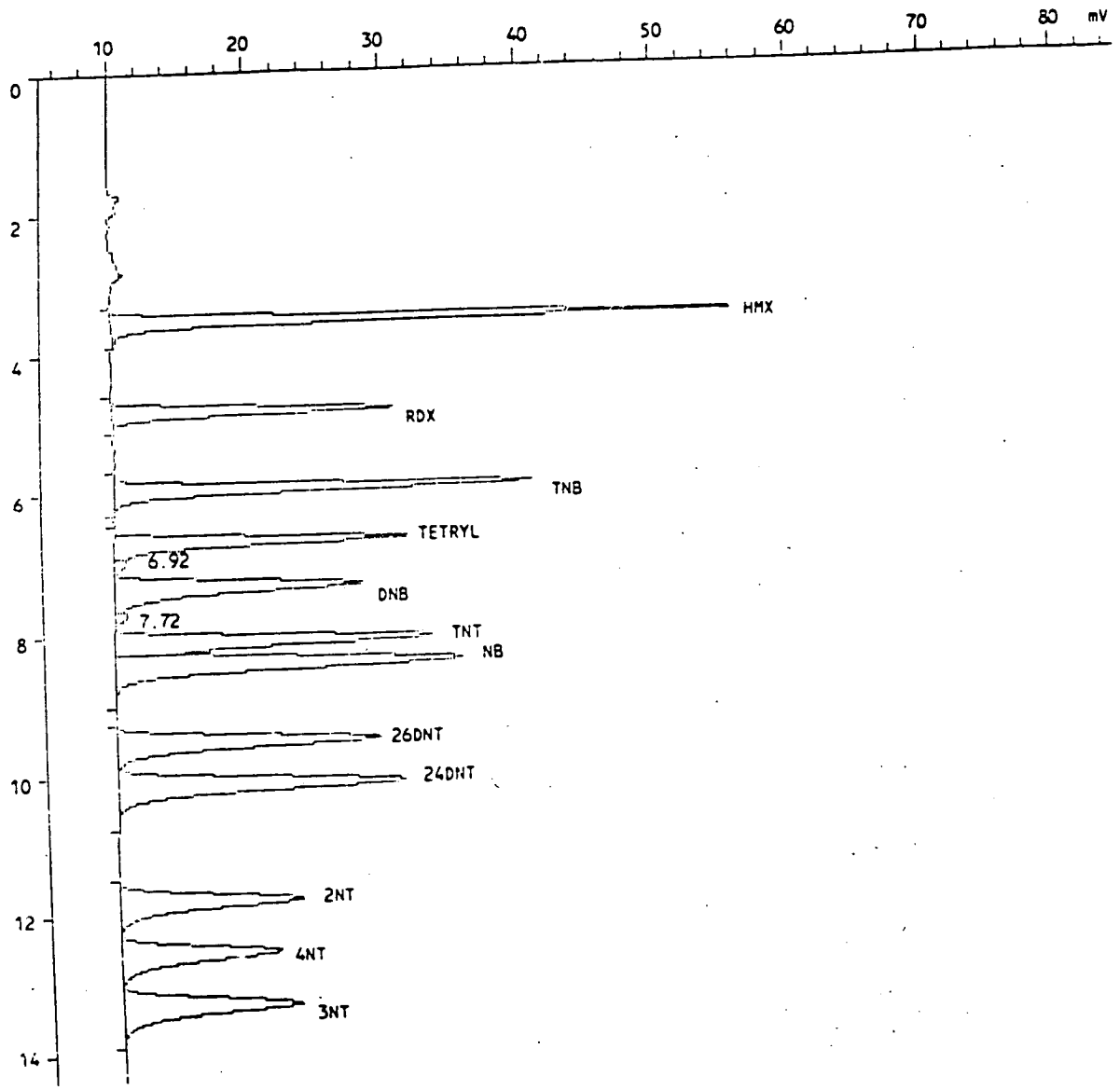
PEAK INFORMATION

Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
1	3.542	37586	272487	4.956	HMX
2	4.836	16723	142551	2.070	RDX
3	5.960	24898	223977	1.476	TNB
4	6.711	17927	160952	1.806	TETRYL
6	7.347	14546	178805	0.796	DNB
8	8.111	18964	195961	1.333	TNT
9	8.431	20441	261386	1.603	NB
10	9.511	15628	198749	1.884	26DNT
11	10.120	17131	236530	1.145	24DNT

INJECTION REPORT

Injection F: <MC3> 1 1EX0711,8,1

Acquired on 11-Jul-96 at 19:04:25
Modified on 13-Jul-80 at 14:27:28
Reported on 13-Jul-96 at 14:33:38
INST ID: IN13, COL. TYPE: ODS-II, ID: ODS-10, INJ:200,COL#:1



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INJECTION REPORT

Injection F: <MC3> 1 1EX0711,8,1

Acquired on 11-Jul-96 at 19:04:25
Modified on 13-Jul-80 at 14:27:28
Reported on 13-Jul-96 at 14:31:35

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: PV
Comment.....: INST. ID: IN13, COL. TYPE: ODS-II, ID: ODS-10,INJ:200,COL#:1
Number of samples.: 49
Calibration file..: 1EX0711 Last modified on 13-Jul-80 at 14:29:02
Method file.....: EXPLOS Last modified on 13-Jul-80 at 14:30:06
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: EXPLOSIVE LSA STANDARD
Sample ID.....: 15-207-5:IA5
Sample type.....: Standard
Sample amount.....: 1.0000
Number of injections.....: 1
Bottle Number.....: 1

Retention time update.....: No update
Calibration level identifier.....: 5
Response factor update.....: Initialise

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000
PH..... .....: 0.000

User factors:
Volume (mL).....: 1.000
Dilution.....: 1.000
Amount (g or mL).....: 1.000

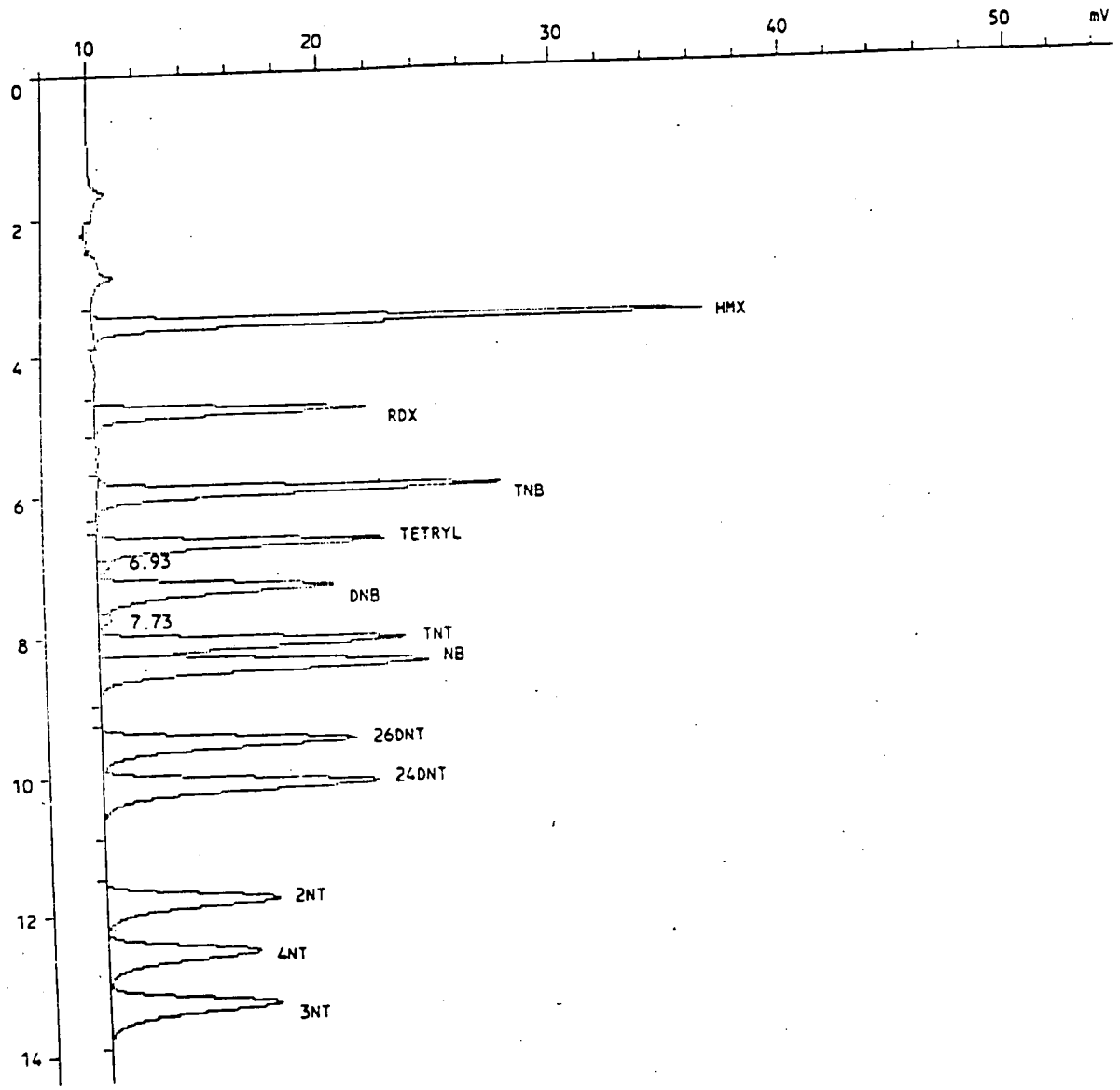
PEAK INFORMATION

Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
1	3.520	45727	321086	6.216	HMX
2	4.818	20870	170612	2.605	RDX
3	5.942	30862	271687	1.876	TNB
4	6.693	21567	191368	2.280	TETRYL
6	7.329	18207	217613	1.011	DNB
8	8.089	23277	236636	1.674	TNT
9	8.409	25603	318654	2.051	NB
10	9.489	19421	243958	2.420	26DNT
11	10.098	21325	289611	1.472	24DNT

INJECTION REPORT

Injection F: <MC3> 1 1EX0711,22,1

Acquired on 12-Jul-96 at 01:46:13
Modified on 13-Jul-80 at 14:42:44
Reported on 13-Jul-96 at 14:50:29
INST ID: IN13, COL. TYPE: ODS-II, ID: ODS-10, INJ:200,COL#:1



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INJECTION REPORT

Injection F: <MC3> 1 1EX0711,22,1

Acquired on 12-Jul-96 at 01:46:13
Modified on 13-Jul-80 at 14:42:44
Reported on 13-Jul-96 at 14:43:54

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: PV
Comment.....: INST. ID: IN13, COL. TYPE: ODS-II, ID: ODS-10,INJ:200,COL#:1
Number of samples.: 49
Calibration file..: 1EX0711 Last modified on 13-Jul-80 at 14:42:22
Method file.....: EXPLOS Last modified on 13-Jul-80 at 14:43:38
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: EXPLOSIVE L3A STANDARD
Sample ID.....: 15-209-5:CA1
Sample type.....: Standard
Sample amount.....: 1.0000
Number of injections.....: 1
Bottle Number.....: 1

Retention time update.....: No update
Calibration level identifier.....: 3
Response factor update.....: No update

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000
PH.....: 0.000

User factors:
Volume (mL).....: 1.000
Dilution.....: 1.000
Amount (g or mL).....: 1000.000

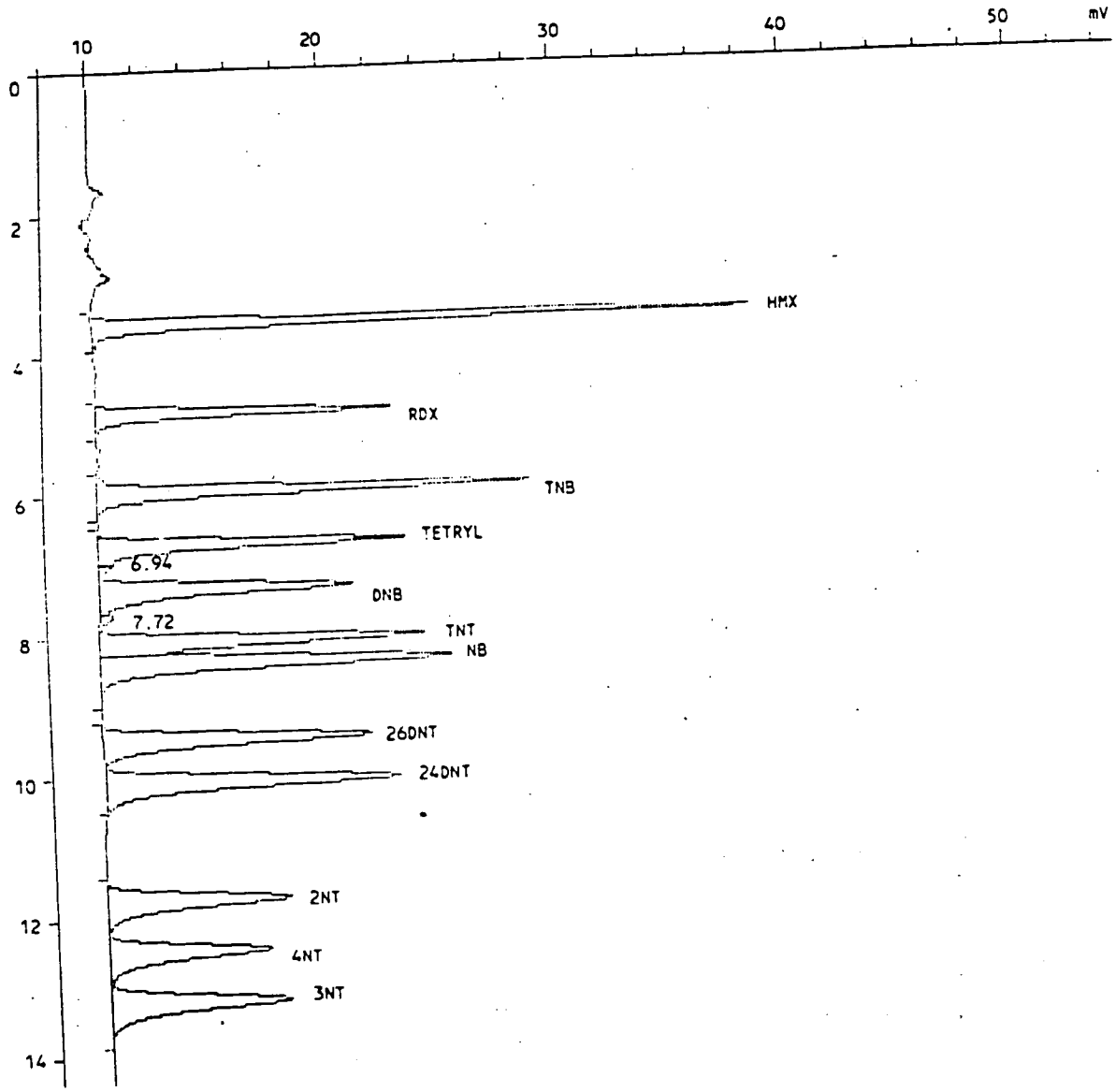
PEAK INFORMATION

Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
1	3.538	26468	182738	3.474	HMX
2	4.827	11781	97680	1.428	RDX
3	5.951	17451	154339	1.047	TNB
4	6.707	12423	110409	1.269	TETRYL
6	7.338	10196	123303	0.566	DNB
8	8.102	13222	133752	0.910	TNT
9	8.418	14288	179649	1.134	NB
10	9.498	11007	138156	1.339	26DNT
11	10.102	12045	165155	0.873	24DNT

INJECTION REPORT

Injection F: <MC3> 1 1EX0711,32,1

Acquired on 12-Jul-96 at 11:29:29
Modified on 13-Jul-80 at 14:42:50
Reported on 13-Jul-96 at 14:49:39
INST ID: IN13, COL. TYPE: ODS-II, ID: ODS-10, INJ:200,COL#:1



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INJECTION REPORT

Injection F: <MC3> 1 1EX0711,32,1

Acquired on 12-Jul-96 at 11:29:29
Modified on 13-Jul-80 at 14:42:50
Reported on 13-Jul-96 at 14:44:14

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: PV
Comment.....: INST. ID: IN13, COL. TYPE: ODS-TI, ID: ODS-10,INJ:200,COL#:1
Number of samples.: 49
Calibration file..: 1EX0711 Last modified on 13-Jul-80 at 14:42:22
Method file.....: EXPLOS Last modified on 13-Jul-80 at 14:43:38
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: EXPLOSIVE L3A STANDARD
Sample ID.....: 15-209-5:CA2
Sample type.....: Standard
Sample amount.....: 1.0000
Number of injections.....: 1
Bottle Number.....: 1

Retention time update.....: No update
Calibration level identifier.....: 3
Response factor update.....: No update

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000
PH.....: 0.000

User factors:
Volume (mL).....: 1.000
Dilution.....: 1.000
Amount (g or mL).....: 1000.000

PEAK INFORMATION

Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
1	3.551	28579	190335	3.619	HMX
2	4.836	12778	100789	1.474	RDX
3	5.951	18685	160084	1.086	TNB
4	6.693	13305	115270	1.325	TETRYL
6	7.324	10950	126906	0.583	DNB
8	8.080	14083	138713	0.944	TNT
9	8.396	15207	184281	1.163	NB
10	9.462	11751	141946	1.375	26DNT
11	10.062	12783	165037	0.872	24DNT

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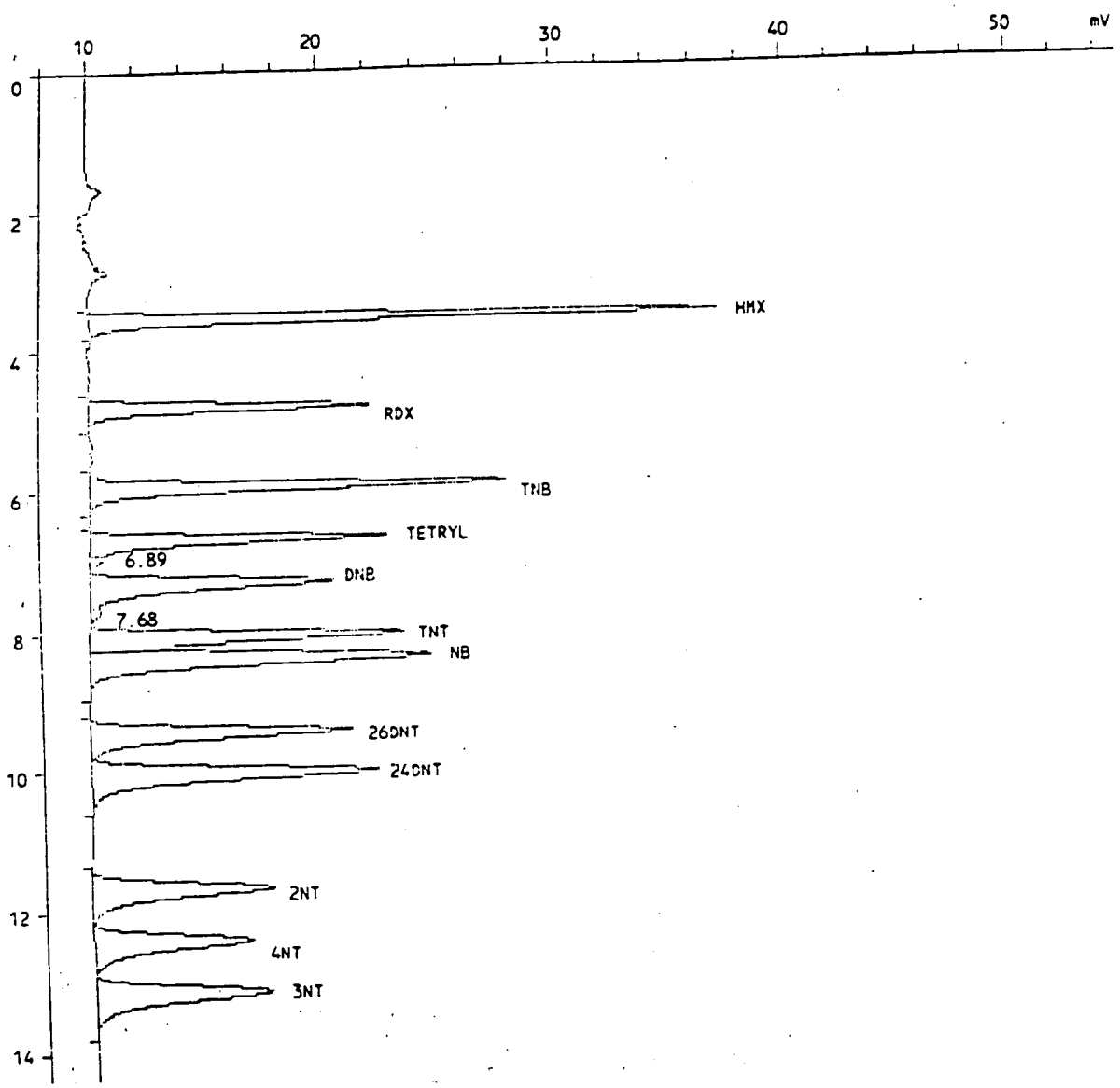
INJECTION REPORT

Injection F: <MC3> 1.1EX0711,42,1

Acquired on 12-Jul-96 at 16:16:34
Modified on 13-Jul-80 at 14:42:58
Reported on 13-Jul-96 at 14:49:06
INST ID: IN13, COL. TYPE: ODS-II, ID: ODS-10, INJ:200,COL#:1



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INJECTION REPORT

Injection F: <MC3> 1 1EX0711,42,1

Acquired on 12-Jul-96 at 16:16:34
Modified on 13-Jul-80 at 14:42:58
Reported on 13-Jul-96 at 14:44:36

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: PV
Comment.....: INST. ID: IN13, COL. TYPE: ODS-II, ID: ODS-10,INJ:200,COL#:1
Number of samples.: 49
Calibration file..: 1EX0711 Last modified on 13-Jul-80 at 14:42:22
Method file.....: EXPLOS Last modified on 13-Jul-80 at 14:43:38
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: EXPLOSIVE L3A STANDARD
Sample ID.....: 15-209-5:CA3
Sample type.....: Standard
Sample amount.....: 1.0000
Number of injections.....: 1
Bottle Number.....: 1

Retention time update.....: No update
Calibration level identifier.....: 3
Response factor update.....: No update

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000
PH.....: 0.000

User factors:
Volume (mL).....: 1.000
Dilution.....: 1.000
Amount (g or mL).....: 1000.000

PEAK INFORMATION

Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
1	3.538	27253	184958	3.516	HMX
2	4.822	12185	98470	1.440	RDX
3	5.933	17989	156215	1.060	TNB
4	6.667	12815	111411	1.281	TETRYL
6	7.298	10487	123023	0.565	DNB
8	8.049	13553	134913	0.918	TNT
9	8.360	14732	180993	1.143	NB
10	9.418	11323	139403	1.351	26DNT
11	10.013	12396	166019	0.877	24DNT

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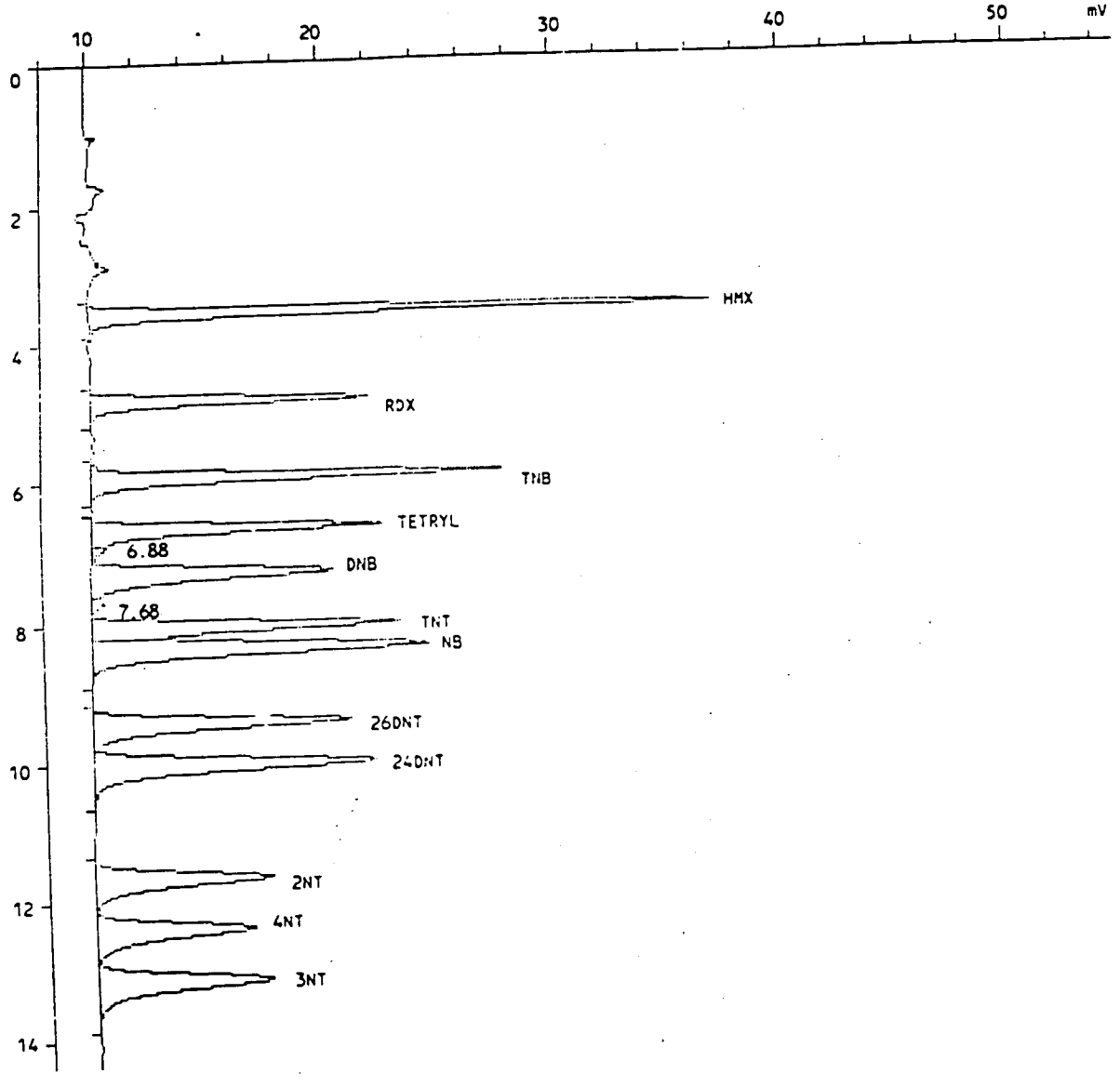
INJECTION REPORT

Injection F: <MC3> 1 1EX0711,48,1

Acquired on 12-Jul-96 at 19:09:03
Modified on 13-Jul-80 at 14:43:02
Reported on 13-Jul-96 at 14:48:10
INST ID: IN13, COL. TYPE: ODS-II, ID: ODS-10, INJ:200,COL#:1



F-43

INJECTION REPORT

Injection F: <MC3> 1 1EX0711,48,1

Acquired on 12-Jul-96 at 19:09:03
Modified on 13-Jul-80 at 14:43:02
Reported on 13-Jul-96 at 14:44:58

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: PV
Comment.....: INST. ID: IN13, COL. TYPE: ODS-II, ID: ODS-10,INJ:200,COL#:1
Number of samples.: 49
Calibration file..: 1EX0711 Last modified on 13-Jul-80 at 14:42:22
Method file.....: EXPLOS Last modified on 13-Jul-80 at 14:43:38
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: EXPLOSIVE L3A STANDARD
Sample ID.....: 15-209-5:CA4
Sample type.....: Standard
Sample amount.....: 1.0000
Number of injections.....: 1
Bottle Number.....: 1

Retention time update.....: No update
Calibration level identifier.....: 3
Response factor update.....: No update

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000
PH.....: 0.000

User factors:
Volume (mL).....: 1.000
Dilution..... .....: 1.000
Amount (g or mL).....: 1000.000

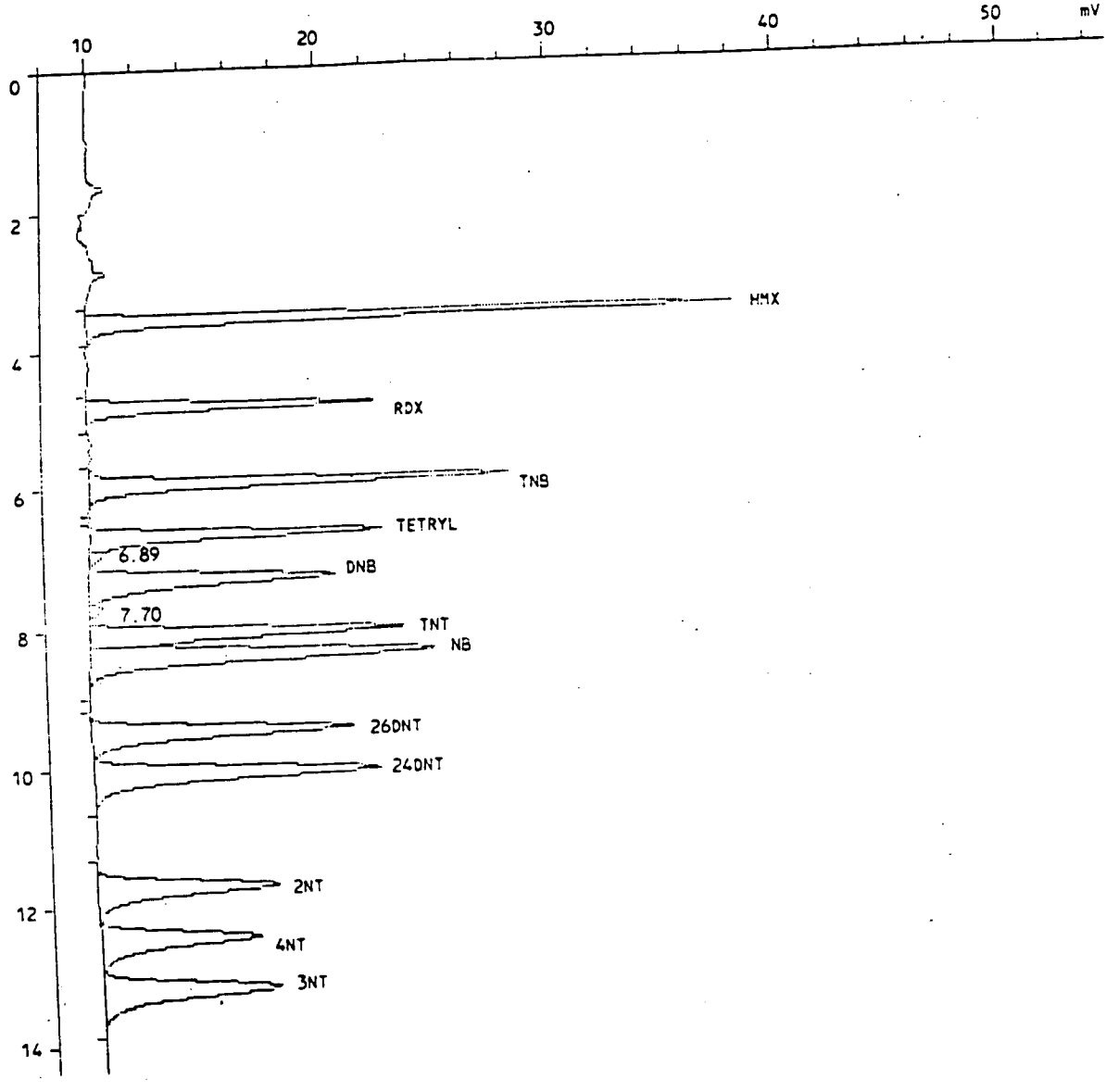
PEAK INFORMATION

Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
1	3.538	27036	185974	3.536	HMX
2	4.818	12057	98231	1.436	RDX
3	5.929	17744	155220	1.053	TNB
4	6.658	12596	110906	1.275	TETRYL
6	7.284	10409	123368	0.566	DNB
8	8.036	13422	134051	0.912	TNT
9	8.347	14601	181276	1.144	NB
10	9.400	11206	139183	1.349	26DNT
11	9.996	12273	165487	0.875	24DNT

INJECTION REPORT

Injection F: <MC3> 1 1EX0711A,9,1

Acquired on 13-Jul-96 at 00:00:37
Modified on 13-Jul-80 at 14:12:36
Reported on 15-Jul-96 at 15:07:46
INST ID: IN13, COL. TYPE: ODS-II, ID: ODS-10, INJ:200,COL#:1



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INJECTION REPORT

Injection F: <MC3> 1 1EX0711A,9,1

Acquired on 13-Jul-96 at 00:00:37
Modified on 13-Jul-80 at 14:12:36
Reported on 15-Jul-96 at 15:07:00

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: PV
Comment.....: INST. ID: IN13, COL. TYPE: ODS-II, ID: ODS-10,INJ:200,COL#:1
Number of samples.: 20
Calibration file..: 1EX0711 Last modified on 13-Jul-80 at 14:42:22
Method file.....: EXPLOS Last modified on 15-Jul-80 at 14:57:22
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: EXPLOSIVE L3A STANDARD
Sample ID.....: 15-209-5:CA5
Sample type.....: Standard
Sample amount.....: 1.0000
Number of injections.....: 1
Bottle Number.....: 1

Retention time update.....: No update
Calibration level identifier.....: 3
Response factor update.....: No update

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000
PH.....: 0.000

User factors:
Volume (mL).....: 1.000
Dilution.....: 1.000
Amount (g or mL).....: 1000.000

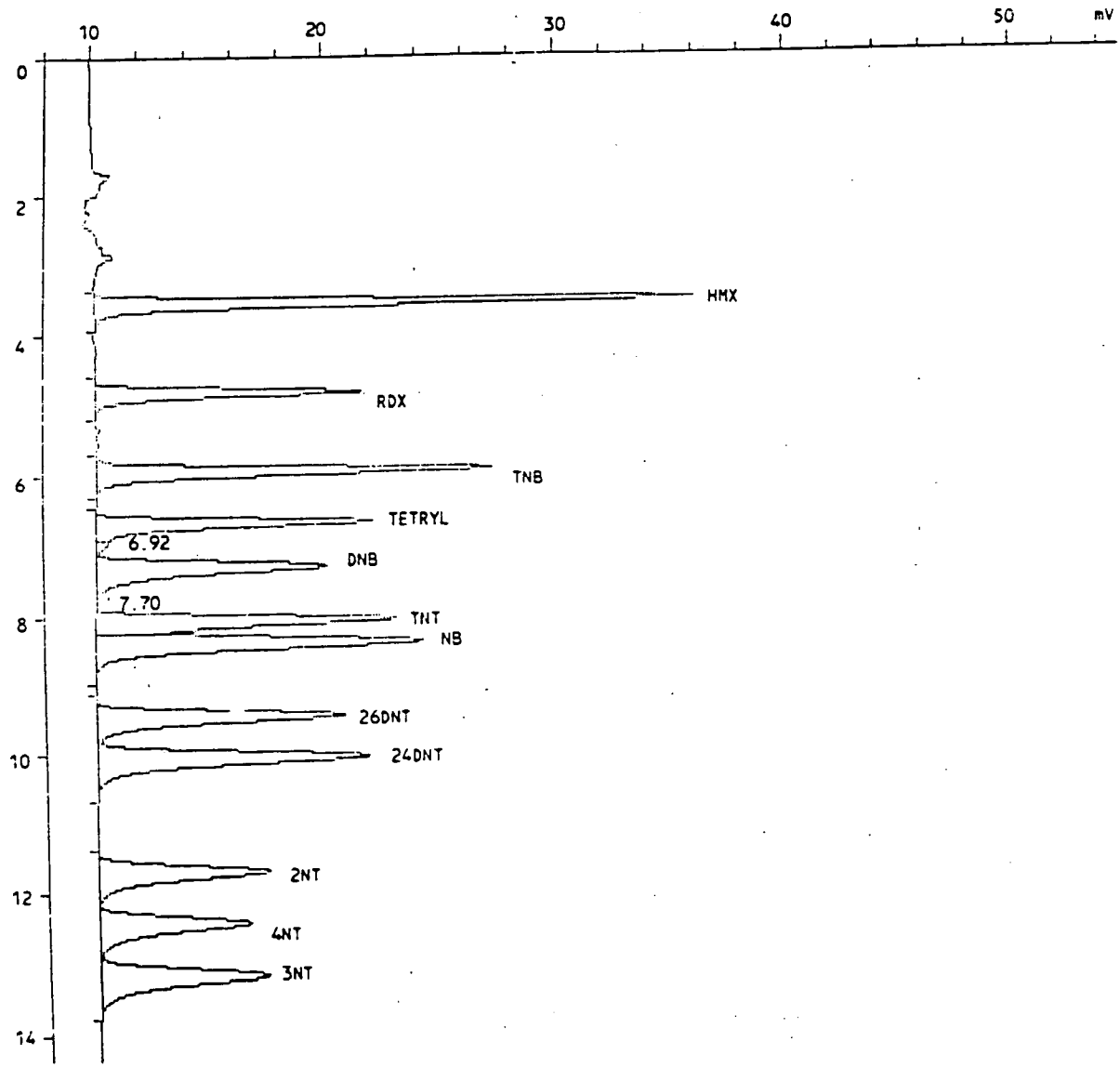
PEAK INFORMATION

Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
1	3.542	28203	189743	3.553	HMX
2	4.827	12547	100034	1.446	RDX
3	5.938	18266	158273	1.078	TNB
4	6.680	12750	110765	1.262	TETRYL
6	7.311	10715	125035	0.563	DNB
8	8.062	13733	136498	0.926	TNT
9	8.378	15121	184742	1.146	NB
10	9.444	11511	141891	1.362	26DNT
11	10.044	12569	167240	0.885	24DNT

INJECTION REPORT

Injection F: <MC3> 1 1EX0711A,19,1

Acquired on 13-Jul-96 at 04:48:00
Modified on 13-Jul-80 at 14:14:12
Reported on 15-Jul-96 at 15:08:37
INST ID: IN13, COL. TYPE: ODS-II, ID: ODS-10, INJ:200,COL#:1



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INJECTION REPORT

Injection F: <MC3> 1 1EX0711A,19,1

Acquired on 13-Jul-96 at 04:48:00
Modified on 13-Jul-80 at 14:14:12
Reported o.. 15-Jul-96 at 15:08:10

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: PV
Comment.....: INST. ID: IN13, COL. TYPE: ODS-II, ID: ODS-10,INJ:200,COL#:1
Number of samples.: 20
Calibration file..: 1EX0711 Last modified on 13-Jul-80 at 14:42:22
Method file.....: EXPLOS Last modified on 15-Jul-80 at 15:07:56
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: EXPLOSIVE L3A STANDARD
Sample ID.....: 15-209-5:CA6
Sample type.....: Standard
Sample amount.....: 1.0000
Number of injections.....: 1
Bottle Number.....: 1

Retention time update.....: No update
Calibration level identifier.....: 3
Response factor update.....: No update

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000
PH.....: 0.000

User factors:
Volume (mL).....: 1.000
Dilution.....: 1.000
Amount (g or mL).....: 1000.000

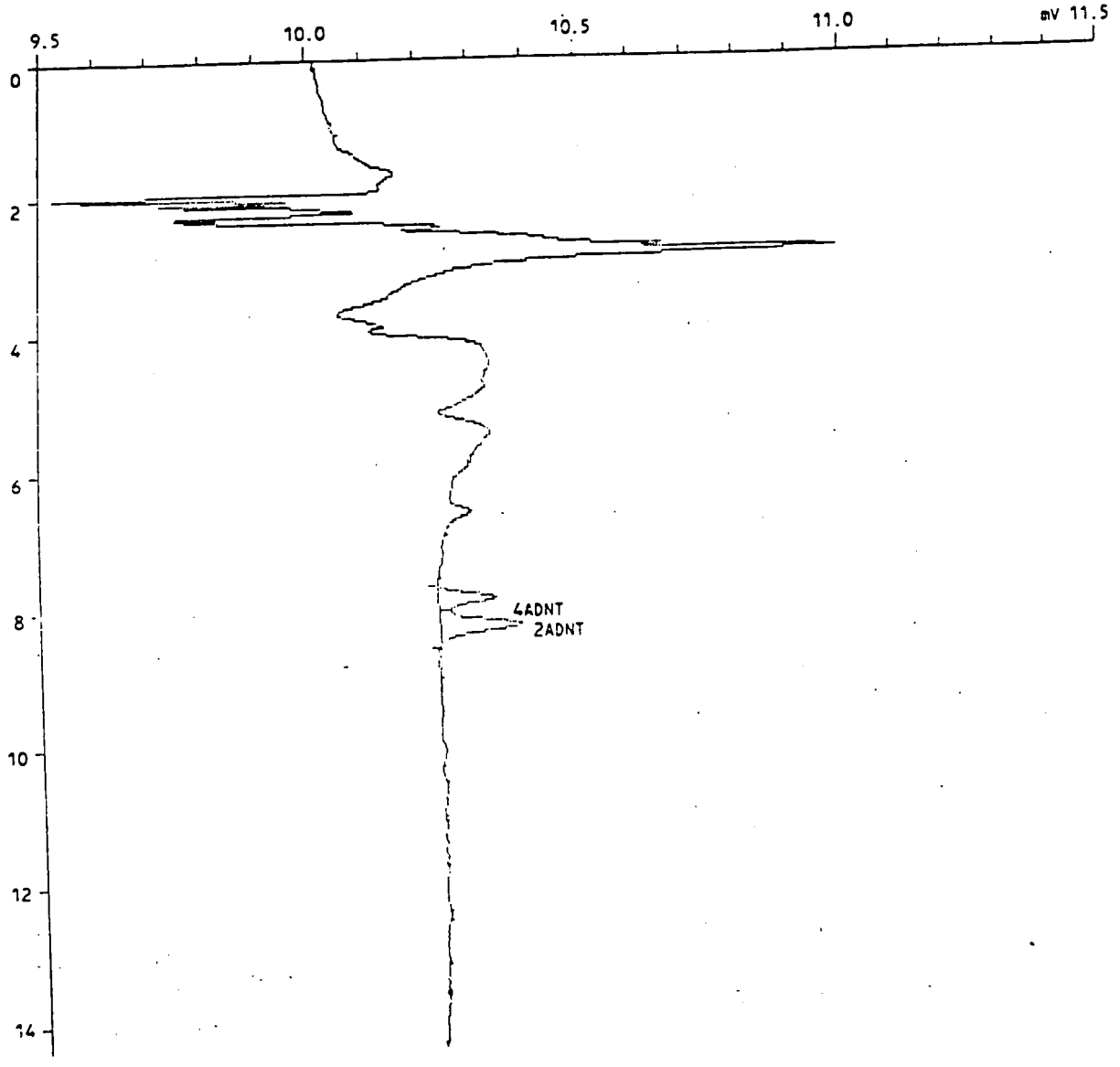
PEAK INFORMATION

Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
1	3.542	25955	183896	3.444	HMX
2	4.827	11586	97779	1.413	RDX
3	5.938	17163	153220	1.044	TNB
4	6.680	11978	107643	1.226	TETRYL
6	7.311	10069	122605	0.552	DNB
8	8.062	13044	133206	0.904	TNT
9	8.373	14174	179838	1.116	NB
10	9.440	10845	136899	1.314	26DNT
11	10.036	11871	162797	0.861	24DNT

INJECTION REPORT

Injection F: <MC3> 1 1EX0711,9,1

Acquired on 11-Jul-96 at 19:33:05
Modified on 18-Jul-80 at 10:08:58
Reported on 18-Jul-96 at 10:13:45
INST ID: IN13, COL. TYPE: ODS-II, ID: ODS-10, INJ:200,COL#:1



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INJECTION REPORT

Injection F: <MC3> 1 1EX0711,9,1

Acquired on 11-Jul-96 at 19:33:05
Modified on 18-Jul-80 at 10:08:58
Reported on 18-Jul-96 at 10:09:01

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: PV
Comment.....: INST. ID: IN13, COL. TYPE: ODS-II, ID: ODS-10,INJ:200,COL#:1
Number of samples.: 49
Calibration file..: 1EX0711B Last modified on 18-Jul-80 at 10:08:58
Method file.....: EXPLOS Last modified on 18-Jul-80 at 10:04:02
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: EXPLOSIVE L1B STANDARD
Sample ID.....: 15-218-5:IA1
Sample type.....: Standard
Sample amount.....: 1.0000
Number of injections.....: 1
Bottle Number.....: 1

Retention time update.....: No update
Calibration level identifier.....: 1
Response factor update.....: Initialise

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000
PH.....: 0.000

User factors:
Volume (mL).....: 1.000
Dilution.....: 1.000
Amount (g or mL).....: 1.000

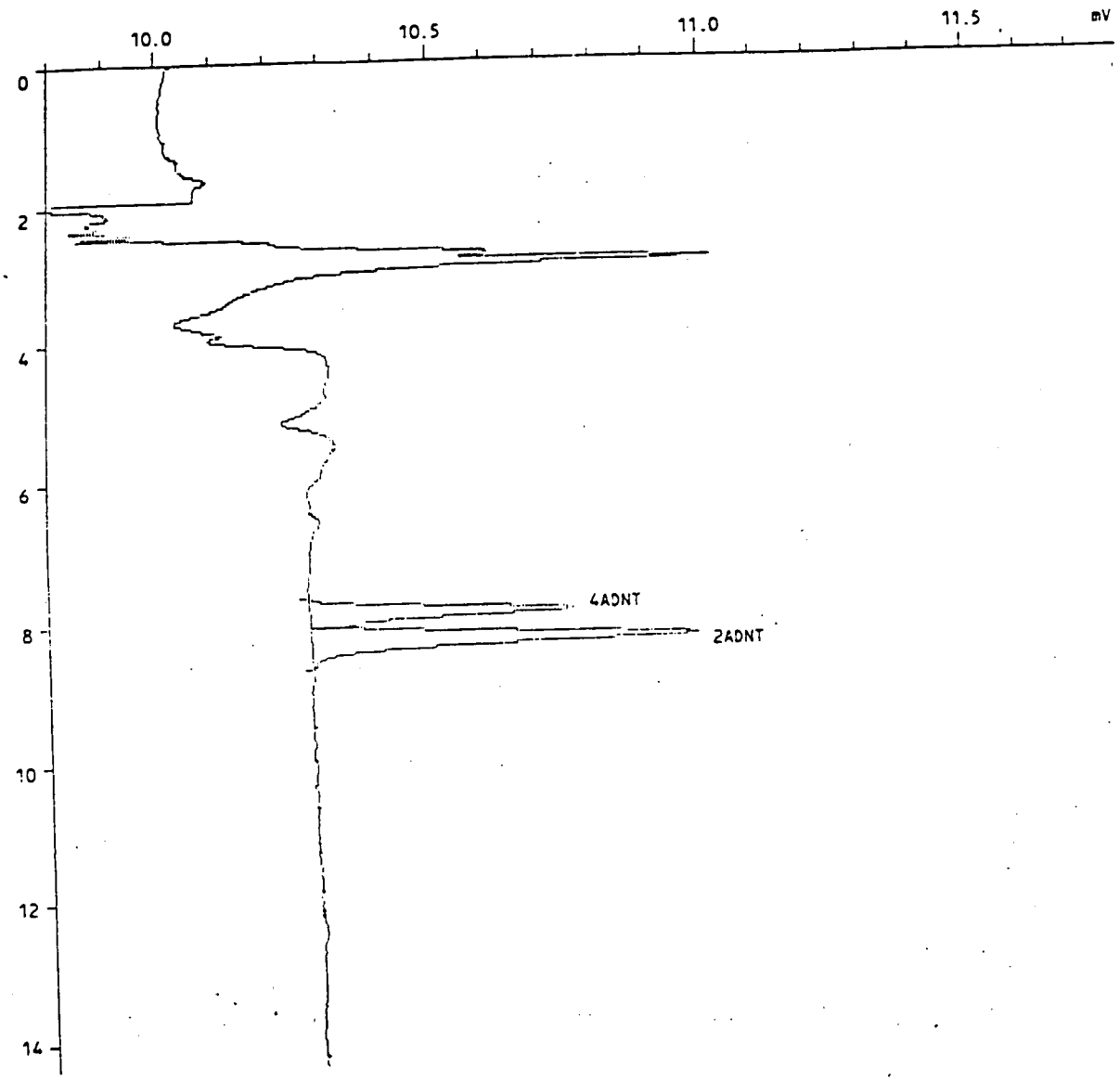
PEAK INFORMATION

Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
1	7.889	108	1123	0.012	4ADNT
2	8.258	154	1921	0.014	2ADNT
Total		261	3044	0.026	
Residual		0	0	0.000	

INJECTION REPORT

Injection F: <MC3> 1 1EX0711,10,1

Acquired on 11-Jul-96 at 20:01:45
Modified on 18-Jul-80 at 10:08:00
Reported on 18-Jul-96 at 10:13:07
INST ID: IN13, COL. TYPE: ODS-II, ID: ODS-10, INJ:200,COL#:1



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INJECTION REPORT

Injection F: <MC3> 1 1EX0711,10,1

Acquired on 11-Jul-96 at 20:01:45
Modified on 18-Jul-80 at 10:08:00
Reported on 18-Jul-96 at 10:09:45

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: PV
Comment.....: INST. ID: IN13, COL. TYPE: ODS-II, ID: ODS-10,INJ:200,COL#:1
Number of samples.: 49
Calibration file..: 1EX0711B Last modified on 18-Jul-80 at 10:08:58
Method file.....: EXPLOS Last modified on 18-Jul-80 at 10:04:02
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: EXPLOSIVE L2B STANDARD
Sample ID.....: 15-217-5:IA2
Sample type.....: Standard
Sample amount.....: 1.0000
Number of injections.....: 1
Bottle Number.....: 1

Retention time update.....: No update
Calibration level identifier.....: 2
Response factor update.....: Initialise

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000
PH.....: 0.000

User factors:
Volume (mL).....: 1.000
Dilution.....: 1.000
Amount (g or mL).....: 1.000

PEAK INFORMATION

Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
1	7.880	485	5360	0.057	4ADNT
2	8.262	714	8764	0.061	2ADNT
Total		1199	14124	0.118	
Residual		0	0	0.000	

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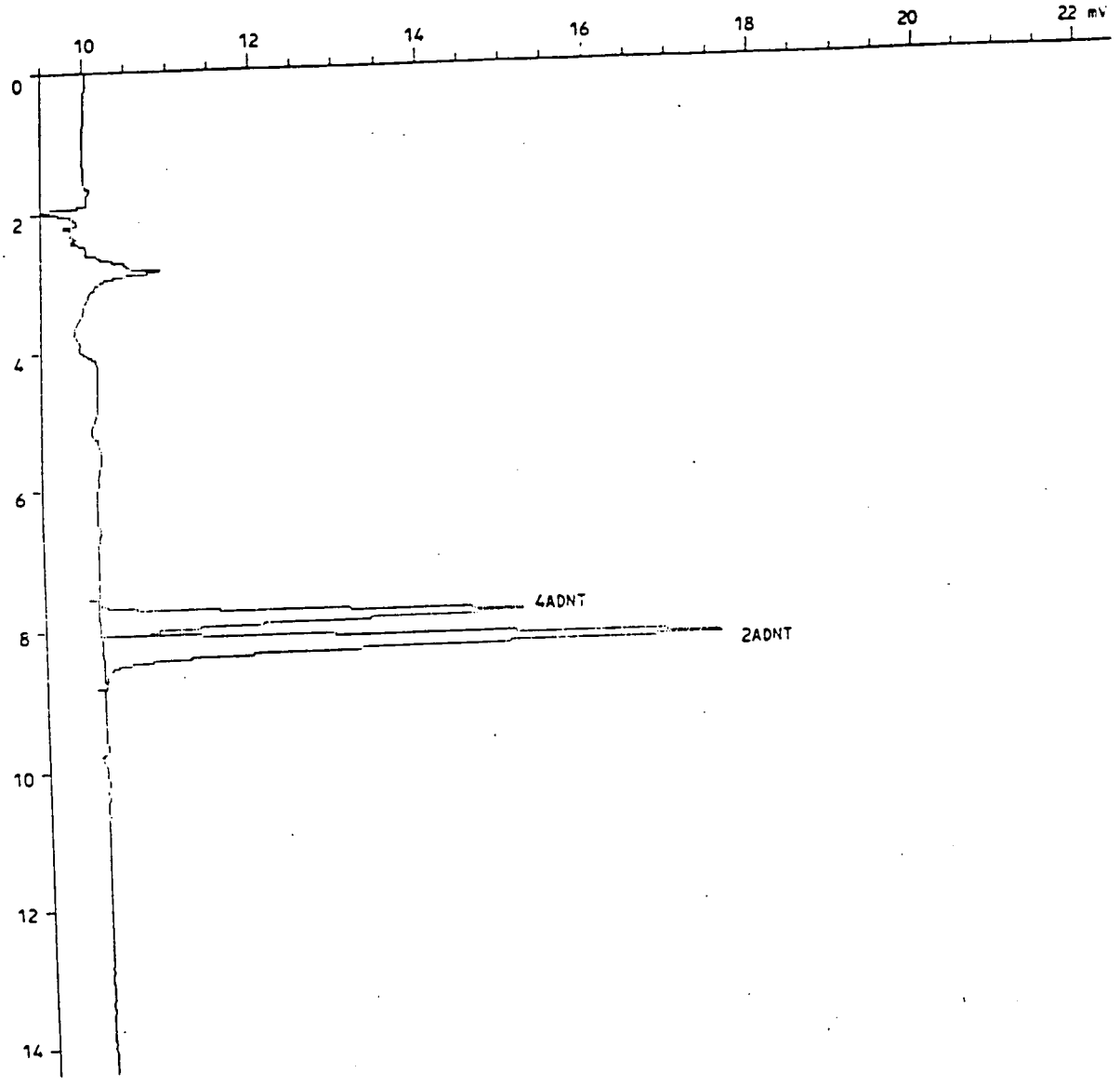
INJECTION REPORT

Injection F: <MC3> 1 1EX0711,11,1

Acquired on 11-Jul-96 at 20:30:26
Modified on 18-Jul-80 at 10:08:04
Reported on 18-Jul-96 at 10:12:20
INST ID: IN13, COL. TYPE: ODS-II, ID: ODS-10, INJ:200,COL#:1



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INJECTION REPORT

Injection F: <MC3> 1 1EX0711,11,1

Acquired on 11-Jul-96 at 20:30:26
Modified on 18-Jul-80 at 10:08:04
Reported on 18-Jul-96 at 10:10:01

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: PV
Comment.....: INST. ID: IN13, COL. TYPE: ODS-II, ID: ODS-10,INJ:200,COL#:1
Number of samples.: 49
Calibration file..: 1EX0711B Last modified on 18-Jul-80 at 10:08:58
Method file.....: EXPLOS Last modified on 18-Jul-80 at 10:04:02
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: EXPLOSIVE L3B STANDARD
Sample ID.....: 15-216-5:IA3
Sample type.....: Standard
Sample amount.....: 1.0000
Number of injections.....: 1
Bottle Number.....: 1

Retention time update.....: No update
Calibration level identifier.....: 3
Response factor update.....: Initialise

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000
PH.....: 0.000

User factors:
Volume (mL).....: 1.000
Dilution.....: 1.000
Amount (g or mL).....: 1.000

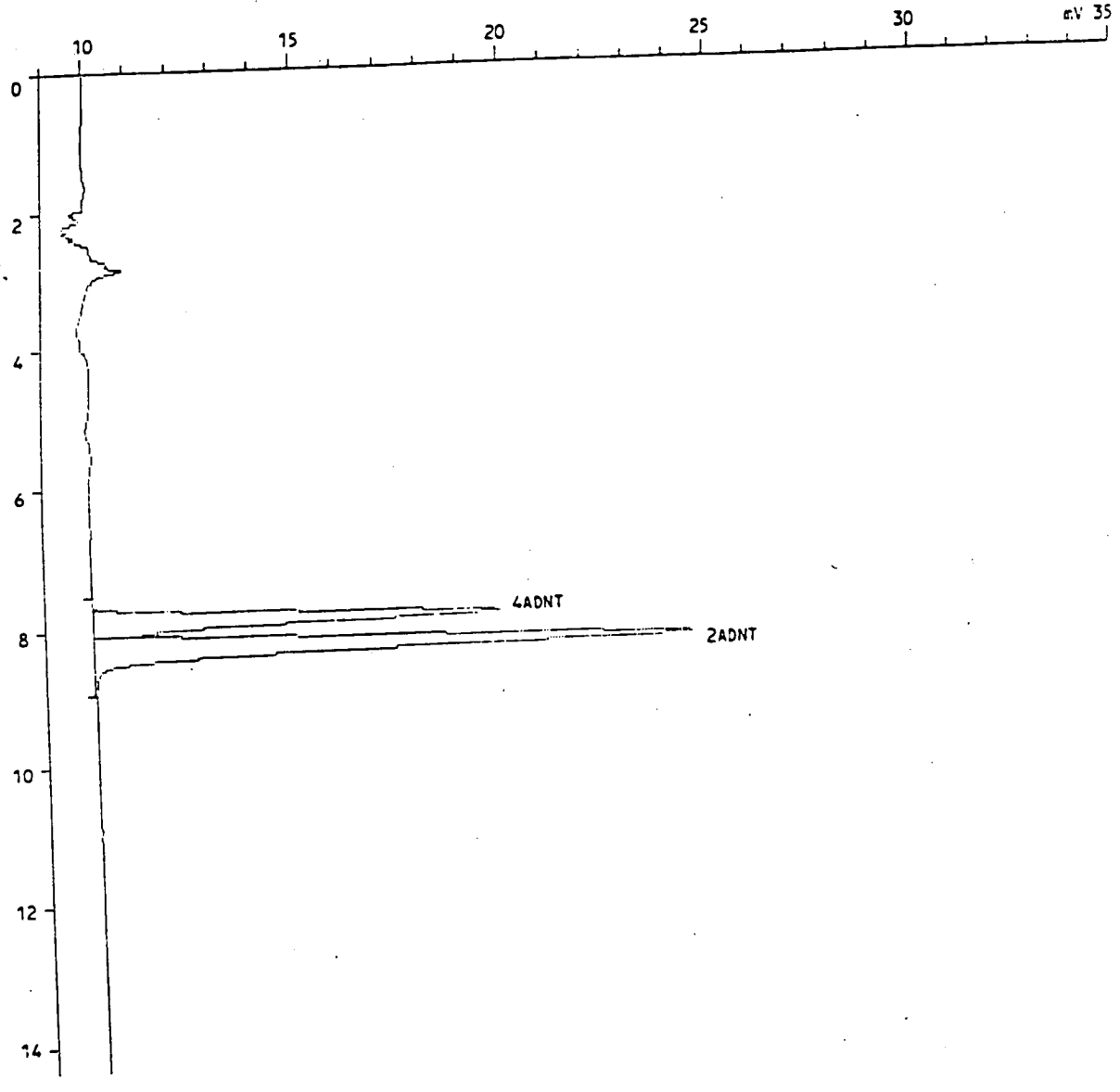
PEAK INFORMATION

Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
1	7.876	5066	55663	0.589	4ADNT
2	8.262	7441	90991	0.633	2ADNT
Total		12507	146654	1.222	
Residual		0	0	0.000	

INJECTION REPORT

Injection F: <MC3> 1 1EX0711,12,1

Acquired on 11-Jul-96 at 20:59:07
Modified on 18-Jul-80 at 10:08:08
Reported on 18-Jul-96 at 10:11:46
INST ID: IN13, COL. TYPE: ODS-II, ID: ODS-10, INJ:200,COL#:1



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INJECTION REPORT

Injection F: <MC3> 1 1EX0711,12,1

Acquired on 11-Jul-96 at 20:59:07
Modified on 18-Jul-80 at 10:08:08
Reported on 18-Jul-96 at 10:10:17

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: PV
Comment.....: INST. ID: IN13, COL. TYPE: ODS-II, ID: ODS-10,INJ:200,COL#:1
Number of samples.: 49
Calibration file..: 1EX0711B Last modified on 18-Jul-80 at 10:08:58
Method file.....: EXPLOS Last modified on 18-Jul-80 at 10:04:02
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: EXPLOSIVE L4B STANDARD
Sample ID.....: 15-215-5:IA4
Sample type.....: Standard
Sample amount.....: 1.0000
Number of injections.....: 1
Bottle Number.....: 1

Retention time update.....: No update
Calibration level identifier.....: 4
Response factor update.....: Initialise

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000
PH.....: 0.000

User factors:
Volume (mL).....: 1.000
Dilution.....: 1.000
Amount (g or mL).....: 1.000

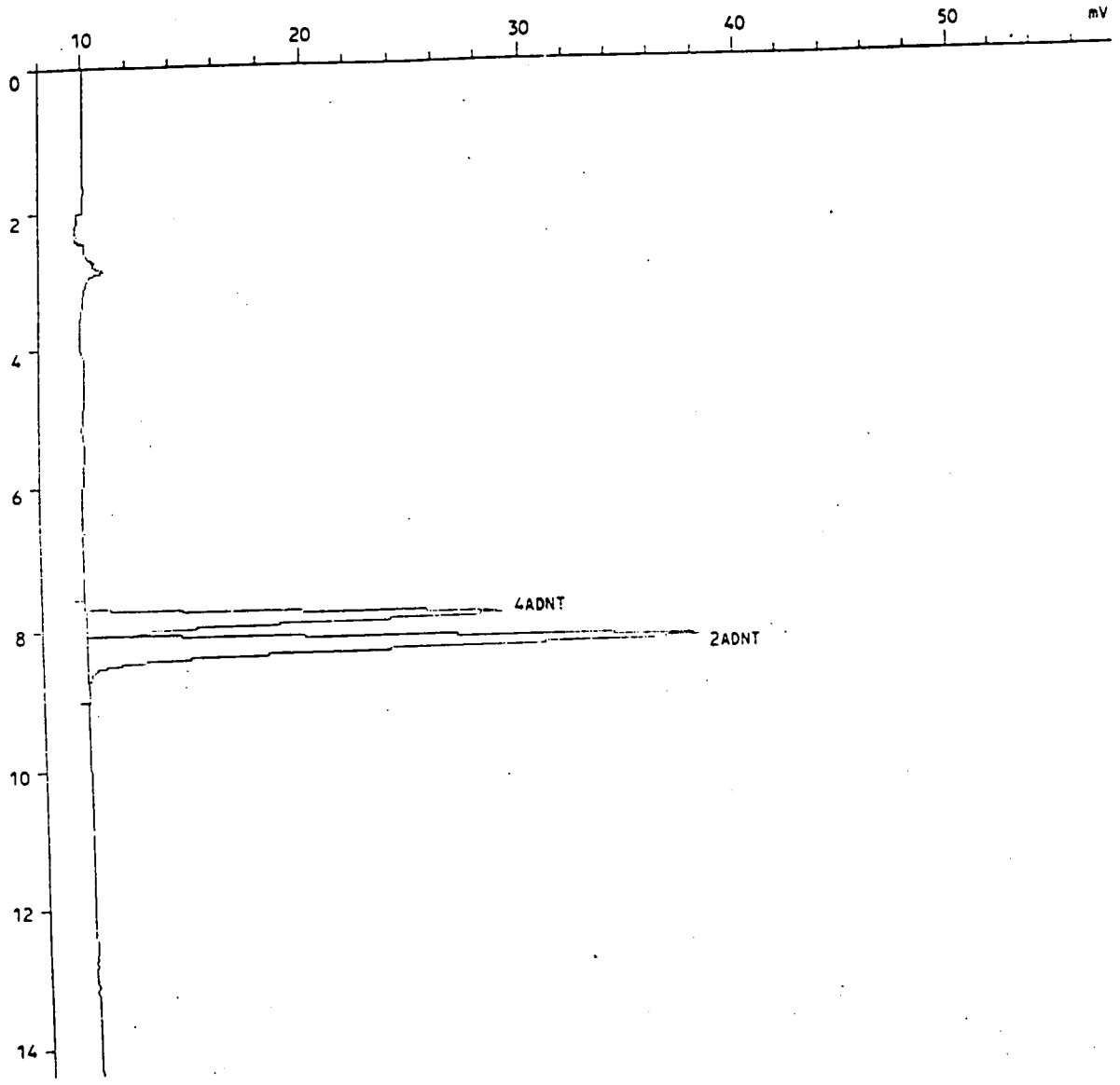
PEAK INFORMATION

Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
1	7.876	9823	109164	1.156	4ADNT
2	8.258	14452	179981	1.251	2ADNT
Total		24275	289146	2.407	
Residual		0	0	0.000	

INJECTION REPORT

Injection F: <MC3> 1 1EX0711,13,1

Acquired on 11-Jul-96 at 21:27:48
Modified on 18-Jul-80 at 10:08:12
Reported on 18-Jul-96 at 10:11:12
INST ID: IN13, COL. TYPE: ODS-II, ID: ODS-10, INJ:200,COL#:1



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INJECTION REPORT

Injection F: <MC3> 1 1EX0711,13,1

Acquired on 11-Jul-96 at 21:27:48
Modified on 18-Jul-80 at 10:08:12
Reported on 18-Jul-96 at 10:10:35

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: PV
Comment.....: INST. ID: IN13, COL. TYPE: ODS-II, ID: ODS-10,INJ:200,COL#:1
Number of samples.: 49
Calibration file..: 1EX0711B Last modified on 18-Jul-80 at 10:08:58
Method file.....: EXPLOS Last modified on 18-Jul-80 at 10:04:02
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: EXPLOSIVE L5B STANDARD
Sample ID.....: 15-214-5:IA5
Sample type.....: Standard
Sample amount.....: 1.0000
Number of injections.....: 1
Bottle Number.....: 1

Retention time update.....: No update
Calibration level identifier.....: 5
Response factor update.....: Initialise

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000
PH.....: 0.000

User factors:
Volume (mL).....: 1.000
Dilution.....: 1.000
Amount (g or mL).....: 1.000

PEAK INFORMATION

Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
1	7.876	19040	213444	2.285	4ADNT
2	8.258	28106	352342	2.486	2ADNT
Total		47146	565786	4.771	
Residual		0	0	0.000	

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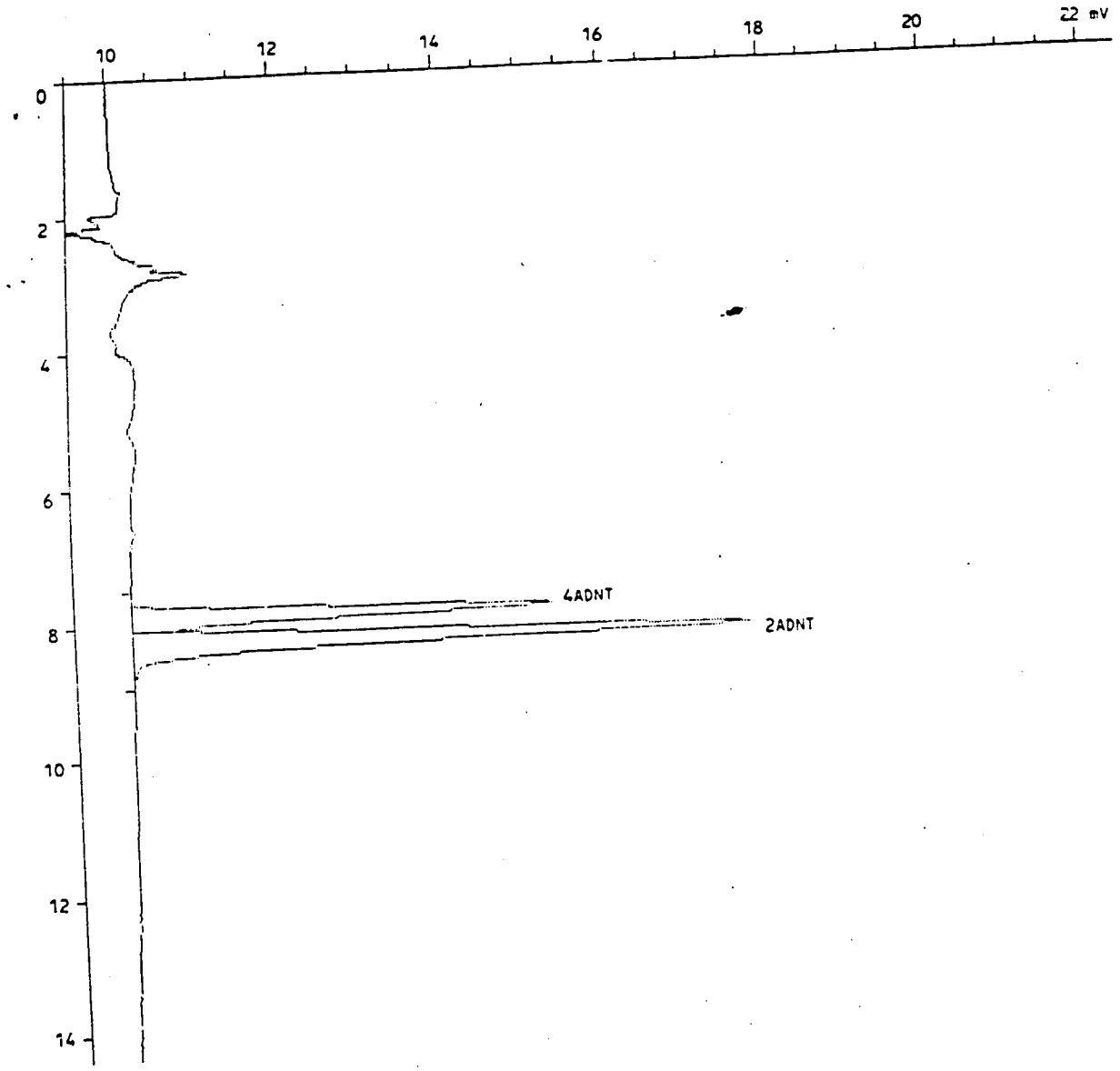
INJECTION REPORT

Injection F: <MC3> 1 1EX0711,23,1

Acquired on 12-Jul-96 at 02:14:58
Modified on 18-Jul-80 at 10:15:10
Reported on 18-Jul-96 at 10:23:37
INST ID: IN13, COL. TYPE: ODS-II, ID: ODS-10, INJ:200,COL#:1



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INJECTION REPORT

Injection F: <MC3> 1 1EX0711,23,1

Acquired on 12-Jul-96 at 02:14:58
Modified on 19-Jul-80 at 10:15:10
Reported on 18-Jul-96 at 10:23:18

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: PV
Comment.....: INST. ID: IN13, COL. TYPE: ODS-II, ID: ODS-10,INJ:200,COL#:1
Number of samples.: 49
Calibration file..: 1EX0711B Last modified on 18-Jul-80 at 10:16:30
Method file.....: EXPLOS Last modified on 18-Jul-80 at 10:23:08
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: EXPLOSIVE L3B STANDARD
Sample ID.....: 15-216-5:CA1
Sample type.....: Standard
Sample amount.....: 1.0000
Number of injections.....: 1
Bottle Number.....: 1

Retention time update.....: No update
Calibration level identifier.....: 3
Response factor update.....: No update

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000
PH.....: 0.000

User factors:
Volume (mL).....: 1.000
Dilution.....: 1.000
Amount (g or mL).....: 1000.000

PEAK INFORMATION

Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
1	7.876	5163	56333	0.593	4ADNT
2	8.262	7587	92115	0.636	2ADNT
Total		12751	148447	1.229	
Residual		0	0	0.000	

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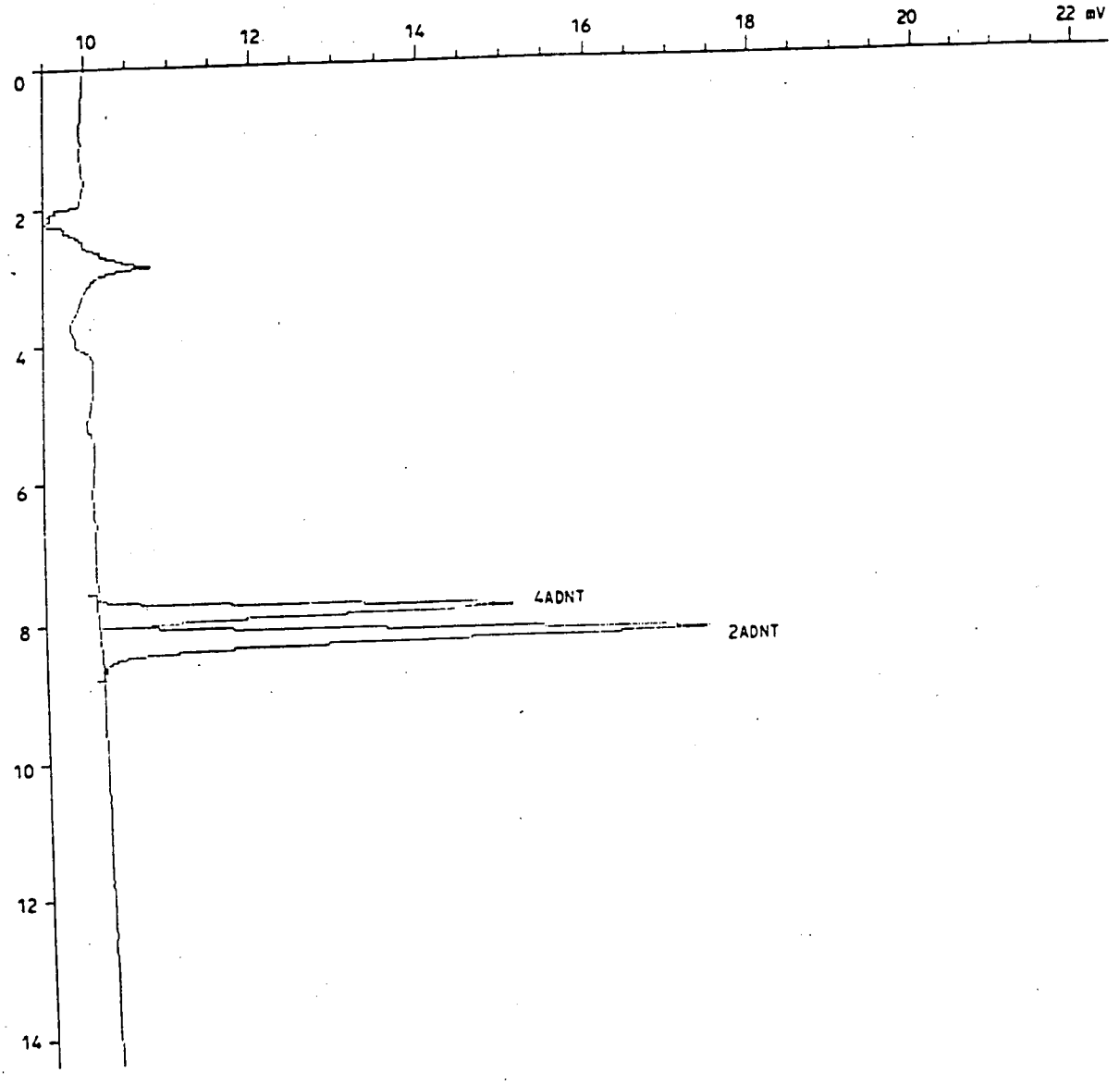
INJECTION REPORT

Injection F: <MC3> 1 1EX0711,33,1

Acquired on 12-Jul-96 at 11:58:10
Modified on 18-Jul-80 at 10:15:22
Reported on 18-Jul-96 at 10:24:16
INST ID: IN13, COL. TYPE: ODS-II, ID: ODS-10, INJ:200,COL#:1



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INJECTION REPORT

Injection F: <MC3> 1 1EX0711,33,1

Acquired on 12-Jul-96 at 11:58:10
Modified on 18 Jul-80 at 10:15:22
Reported on 18-Jul-96 at 10:23:58

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: PV
Comment.....: INST. ID: IN13, COL. TYPE: ODS-II, ID: ODS-10,INJ:200,COL#:1
Number of samples.: 49
Calibration file..: 1EX0711B Last modified on 18-Jul-80 at 10:16:30
Method file.....: EXPLOS Last modified on 18-Jul-80 at 10:23:52
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: EXPLOSIVE L3B STANDARD
Sample ID.....: 15-216-5:CA2
Sample type.....: Standard
Sample amount.....: 1.0000
Number of injections.....: 1
Bottle Number.....: 1

Retention time update.....: No update
Calibration level identifier.....: 3
Response factor update.....: No update

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000
PH.....: 0.000

User factors:
Volume (mL).....: 1.000
Dilution.....: 1.000
Amount (g or mL).....: 1000.000

PEAK INFORMATION

Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
1	7.858	4992	54642	0.575	4ADNT
2	8.240	7344	89287	0.617	2ADNT
Total		12336	143929	1.192	
Residual		0	0	0.000	

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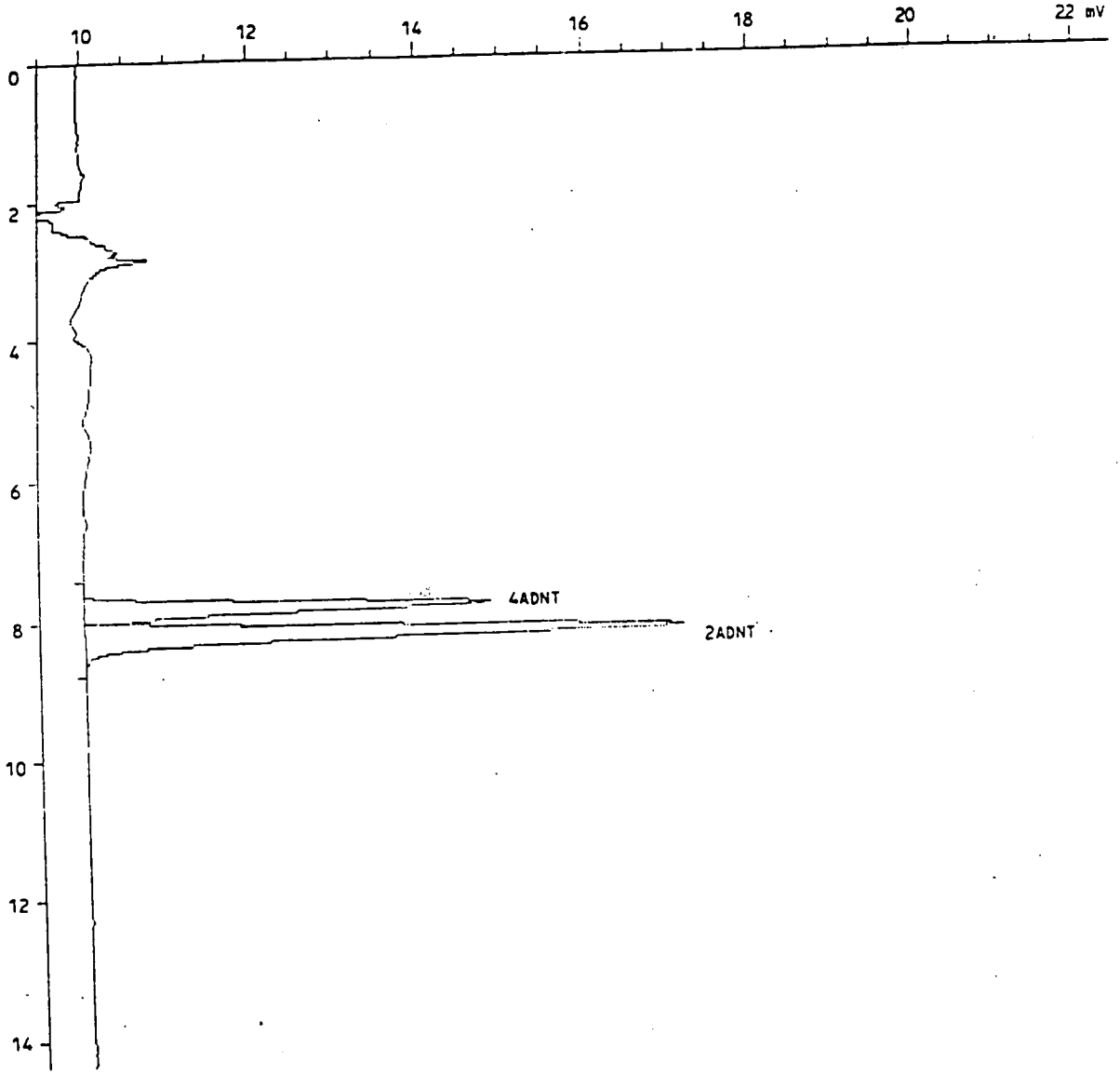
INJECTION REPORT

Injection F: <MC3> 1 1EX0711,43,1

Acquired on 12-Jul-96 at 16:45:18
Modified on 18-Jul-80 at 10:15:28
Reported on 18-Jul-96 at 10:24:44
INST ID: IN13, COL. TYPE: ODS-II, ID: ODS-10, INJ:200,COL#:1



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INJECTION REPORT

Injection F: <MC3> 1 1EX0711,43,1

Acquired on 12-Jul-96 at 16:45:18
Modified on 18-Jul-80 at 10:15:28
Reported on 18-Jul-96 at 10:25:03

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: PV
Comment.....: INST. ID: IN13, COL. TYPE: ODS-II, ID: ODS-10,INJ:200,COL#:1
Number of samples.: 49
Calibration file..: 1EX0711B Last modified on 18-Jul-80 at 10:16:30
Method file.....: EXPLOS Last modified on 18-Jul-80 at 10:24:58
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: EXPLOSIVE L3B STANDARD
Sample ID.....: 15-216-5:CA3
Sample type.....: Standard
Sample amount.....: 1.0000
Number of injections.....: 1
Bottle Number.....: 1

Retention time update.....: No update
Calibration level identifier.....: 3
Response factor update.....: No update

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000
PH.....: 0.000

User factors:
Volume (mL).....: 1.000
Dilution.....: 1.000
Amount (g or mL).....: 1000.000

PEAK INFORMATION

Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
1	7.813	4888	52561	0.553	4ADNT
2	8.196	7211	86298	0.596	2ADNT
Total		12098	138859	1.149	
Residual		0	0	0.000	

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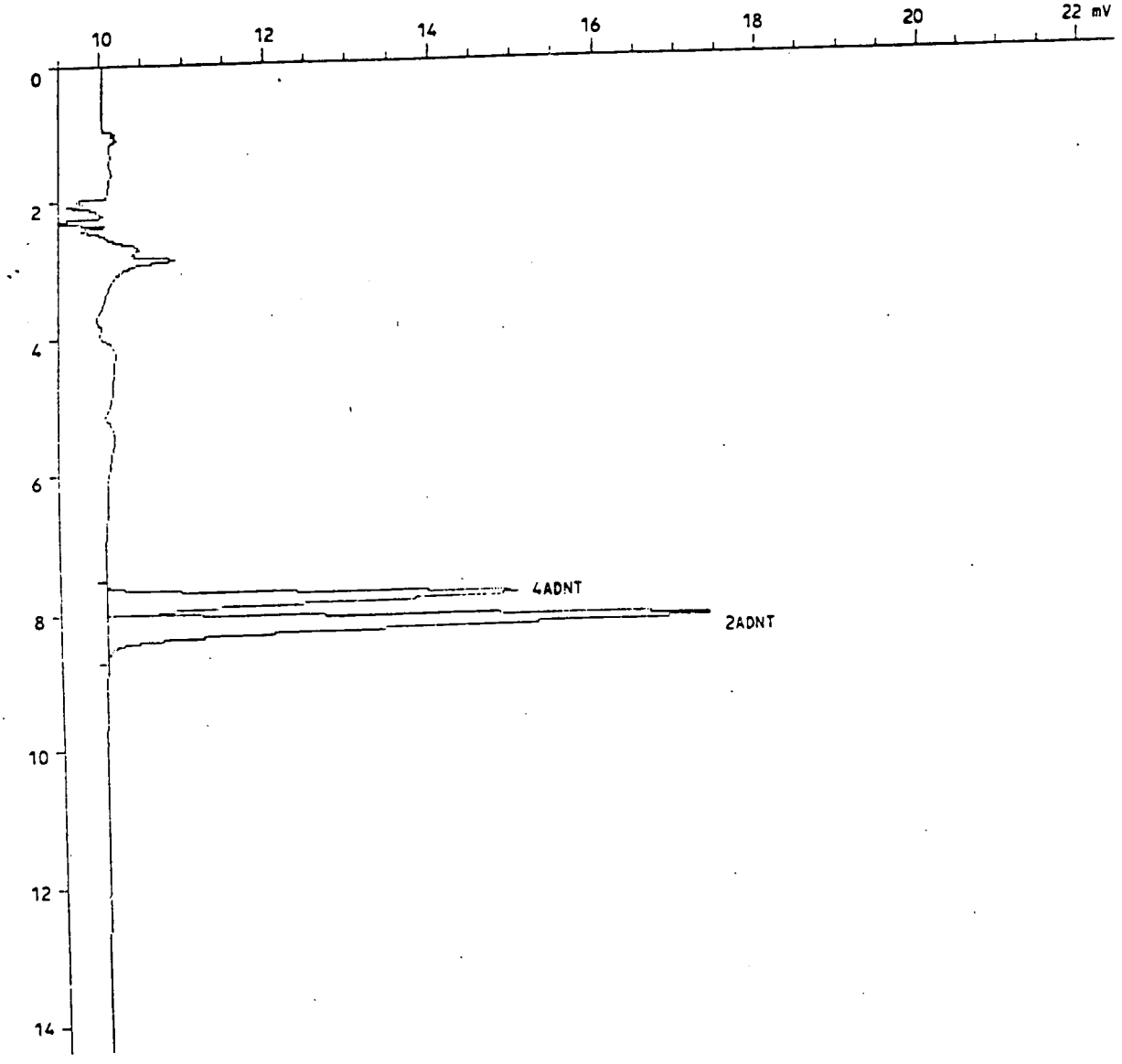
INJECTION REPORT

Injection F: <MC3> 1 1EX0711,49,1

Acquired on 12-Jul-96 at 19:42:26
Modified on 18-Jul-80 at 10:15:34
Reported on 18-Jul-96 at 10:25:43
INST ID: IN13, COL. TYPE: ODS-II, ID: ODS-10, INJ:200,COL#:1



F-65

INJECTION REPORT

Injection F: <MC3> 1 1EX0711,49,1

Acquired on 12-Jul-96 at 19:42:26
Modified on 1°-Jul-80 at 10:15:34
Reported on 18-Jul-96 at 10:25:24

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: PV
Comment.....: INST. ID: IN13, COL. TYPE: ODS-II, ID: ODS-10,INJ:200,COL#:1
Number of samples.: 49
Calibration file..: 1EX0711B Last modified on 18-Jul-80 at 10:16:30
Method file.....: EXPLOS Last modified on 18-Jul-80 at 10:24:58
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: EXPLOSIVE L3B STANDARD
Sample ID.....: 15-216-5:CA4
Sample type.....: Standard
Sample amount.....: 1.0000
Number of injections.....: 1
Bottle Number.....: 1

Retention time update.....: No update
Calibration level identifier.....: 3
Response factor update.....: No update

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000
PH.....: 0.000

User factors:
Volume (mL).....: 1.000
Dilution.....: 1.000
Amount (g or mL).....: 1000.000

PEAK INFORMATION

Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
1	7.804	5019	55010	0.579	4ADNT
2	8.182	7374	90359	0.624	2ADNT
Total		12393	145370	1.203	
Residual		0	0	0.000	

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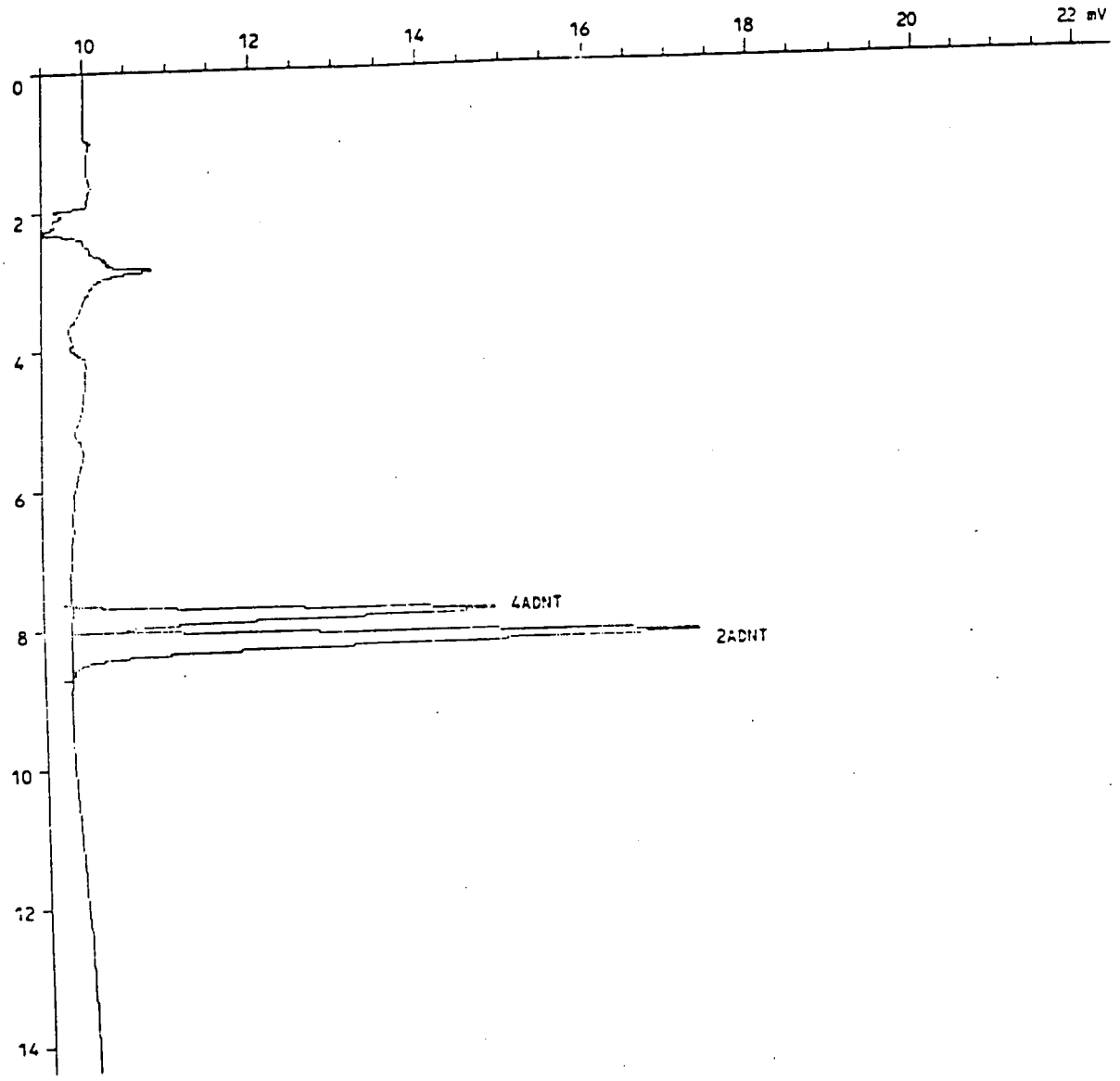
INJECTION REPORT

Injection F: <MC3> 1 1EX0711A,10,1

Acquired on 13-Jul-96 at 00:29:20
Modified on 18-Jul-80 at 10:15:46
Reported on 18-Jul-96 at 10:22:12
INST ID: IN13, COL. TYPE: ODS-II, ID: ODS-10, INJ:200,COL#:1



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INJECTION REPORT

Injection F: <MC3> 1 1EX0711A,10,1

Acquired on 13-Jul-96 at 00:29:20
Modified on 18-Jul-80 at 10:15:46
Reported on 18-Jul-96 at 10:21:53

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: PV
Comment.....: INST. ID: IN13, COL. TYPE: ODS-II, ID: ODS-10,INJ:200,COL#:1
Number of samples.: 20
Calibration file..: 1EX0711B Last modified on 18-Jul-80 at 10:16:30
Method file.....: EXPLOS Last modified on 18-Jul-80 at 10:21:36
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: EXPLOSIVE L3B STANDARD
Sample ID.....: 15-216-5:CA5
Sample type.....: Standard
Sample amount.....: 1.0000
Number of injections.....: 1
Bottle Number.....: 1

Retention time update.....: No update
Calibration level identifier.....: 3
Response factor update.....: No update

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000
PH.....: 0.000

User factors:
Volume (mL).....: 1.000
Dilution.....: 1.000
Amount (g or mL).....: 1000.000

PEAK INFORMATION

Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
1	7.831	5119	56665	0.596	4ADNT
2	8.213	7564	93782	0.648	2ADNT
Total		12683	150447	1.244	
Residual		0	0	0.000	

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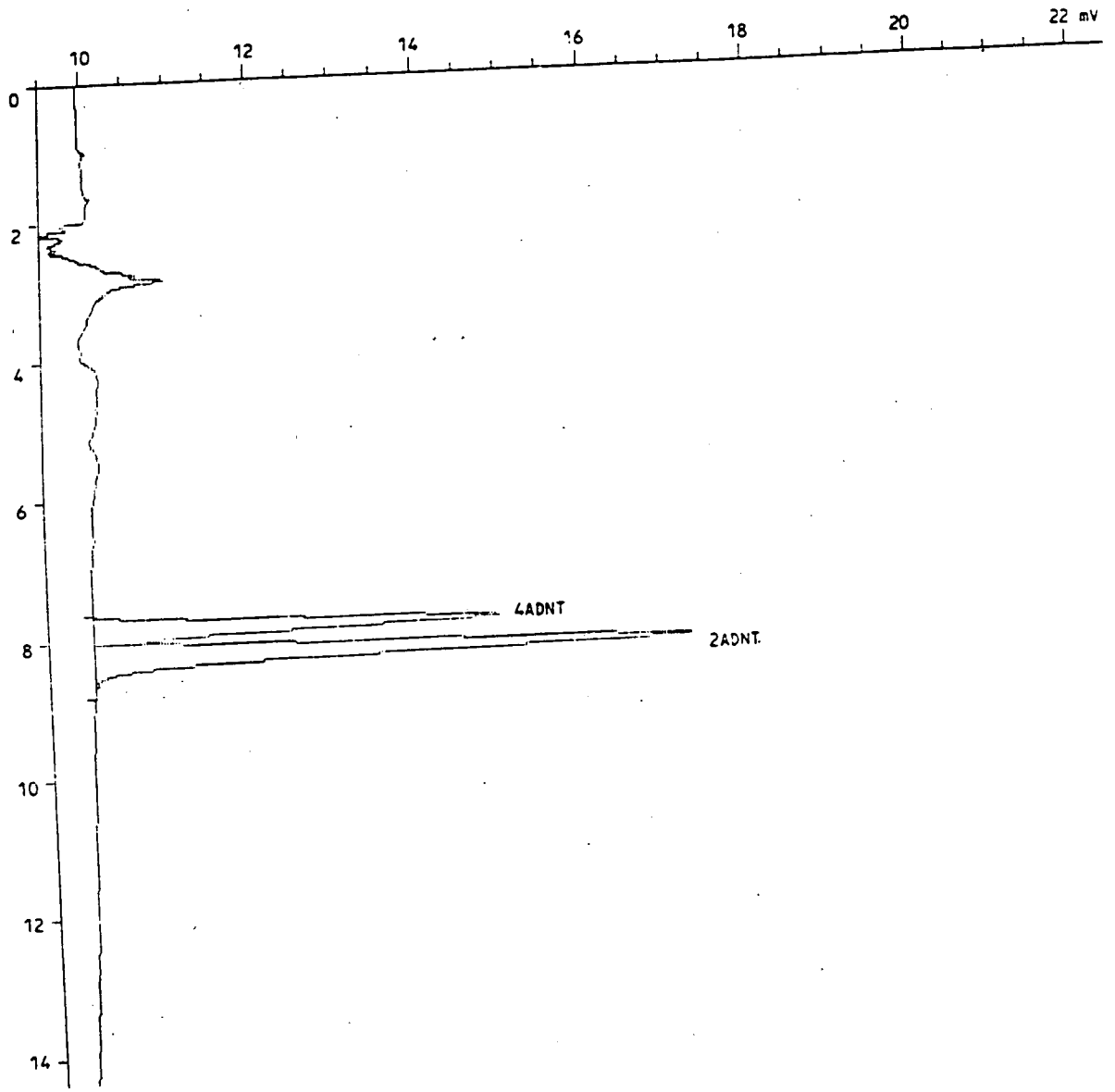
INJECTION REPORT

Injection F: <MC3> 1 1EX0711A,20,1

Acquired on 13-Jul-96 at 05:16:47
Modified on 18-Jul-80 at 10:15:52
Reported on 18-Jul-96 at 10:22:55
INST ID: IN13, COL. TYPE: ODS-II, ID: ODS-10, INJ:200,COL#:1



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INJECTION REPORT

Injection F: <MC3> 1 1EX0711A,20,1

Acquired on 13-Jul-96 at 05:16:47
Modified on 18-Jul-80 at 10:15:52
Reported on 18-Jul-96 at 10:22:36

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: PV
Comment.....: INST. ID: IN13, COL. TYPE: ODS-II, ID: ODS-10,INJ:200,COL#:1
Number of samples.: 20
Calibration file..: 1EX0711B Last modified on 18-Jul-80 at 10:16:30
Method file.....: EXPLOS Last modified on 18-Jul-80 at 10:22:26
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: EXPLOSIVE L3B STANDARD
Sample ID.....: 15-216-5:CA6
Sample type.....: Standard
Sample amount.....: 1.0000
Number of injections.....: 1
Bottle Number.....: 1

Retention time update.....: No update
Calibration level identifier.....: 3
Response factor update.....: No update

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000
PH.....: 0.000

User factors:
Volume (mL).....: 1.000
Dilution.....: 1.000
Amount (g or mL).....: 1000.000

PEAK INFORMATION

Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
1	7.836	4891	54540	0.574	4ADNT
2	8.218	7235	90240	0.623	2ADNT
Total		12126	144780	1.197	
Residual		0	0	0.000	

EXPLOSIVES ANALYSIS DATA SHEET

EPA SAMPLE NO:

BL0701SA

Lab Name: SWL-TULSA

Lab Code: SWOK Case No: SDG:

Matrix: (soil/water) SOIL Lab Sample ID: BL0701SA

Sample Amt: 2g % Moisture 0 Date Received:

Extraction Volume: 10.00mL Date Extracted: 07/01/96

Extraction Method: SONC Date Analyzed: 07/11/96

GPC Clean-up (Y/N) N Dilution Factor: 2

COMPOUND	CONCENTRATION UNITS: (ug/L or ug/Kg) ug/Kg	Q
HMX	2200	U
RDX	1000	U
TNB	250	U
TETRYL	650	U
DNB	250	U
TNT	250	U
NB	260	U
26DNT	260	U
24DNT	250	U
2NT	250	U
4NT	250	U
3NT	250	U
2ADNT	300	U
4ADNT	300	U

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FORM I

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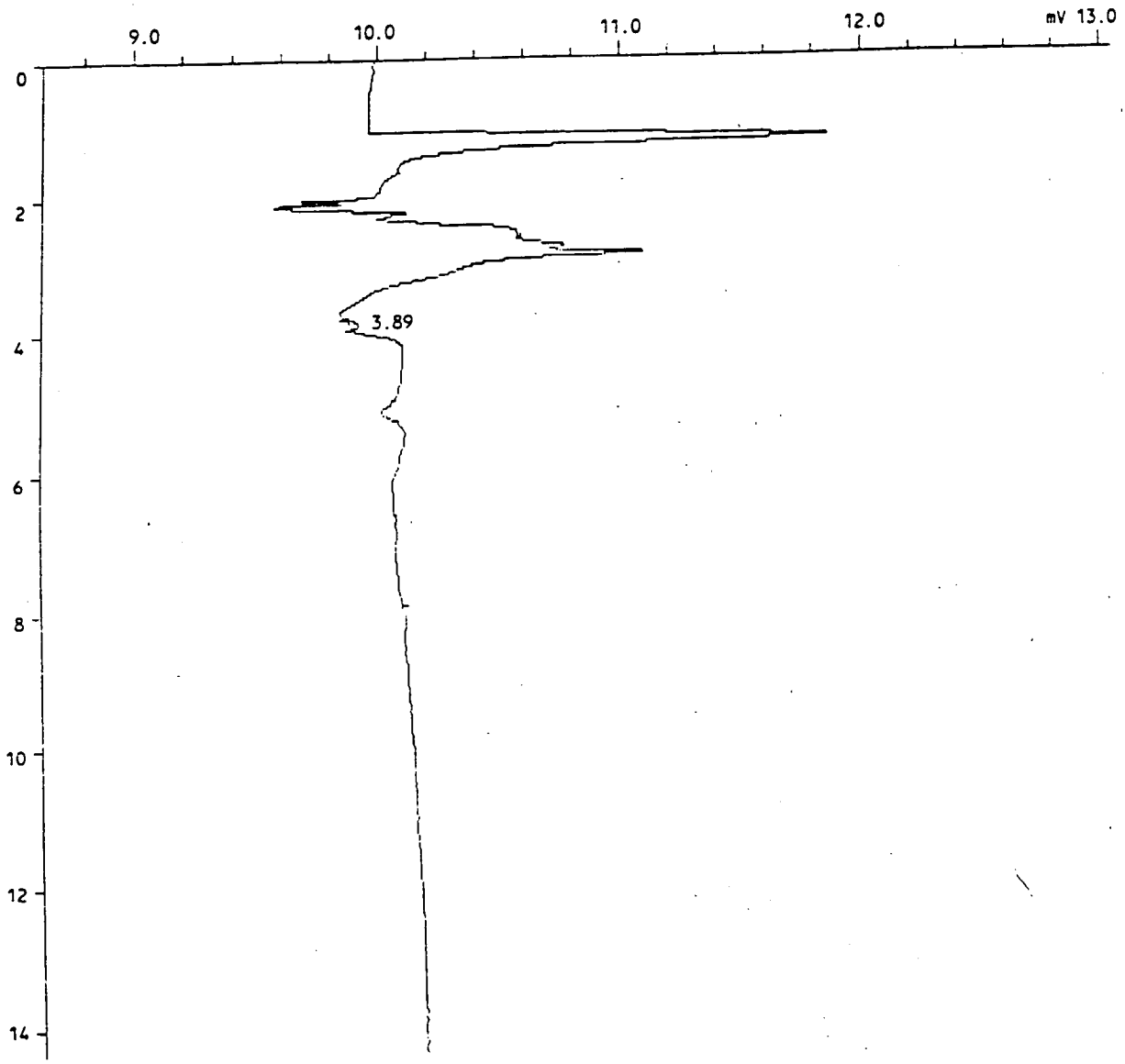
INJECTION REPORT

Injection F: <MC3> 1 1EX0711,14,1

Acquired on 11-Jul-96 at 21:56:29
Modified on 18-Jul-80 at 10:54:02
Reported on 18-Jul-96 at 11:19:37
INST ID: IN13, COL. TYPE: ODS-II, ID: ODS-10, INJ:200,COL#:1



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INJECTION REPORT

Injection F: <MC3> 1 1EX0711,14,1

Acquired on 11-Jul-96 at 21:56:29
Modified on 18-Jul-80 at 10:54:02
Reported on 18-Jul-96 at 10:54:04

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: PV
Comment.....: INST. ID: IN13, COL. TYPE: ODS-II, ID: ODS-10,INJ:200,COL#:1
Number of samples.: 49
Calibration file..: 1EX0711 Last modified on 18-Jul-80 at 10:54:02
Method file.....: EXPLOS Last modified on 18-Jul-80 at 10:53:54
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: BL0701SA
Sample ID.....: BL0701SA
Sample type.....: Sample
Sample amount.....: 2.0000
Number of injections.....: 1
Bottle Number.....: 1

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000
PH.....: 0.000

User factors:
Volume (mL).....: 10.000
Dilution.....: 2.000
Amount (g or mL).....: 2.000

PEAK INFORMATION

Peak RT mins	Hght uV	Area uVs	ug/Kg	Peak name
Total	0	0	0.000	
Residual	35	172	-1.345	

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EXPLOSIVES ANALYSIS DATA SHEET

EPA SAMPLE NO:

BL0701SB

Lab Name: SWL-TULSA

Lab Code: SWOK Case No: SDG:

Matrix: (soil/water) SOIL Lab Sample ID: BL0701SB

Sample Amt: 2g % Moisture 0 Date Received:

Extraction Volume: 10.00mL Date Extracted: 07/01/96

Extraction Method: SONC Date Analyzed: 07/12/96

GPC Clean-up (Y/N) N Dilution Factor: 2

COMPOUND	CONCENTRATION UNITS: (ug/L or ug/Kg) ug/Kg	Q
HMX	2200	U
RDX	1000	U
TNB	250	U
TETRYL	650	U
DNB	250	U
TNT	250	U
NB	260	U
26DNT	260	U
24DNT	250	U
2NT	250	U
4NT	250	U
3NT	250	U
2ADNT	300	U
4ADNT	300	U

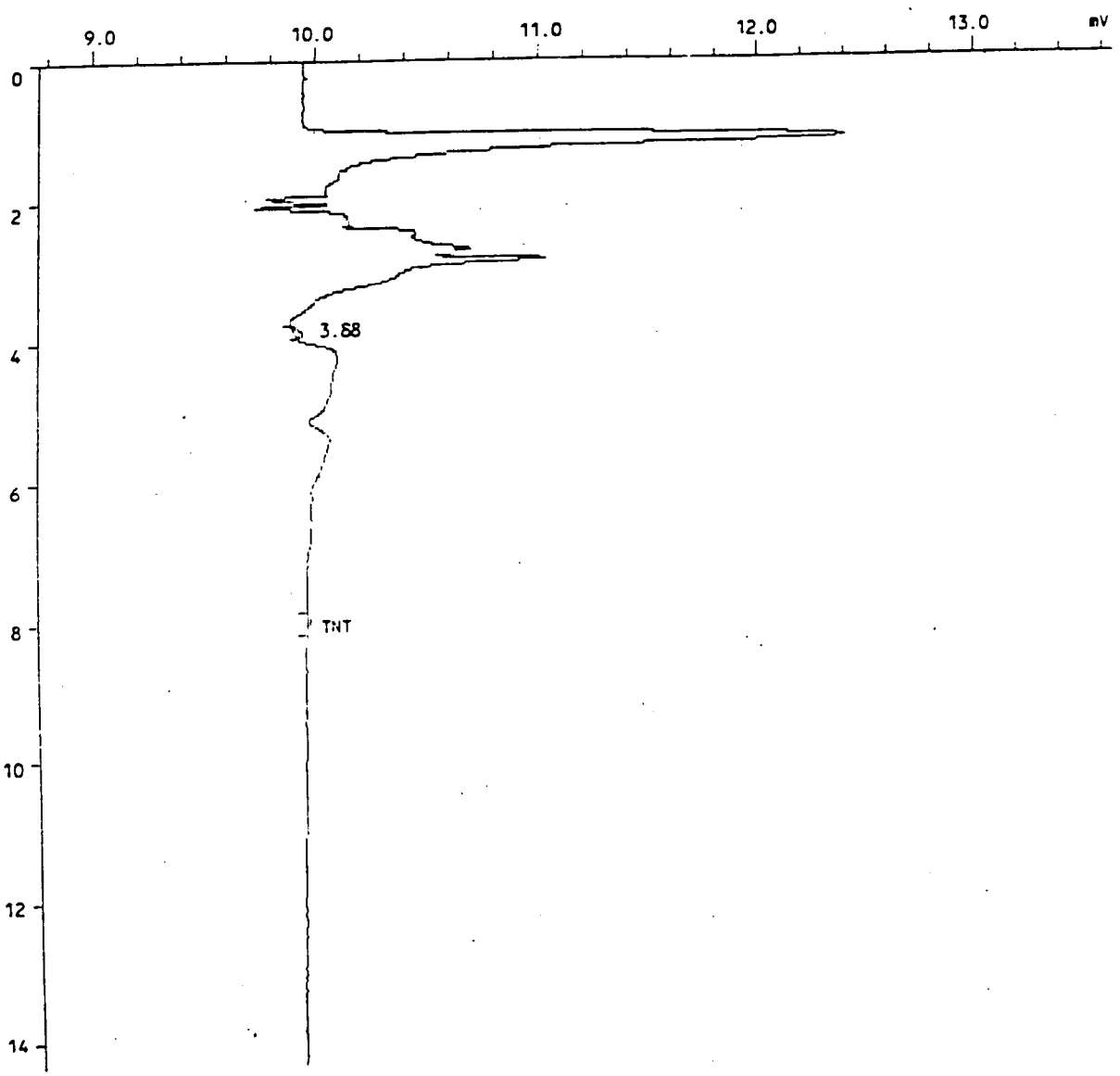
01/07/97

FORM I

INJECTION REPORT

Injection F: <MC3> 1 1EX0711A,1,1

Acquired on 12-Jul-96 at 20:11:06
Modified on 19-Jul-80 at 09:38:18
Reported on 19-Jul-96 at 09:45:28
INST ID: IN13, COL. TYPE: ODS-II, ID: ODS-10, INJ:200,COL#:1



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INJECTION REPORT

Injection F: <MC3> 1 1EX0711A,1,1

Acquired on 12-Jul-96 at 20:11:06
Modified on 19-Jul-80 at 09:38:18
Reported on 19-Jul-96 at 09:38:35

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: PV
Comment.....: INST. ID: IN13, COL. TYPE: ODS-II, ID: ODS-10,INJ:200,COL#:1
Number of samples.: 20
Calibration file..: 1EX0711 Last modified on 19-Jul-80 at 09:38:18
Method file.....: EXPLOS Last modified on 19-Jul-80 at 09:37:34
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: BL0701SB
Sample ID.....: BL0701SB
Sample type.....: Sample
Sample amount.....: 2.0000
Number of injections.....: 1
Bottle Number.....: 1

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000
PH.....: 0.000

User factors:
Volume (mL).....: 10.000
Dilution.....: 2.000
Amount (g or mL).....: 2.000

PEAK INFORMATION

Peak	RT mins	Hght uV	Area uVs	ug/Kg	Peak name
2	7.987	10	74	5.009	TNT
Total		10	74	5.009	
Residual		38	197	-1.543	

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Quanterra Environmental Services

Quanterra Incorporated
880 Riverside Parkway
West Sacramento, California 95605

916 373-5600 Telephone
916 372-1059 Fax

August 7, 1998

QUANTERRA INCORPORATED PROJECT NUMBER: 300612
PO/CONTRACT: [redacted]

[redacted]

Dear Mr. [redacted]

This report contains the analytical results for the one sample received under chain of custody by Quanterra Incorporated on July 25, 1998. This sample is associated with your [redacted] project.

The case narrative is an integral part of this report.

If you have any questions, please feel free to call me at (916)374-4362.

Sincerely,

Diana Brooks
Project Manager

F1-1

Quanterra

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- Initial Calibration
- Continuing Calibration
- Method Blank Data
- Laboratory Control Sample Data
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Quanterra

CASE NARRATIVE

QUANTERRA INCORPORATED PROJECT NUMBER 300612

There were no anomalies associated with this project.

Quanterra

Quanterra - Western Region

Quality Control Definitions

QC Parameter	Definition
QC Batch	A set of up to 20 field samples plus associated laboratory QC samples that are similar in composition (matrix) and that are processed within the same time period with the same reagent and standard lots.
Duplicate Control Sample (DCS)	Consist of a pair of LCSs analyzed within the same QC batch to monitor precision and accuracy independent of sample matrix effects. This QC is performed only if required by client or when insufficient sample is available to perform MS/MSD.
Duplicate Sample (DU)	A second aliquot of an environmental sample, taken from the same sample container when possible, that is processed independently with the first sample aliquot. The results are used to assess the effect of the sample matrix on the precision of the analytical process. The precision estimated using this sample is not necessarily representative of the precision for other samples in the batch.
Laboratory Control Sample (LCS)	A volume of reagent water for aqueous samples or a contaminant-free solid matrix (Ottawa sand) for soil and sediment samples which is spiked with known amounts of representative target analytes and required surrogates. An LCS is carried through the entire analytical process and is used to monitor the accuracy of the analytical process independent of potential matrix effects.
Matrix Spike and Matrix Spike Duplicate (MS/MSD)	A field sample fortified with known quantities of target analytes that are also added to the LCS. Matrix spike duplicate is a second matrix spike sample. MSs/MSDs are carried through the entire analytical process and are used to determine sample matrix effect on accuracy of the measurement system. The accuracy and precision estimated using MS/MSD is only representative of the precision of the sample that was spiked.
Method Blank (MB)	A sample composed of all the reagents (in the same quantities) in reagent water carried through the entire analytical process. The method blank is used to monitor the level of contamination introduced during sample preparation steps.
Surrogate Spike	Organic constituents not expected to be detected in environmental media and are added to every sample and QC at a known concentration. Surrogates are used to determine the efficiency of the sample preparation and the analytical process.

Source: Quanterra® Quality Control Program, Policy QA-003, Rev. 0, 8/19/96.

Quanterra

SAMPLE DESCRIPTION INFORMATION
for
[redacted] Corporation

Lab ID	Client ID	Matrix	Sampled Date	Sampled Time	Received Date
300612-0001-SA	[redacted]	AQUEOUS	24 JUL 98	12:30	25 JUL 98

Chain of Custody Record

Quanterra

OUA-4124

Client	Project Manager	Date	Chain Of Custody Number 31517
Address	Telephone Number (Area Code)/Fax Number	Lab Number	Page _____ of _____
City / State / Zip Code	Site Contact		
Project Name	Carrier/Waybill Number		
Contract/Purchase Order/Quote No.			

Sample I.D. No. and Description	Date	Time	Sample Type	Total Volume	Containers Type	Containers No.	Preservative	Condition on Receipt	Analysis

Special Instructions

Possible Hazard Identification
☐ Non-Hazard ☐ Flammable ☐ Skin Irritant ☐ Poison B ☐ Unknown

Sample Disposal
☐ Return To Client ☐ Disposal By Lab ☐ Archive For _____ Months

Turn Around Time Required
☐ Normal ☐ Rush

QC Level
☐ I. ☐ II. ☐ III.

Project Specific (Specify)

	Date	Time		Date	Time
1. Relinquished By			1. Received By		
2. Relinquished By			2. Received By		
3. Relinquished By			3. Received By		

Comments

DISTRIBUTION: W[illegible] - Stays with Sample; CANARY - Returned to Client with Report; PINK - Field Copy

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Quanterra

Nitroaromatics and Nitramines (Aqueous) - Method 8330

Quanterra

Nitroaromatics and Nitramines by HPLC
Method 8330

Client Name: [redacted]
Client ID: [redacted]
LAB ID: 300612-0001-SA
Matrix: AQUEOUS
Authorized: 25 JUL 98

Sampled: 24 JUL 98
Prepared: 28 JUL 98

Received: 25 JUL 98
Analyzed: 04 AUG 98

Dilution Factor: 1.0

Parameter	Result	Units	Reporting Limit	Qualifier
RDX	1.3	ug/L	5.0	J
1,3-Dinitrobenzene	ND	ug/L	5.0	
Nitrobenzene	ND	ug/L	5.0	
2,4,6-Trinitrotoluene	ND	ug/L	5.0	
Tetryl	ND	ug/L	5.0	
2,4-Dinitrotoluene	ND	ug/L	5.0	
2,6-Dinitrotoluene	ND	ug/L	5.0	
2-Am-DNT	ND	ug/L	5.0	
4-Am-DNT	ND	ug/L	5.0	
2-Nitrotoluene	ND	ug/L	10	
4-Nitrotoluene	ND	ug/L	8.5	
3-Nitrotoluene	ND	ug/L	7.9	

Surrogate	Recovery	Acceptable Range
2,4-Dinitrofluorobenzene	87 %	65 - 135

Note J = Result is detected below the reporting limit or is an estimated concentration.
ND = Not Detected

Reported By: Jon Edmondson

Approved By: Emily Uebelhoer

The cover letter is an integral part of this report.
Rev 230787

Quanterra

QC LOT ASSIGNMENT REPORT
HPLC Analysis Area

Laboratory Sample Number	QC Matrix	QC Category	QC Lot Number (DCS)	QC Run Number (LCS/BLANK)
300612-0001-SA	AQUEOUS	8330-COE-A	28 JUL 98-13D	28 JUL 98-13D

Quanterra

METHOD BLANK REPORT
HPLC Analysis Area
Project: 300612

Test: 8330-IRPMS-1C-A Nitroaromatics and Nitramines by HPLC
Method: 8330
Matrix: AQUEOUS
QC Lot: 28 JUL 98-13D QC Run: 28 JUL 98-13D
Analyzed: 04 AUG 98 Time: 18:16

Analyte	Result	Units	Reporting Limit	Qualifier
RDX	ND	ug/L	5.0	
1,3-Dinitrobenzene	ND	ug/L	5.0	
Nitrobenzene	ND	ug/L	5.0	
2,4,6-Trinitrotoluene	ND	ug/L	5.0	
Tetryl	ND	ug/L	5.0	
2,4-Dinitrotoluene	ND	ug/L	5.0	
2,6-Dinitrotoluene	ND	ug/L	5.0	
2-Am-DNT	ND	ug/L	5.0	
4-Am-DNT	ND	ug/L	5.0	
2-Nitrotoluene	ND	ug/L	10	
4-Nitrotoluene	ND	ug/L	8.5	
3-Nitrotoluene	ND	ug/L	7.9	

Surrogate	% Recovery	Acceptable Range
2,4-Dinitrofluorobenzene	89	65 -135

ND = Not Detected

Quanterra

DUPLICATE CONTROL SAMPLE REPORT
HPLC Analysis Area
Project: 300612

Category: 8330-COE-A Explosives by HPLC
Testcode: 8330-IRPMS-1C-A Method: 8330
Matrix: AQUEOUS Concentration Units: ug/L
QC Lot: 28 JUL 98-13D Analyzed Date: 04 AUG 98 Time: 19:28

Analyte	Concentration Spiked	Measured DCS1	Measured DCS2	Accuracy (%) DCS1	Accuracy (%) DCS2	Limits	Precision (RPD) DCS	Limit
HMX	0.500	0.468	0.473	94	95	65-135	1.2	35
1,3,5-Trinitrobenzene	0.500	0.468	0.470	94	94	65-135	0.51	35
RDX	0.500	0.432	0.453	86	91	65-135	4.6	35
1,3-Dinitrobenzene	0.500	0.425	0.461	85	92	65-135	8.1	35
Nitrobenzene	0.500	0.369	0.458	74	92	65-135	22	35
2,4,6-Trinitrotoluene	0.500	0.511	0.495	102	99	65-135	3.2	35
Tetryl	0.500	0.510	0.512	102	102	65-135	0.47	35
2,4-Dinitrotoluene	0.500	0.486	0.485	97	97	65-135	0.12	35
2,6-Dinitrotoluene	0.500	0.432	0.487	86	97	65-135	12	35
2-Am-DNT	0.500	0.453	0.478	91	96	65-135	5.2	35
4-Am-DNT	0.500	0.482	0.456	96	91	65-135	5.5	35
2-Nitrotoluene	0.500	0.414	0.504	33	101	65-135	19	35
4-Nitrotoluene	0.500	0.413	0.552	83	110	65-135	29	35
3-Nitrotoluene	0.500	0.430	0.523	86	105	65-135	20	35

Surrogate	Concentration Spiked	Measured DCS1	Measured DCS2	Accuracy(%) DCS1	Accuracy(%) DCS2	Limits
2,4-Dinitrofluorobenzene	2.6	2.4	2.4	90	92	65-135

Calculations are performed before rounding to avoid round-off errors in calculated results.

RUSH TAT

QUANTERRA - SACRAMENTO
PROJECT DETAIL: GC DEPARTMENT

PROJECT ID: 300612
CLIENT MANAGER: DBROOKS TEAM: Environmental Servic
PROJECT DUE DATE: 06 AUG 98
CLIENT/PROJECT NAME: [redacted] 072598

8330-IRPMS-1C-A (90.3) 1.(T: 1).

DATE DELIVERED FOR LIMS ENTRY : 05 AUG 98 TIME DELIVERED: 16:46

LIMS ENTER: _____ - 98 Formaster ENTER: _____ - 98

DATE TO FINAL REVIEW: _____ - 98 PEER: _____ - 98

FINAL: a 8/5 - 98 FINAL: _____ - 98

DATE DELIVERED TO DATA CONTROL: 8/5 - 98

_____ No problems (d+i) X See Anomaly Sheet (d+i)

COMMENTS:

GC-0037 (1.0) 7/9/91

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Organics Raw Data Package

FF 13

Sample Data

Nitroaromatics and Nitramines by HPLC
Method 8330

Client Name: [redacted]
Client ID: [redacted]
LAB ID: 300612-0001-SA
Matrix: AQUEOUS
Authorized: 25 JUL 98

Sampled: 24 JUL 98
Prepared: 28 JUL 98

Received: 25 JUL 98
Analyzed: 04 AUG 98

Dilution Factor: 1.0

Parameter	Result	Units	Reporting Limit	Qualifier
RDX	1.3	ug/L	5.0	J
1,3-Dinitrobenzene	ND	ug/L	5.0	
Nitrobenzene	ND	ug/L	5.0	
2,4,6-Trinitrotoluene	ND	ug/L	5.0	
Tetryl	ND	ug/L	5.0	
2,4-Dinitrotoluene	ND	ug/L	5.0	
2,6-Dinitrotoluene	ND	ug/L	5.0	
2-Am-DNT	ND	ug/L	5.0	
4-Am-DNT	ND	ug/L	5.0	
2-Nitrotoluene	ND	ug/L	10	
4-Nitrotoluene	ND	ug/L	8.5	
3-Nitrotoluene	ND	ug/L	7.9	

Surrogate	Recovery	Acceptable Range
2,4-Dinitrofluorobenzene	87 %	65 - 135

Note J = Result is detected below the reporting limit or is an estimated concentration.
ND = Not Detected

Reported By: Jon Edmondson

Approved By: Emily Uebelhoer

The cover letter is an integral part of this report.
Rev 230787

Injection Report

Acquired on 4-AUG-1998 at 23:06

Quanterra, Inc (West Sacramento)

Analyst Name : LC4UV
Lims Id : 109LC4UV9050
Comment : 75:25:0/2-22-50:00:50/2"- H2O:2%THF/MeOH:Methanol (Analysis)
Method Title : Var-LC5500 UV Hypersil-C8 150x4.6mm 5um SN251486 8330M
Sample Name : 300612-1 770ML/20ML
Sample Id :
Sample Type : Sample Amount=1.00000
Bottle No : 8

PEAK INFORMATION

Peak	RT mins	RT Exp	Hght uV	ppb	Peak name	Width
1	3.422	0.000	18	0.00000		8.0
2	3.804	3.769	3377	7.31382	HMX	5.3
3	7.031	6.907	445	1.29424	RDX	11.7
4	8.000	0.000	130	0.00000		13.9
5	8.569	8.476	1564	2.25634	2,4-Dinitrofluorobenzene(Surr)	12.3
6	10.622	0.000	81	0.00000		12.8
7	13.138	0.000	127	0.00000		20.3
8	13.956	14.078	17	0.03354	TETRYL	11.2
9	14.453	0.000	29	0.00000		13.9
10	18.187	0.000	24	0.00000		18.1
11	21.467	0.000	74	0.00000		32.0
12	23.600	0.000	37	0.00000		14.9
13	24.373	0.000	64	0.00000		3.2
14	24.871	0.000	199	0.00000		5.3

Totals		
Unknowns	783	0.00000
Named	5403	10.89795
Grand Total	6186	10.89795

ANALYSIS SUMMARY

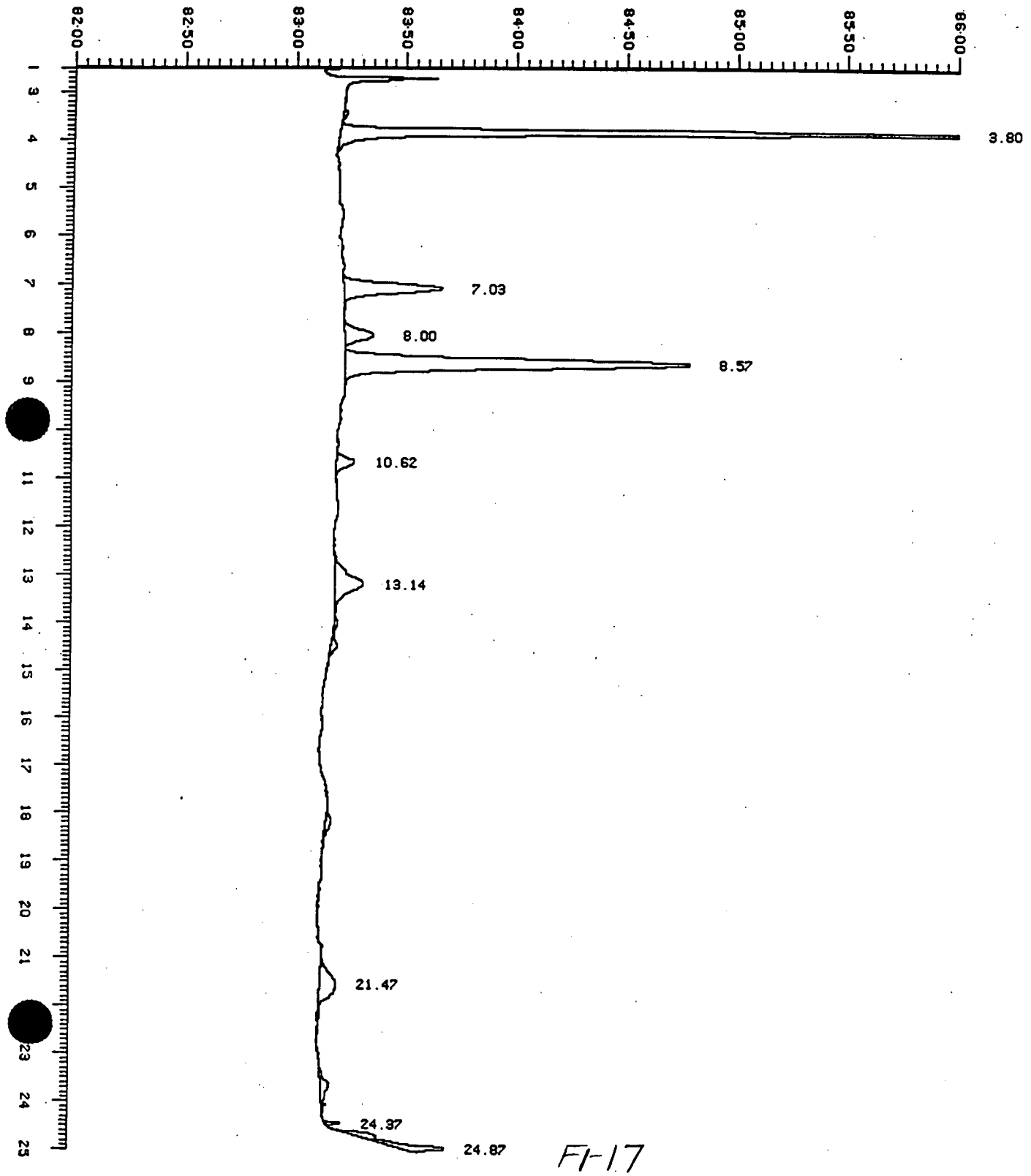
Method..... 8330 HY
Run sequence..... EXP4HY1
Calibration..... 03AUG98
External standard calibration using height
Calibration last modified on 4-AUG-1998 at 22:55
Calibration initialised on 12-DEC-1997 at 21:47
Method last modified on 4-AUG-1998 at 16:40

Uncalibrated peaks use user factor (0.0000)

Quanterra, Inc (West Sacramento)

[AUG_SV] 109 4H_04AUG981450,10,1

300612-1 770ML/20ML. Amount : 1.000.
Varian 5560/UV9050 @250nm flow=1.5 injection volume=200uL
Acquired on 4-AUG-1998 at 23:06
Reported on 4-AUG-1998 at 23:31
Box 1 (of 1)



F1-17

Quanterra / W. Sacramento
Chromatography Summary

Analysis ID: [AUG_SV] 109 4H_04AUG981450,10
Acquired: HPLC #4 4-AUG-1998 at 23:06

Sample: 300612-1 770ML/20ML **Mult:** 20.00 **Dilf:** 1.00 **Div:** 770.0
Method File: 8330_HY 75:25:0/2-22-50:00:50/2"- H2O:2%THF/MeOH:Methanol (Analysis)
Calibration: 03AUG98 8330H 2.5-1000ng/mL Phenomenex Hypersil C8 SN251486

Target Analyte Results

Channel 109 Var-LC5500 UV
Hypersil-C8
150x4.6mm 5um SN251486 8330M

	Peak Name	RT	Diff	Height	~~ppb~~	R	P	RL
1	HMX	3.804	+0.04	3377	7.3138			NA
2	1,3,5-Trinitrobenz							
3	RDX	7.031	+0.12	445	1.2942			5.00
4	2,4-Dinitrofluorob	8.569	+0.09	1564	2.2563		F	
5	1,3-Dinitrobenzene							5.00
6	Nitrobenzene							5.00
7	2,4,6-Trinitrotolu							5.00
8	TETRYL	13.956	-0.12	17	0.0335	U	F	5.00
9	2,4-Dinitrotoluene							5.00
10	2,6-Dinitrotoluene							5.00
11	2-AM-4,6-Dinitroto							5.00
12	4-AM-2,6-Dinitroto							5.00
13	2-Nitrotoluene							10.0
14	4-Nitrotoluene							8.50
15	3-Nitrotoluene							7.90

Surrogate Results

	Peak Name	Amount Spiked	Amount Found	%Rec	-Limits-
4	2,4-Dinitrofluorob	2.60	2.2563	87	(65 - 135)

Notes:
8330-IRPMS-1C-A

Range Flags (R)
U - Under calibration Range
O - Over calibration Range
X - Not calibrated

Peak Type Flags (P)
* - Flat top Peak
F - Forced baseline
M - Modified baseline

Q/WS SUM 3.29A

Reviewer: a Date: 9/5/98
Printed: 08/04/98 23:31:27

FH18

Retention Time Windows

Lab Name: QUANTERRA SACRAMENTO (8330 RT WINDOWS)
Instrument ID: LC#4
Column: Hypersil C8
Serial #: 237022

	DATE	TIME
STANDARD 1:	07/28/98	13:55
STANDARD 2:	07/28/98	19:38
STANDARD 3:	07/29/98	09:17

Component	RT 1	RT 2	RT 3	3X STD. DEVIATION
HMX	3.689	3.724	3.751	0.093
1,3,5-Trinitrobenzene	6.533	6.507	6.498	0.055
RDX	6.942	6.924	6.916	0.040
2,4-Dinitrofluorobenzen	8.578	8.560	8.551	0.041
1,3-Dinitrobenzene	9.564	9.547	9.538	0.040
Nitrobenzene	11.396	11.378	11.440	0.096
2,4,6-Trinitrotoluene	12.924	12.889	12.907	0.053
TETRYL	14.302	14.231	14.213	0.141
2,4-Dinitrotoluene	15.671	15.609	15.644	0.093
2,6-Dinitrotoluene	16.871	16.800	16.853	0.111
2-AM-4,6-Dinitrotoluene	17.476	17.404	17.422	0.112
4-AM-2,6-Dinitrotoluene	18.142	18.089	18.080	0.101
2-Nitrotoluene (o)	18.987	18.951	19.031	0.120
4-Nitrotoluene (p)	19.378	19.333	19.431	0.147
3-Nitrotoluene (m)	20.213	20.187	20.267	0.122

Initial Calibration

Quanterra / W. Sacramento **Calibration ID:** [AUG_SV] 109 03AUG98
Calibration Statistics Summary **Instrument ID:** ICY

File: 03AUG98 **Title:** 8330H 2.5-1000ng/mL Phenomenex Hypersil C8 SN251486
Curve Type: Line forced thro' zero (AVG RF) **Weighting:** 1/X sqd

 # Peaks: 15 **Calibration based on:** Height

RT	Component Name	Calibration Factor	RSD %	Dynamic Range Low	Dynamic Range High
3.77	HMX	11.993	3.7	5.00	1000
6.49	1,3,5-Trinitrobenzene	21.426	9.5	5.00	1000
6.91	RDX	8.9406	6.4	5.00	1000
8.48	2,4-Dinitrofluorobenzene(Surr)	17.998	4.6	5.00	150
9.44	1,3-Dinitrobenzene	21.547	8.7	2.50	1000
11.27	Nitrobenzene	10.802	6.0	5.00	1000
12.73	2,4,6-Trinitrotoluene (TNT)	13.462	6.9	5.00	1000
14.08	TETRYL	13.361	3.5	5.00	1000
15.41	2,4-Dinitrotoluene	14.020	8.6	2.50	1000
16.58	2,6-Dinitrotoluene	8.3239	3.1	5.00	1000
17.18	2-AM-4,6-Dinitrotoluene	10.488	8.4	5.00	1000
17.84	4-AM-2,6-Dinitrotoluene	8.7060	8.6	5.00	1000
18.69	2-Nitrotoluene	5.3495	12	10.0	1000
19.08	4-Nitrotoluene	4.3077	10	10.0	1000
19.94	3-Nitrotoluene	5.6389	6.4	10.0	1000

Analyst: JEDMONDSON Date: 04-AUG-1998 Reviewed: MHR Date: 8/5/98
CalibStats Ver 2.0 Printed at: 04-AUG-1998 16:44:34

F1-22

Quanterra / W. Sacramento
Calibration Statistics Report

Calibration ID: [AUG SV] 109 03AUG98
Instrument ID: LC4

File: 03AUG98 Title: 8330H 2.5-1000ng/mL Phenomenex Hypersil C8 SN251486
Curve Type: Line forced thro' zero (AVG RF) Weighting: 1/X sqd
Quant Base: Height

==========

HMX RT: 3.77 min Window: 3.57 - 3.97 min

Level	ID	Amount/Conc'n	Response Factor
1	1	5.0000	11.772
2	2	10.000	12.276
3	3	25.000	11.789
4	5	100.00	11.308
5	6	500.00	12.501
6	7	1000.0	12.312

Calib Factor: 11.993 RSD: 3.7%

1,3,5-Trinitrobenzene RT: 6.49 min Window: 6.29 - 6.69 min

Level	ID	Amount/Conc'n	Response Factor
1	1	5.0000	18.919
2	2	10.000	20.209
3	3	25.000	20.936
4	5	100.00	20.697
5	6	500.00	23.882
6	7	1000.0	23.916

Calib Factor: 21.426 RSD: 9.5%

RDX RT: 6.91 min Window: 6.71 - 7.11 min

Level	ID	Amount/Conc'n	Response Factor
1	1	5.0000	8.5905
2	2	10.000	8.6367
3	3	25.000	9.2256
4	5	100.00	8.1404
5	6	500.00	9.5120
6	7	1000.0	9.5382

Calib Factor: 8.9406 RSD: 6.4%

2,4-Dinitrofluorobenzene(Surr) RT: 8.48 min Window: 8.23 - 8.73 min

Level	ID	Amount/Conc'n	Response Factor
1	T1	5.0000	19.021
2	1	25.000	16.885
3	2	50.000	18.272
5	3	100.00	18.313
6	5	150.00	17.500

Calib Factor: 17.998 RSD: 4.6%

==========

Analyst: JEDMONDSON Date: 04-AUG-1998 Reviewed: MH Date: 8/5/98
CalibStats Ver 2.0 Printed at: 04-AUG-1998 16:44:34

Quanterra / W. Sacramento
Calibration Statistics Report

Calibration ID: [AUG_SV] 109 03AUG98
Instrument ID: LC4

File: 03AUG98 Title: 8330H 2.5-1000ng/mL Phenomenex Hypersil C8 SN251486
Curve Type: Line forced thro' zero (AVG RF) Weighting: 1/X sqd
Quant Base: Height

1,3-Dinitrobenzene RT: 9.44 min Window: 9.24 - 9.64 min

Level	ID	Amount/Conc'n	Response Factor
1	T1	2.5000	22.319
2	1	5.0000	18.866
3	2	10.000	19.956
4	3	25.000	21.606
5	5	100.00	20.575
6	6	500.00	23.796
7	7	1000.0	23.711

Calib Factor: 21.547 RSD: 8.7%

Nitrobenzene RT: 11.27 min Window: 11.07 - 11.47 min

Level	ID	Amount/Conc'n	Response Factor
1	1	5.0000	9.5636
2	2	10.000	10.884
3	3	25.000	11.206
4	5	100.00	10.715
5	6	500.00	11.317
6	7	1000.0	11.128

Calib Factor: 10.802 RSD: 6.0%

2,4,6-Trinitrotoluene (TNT) RT: 12.73 min Window: 12.43 - 13.03 min

Level	ID	Amount/Conc'n	Response Factor
1	1	5.0000	12.085
2	2	10.000	12.604
3	3	25.000	13.714
4	5	100.00	13.700
5	6	500.00	14.412
6	7	1000.0	14.256

Calib Factor: 13.462 RSD: 6.9%

TETRYL RT: 14.08 min Window: 13.72 - 14.44 min

Level	ID	Amount/Conc'n	Response Factor
1	1	5.0000	14.086
2	2	10.000	13.145
3	3	25.000	13.717
4	5	100.00	13.365
5	6	500.00	13.058
6	7	1000.0	12.798

Calib Factor: 13.361 RSD: 3.5%

2,4-Dinitrotoluene RT: 15.41 min Window: 15.16 - 15.66 min

Level	ID	Amount/Conc'n	Response Factor
1	T1	2.5000	13.816
2	1	5.0000	11.651
3	2	10.000	14.095
4	3	25.000	13.806
5	5	100.00	14.289
6	6	500.00	15.297
7	7	1000.0	15.187

Calib Factor: 14.020 RSD: 8.6%

Quanterra / W. Sacramento
Calibration Statistics Report

Calibration ID: [AUG_SV] 109 03AUG98
Instrument ID: 1C4 PAGE 3 of 4

File: 03AUG98 Title: 8330H 2.5-1000ng/mL Phenomenex Hypersil C8 SN251486
Curve Type: Line forced thro' zero (AVG RF) Weighting: 1/X sqd
Quant Base: Height

2,6-Dinitrotoluene RT: 16.58 min Window: 16.33 - 16.83 min

Level	ID	Amount/Conc'n	Response Factor
1	1	5.0000	8.5235
2	2	10.000	7.9513
3	3	25.000	8.4391
4	5	100.00	8.0365
5	6	500.00	8.5480
6	7	1000.0	8.4452

Calib Factor: 8.3239 RSD: 3.1%

2-AM-4,6-Dinitrotoluene RT: 17.18 min Window: 16.93 - 17.43 min

Level	ID	Amount/Conc'n	Response Factor
1	1	5.0000	12.092
2	2	10.000	9.8497
3	3	25.000	10.011
4	5	100.00	9.6919
5	6	500.00	10.729
6	7	1000.0	10.555

Calib Factor: 10.488 RSD: 8.4%

4-AM-2,6-Dinitrotoluene RT: 17.84 min Window: 17.59 - 18.09 min

Level	ID	Amount/Conc'n	Response Factor
1	1	5.0000	9.8184
2	2	10.000	9.4242
3	3	25.000	8.5127
4	5	100.00	7.8534
5	6	500.00	8.3735
6	7	1000.0	8.2538

Calib Factor: 8.7060 RSD: 8.6%

2-Nitrotoluene RT: 18.69 min Window: 18.44 - 18.94 min

Level	ID	Amount/Conc'n	Response Factor
1	2	10.000	5.7161
2	3	25.000	6.1083
3	5	100.00	5.5956
4	6	500.00	4.7541
5	7	1000.0	4.5735

Calib Factor: 5.3495 RSD: 12 %

4-Nitrotoluene RT: 19.08 min Window: 18.83 - 19.33 min

Level	ID	Amount/Conc'n	Response Factor
1	2	10.000	4.1112
2	3	25.000	5.0372
3	5	100.00	4.4265
4	6	500.00	4.0640
5	7	1000.0	3.8996

Calib Factor: 4.3077 RSD: 10 %

Quanterra / W. Sacramento
Calibration Statistics Report

Calibration ID: [AUG_SV] 109 03AUG98
Instrument ID: LC4

File: 03AUG98 **Title:** 8330H 2.5-1000ng/mL Phenomenex Hypersil C8 SN251486
Curve Type: Line forced thro' zero (AVG RF) **Weighting:** 1/X sqd
Quant Base: Height

3-Nitrotoluene **RT:** 19.94 min **Window:** 19.69 - 20.19 min

Level	ID	Amount/Conc'n	Response Factor
1	2	10.000	5.4292
2	3	25.000	5.9947
3	5	100.00	6.0630
4	6	500.00	5.4304
5	7	1000.0	5.2772

Calib Factor: 5.6389 **RSD:** 6.4%

Injection Report

Acquired on 4-AUG-1998 at 11:30

Quanterra, Inc (West Sacramento)

Analyst Name : LC4UV
Lims Id : 109LC4UV9050
Comment : 75:25:0/2-22-50:00:50/2" H2O:2%THF/MeOH:Methanol (Curve)
Method Title : Var-LC5500 UV Hypersil-C8 150x4.6mm 5um SN251486 8330M
Sample Name : E070898F 25/100ng/mL STD=I
Sample Id :
Sample Type : Standard Amount=1.00000
Bottle No : 8

PEAK INFORMATION

Peak	RT mins	RT Exp	Hght uV	ppb	Peak name	Width
1	3.893	3.893	295	25.00000	HMX	7.5
2	6.551	6.551	523	25.00000	1,3,5-Trinitrobenzene	11.7
3	7.031	7.031	231	25.00000	RDX	13.3
4	8.569	8.569	1831	100.00000	2,4-Dinitrofluorobenzene(Surr)	12.8
5	9.547	9.547	540	25.00000	1,3-Dinitrobenzene	12.8
6	11.360	11.360	280	25.00000	Nitrobenzene	12.8
7	12.836	12.836	343	25.00000	2,4,6-Trinitrotoluene (TNT)	12.8
8	14.222	14.222	343	25.00000	TETRYL	12.8
9	15.573	15.573	345	25.00000	2,4-Dinitrotoluene	16.5
10	16.782	16.782	215	25.00000	2,6-Dinitrotoluene	16.5
11	17.449	17.449	263	25.00000	2-AM-4,6-Dinitrotoluene	17.6
12	18.116	18.116	233	25.00000	4-AM-2,6-Dinitrotoluene	17.1
13	18.844	18.844	176	25.00000	2-Nitrotoluene	19.7
14	19.262	19.262	142	25.00000	4-Nitrotoluene	17.1A
15	20.107	20.107	156	25.00000	3-Nitrotoluene	15.5

Totals		
Unknowns	0	0.00000
Named	5916	450.00000
Grand Total	5916	450.00000

See Reintegration 8-4-98

ANALYSIS SUMMARY

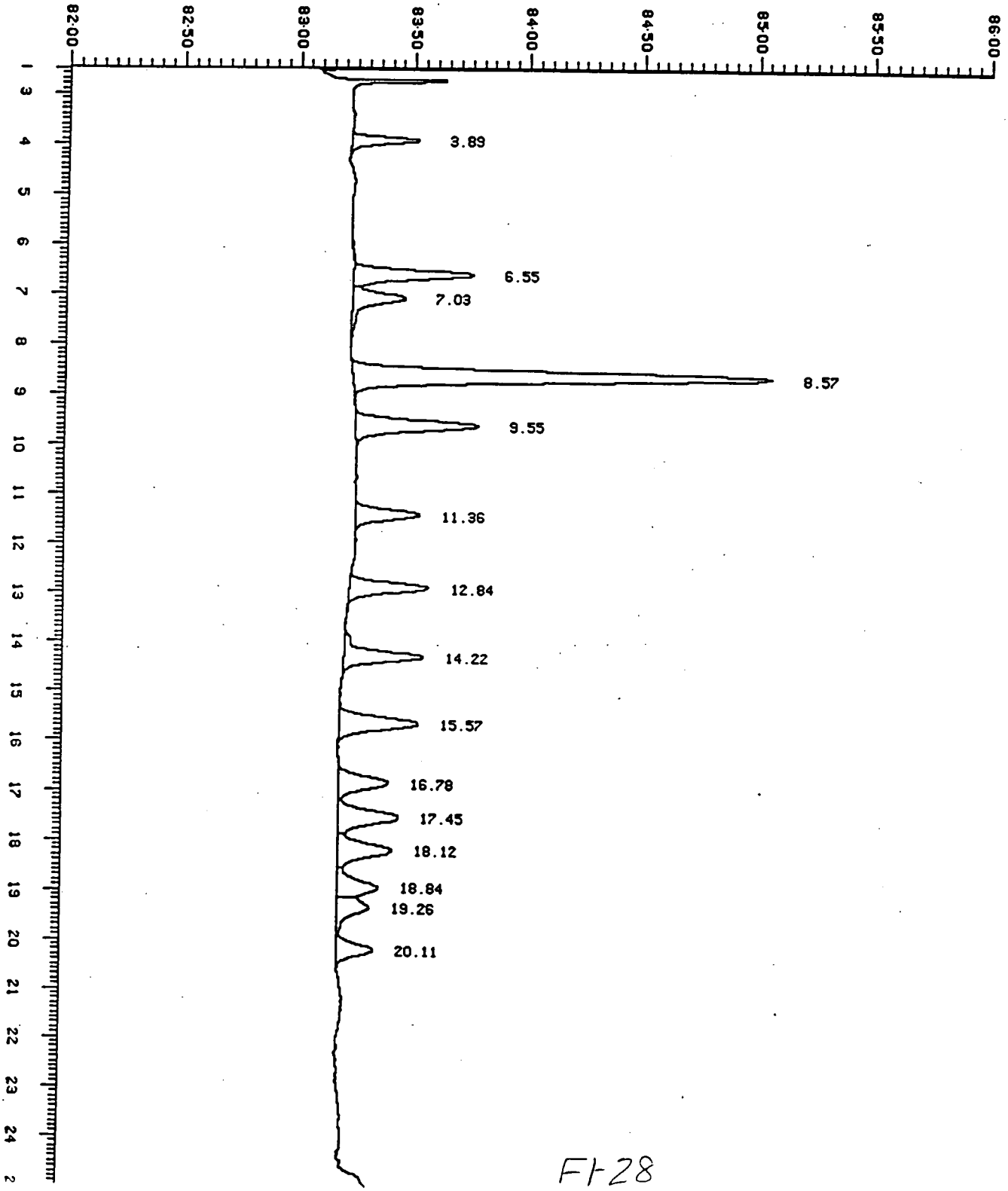
Method..... 8330 HY
Run sequence..... EXP4HY
Calibration..... 03AUG98
External standard calibration using height
Calibration last modified on 4-AUG-1998 at 16:12
Calibration initialised on 12-DEC-1997 at 21:47
Method last modified on 4-AUG-1998 at 15:54

Uncalibrated peaks use user factor (0.0000)

Quanterra, Inc (West Sacramento)

[AUG_SV] 109 4PC04AUG980849,5,1

E070898F 25/100ng/mL STD=I. Amount : 1.000.
Varian 5560/UV9050 @250nm flow=1.5 injection volume=200uL
Acquired on 4-AUG-1998 at 11:30
Reported on 4-AUG-1998 at 16:12
Box 1 (of 1)



FI-28

Injection Report

Acquired on 4-AUG-1998 at 11:30

Quanterra, Inc (West Sacramento)

Analyst Name : LC4UV
Lims Id : 109LC4UV9050
Comment : 75:25:0/2-22-50:00:50/2" H2O:2%THF/MeOH:Methanol (Curve)
Method Title : Var-LC5500 UV Hypersil-C8 150x4.6mm 5um SN251486 8330M
Sample Name : E070898F 25/100ng/mL STD=I
Sample Id :
Sample Type : Standard Amount=1.00000
Bottle No : 8

PEAK INFORMATION

Peak	RT mins	RT Exp	Hght uV	ppb	Peak name	Width
1	3.893	3.893	295	25.00000	HMX	7.5
2	6.551	6.551	523	25.00000	1,3,5-Trinitrobenzene	11.7
3	7.031	7.031	231	25.00000	RDX	13.3
4	8.569	8.569	1831	100.00000	2,4-Dinitrofluorobenzene(Surr)	12.8
5	9.547	9.547	540	25.00000	1,3-Dinitrobenzene	12.8
6	11.360	11.360	280	25.00000	Nitrobenzene	12.8
7	12.836	12.836	343	25.00000	2,4,6-Trinitrotoluene (TNT)	12.8
8	14.222	14.222	343	25.00000	TETRYL	12.8
9	15.573	15.573	345	25.00000	2,4-Dinitrotoluene	16.5
10	16.782	16.782	211	25.00000	2,6-Dinitrotoluene	16.5
11	17.449	17.449	250	25.00000	2-AM-4,6-Dinitrotoluene	17.1
12	18.116	18.116	213	25.00000	4-AM-2,6-Dinitrotoluene	16.5
13	18.844	18.844	153	25.00000	2-Nitrotoluene	17.1
14	19.262	19.262	126	25.00000	4-Nitrotoluene	16.0A
15	20.107	20.107	150	25.00000	3-Nitrotoluene	15.5

Totals		
Unknowns	0	0.00000
Named	5834	450.00000
Grand Total	5834	450.00000

RE-INTEGRATION CODES
1 [illegible] Shape
2 [illegible] Resolution
3 Peak Not Integrated
4 [illegible] Matrix Interference
5 Column Bleed
6 Instrument Noise
7 Other: (reason must be stated)
ALL RE-INTEGRATIONS MUST BE INITIALED, DATED, AND CODED

Baseline drift

ANALYSIS SUMMARY

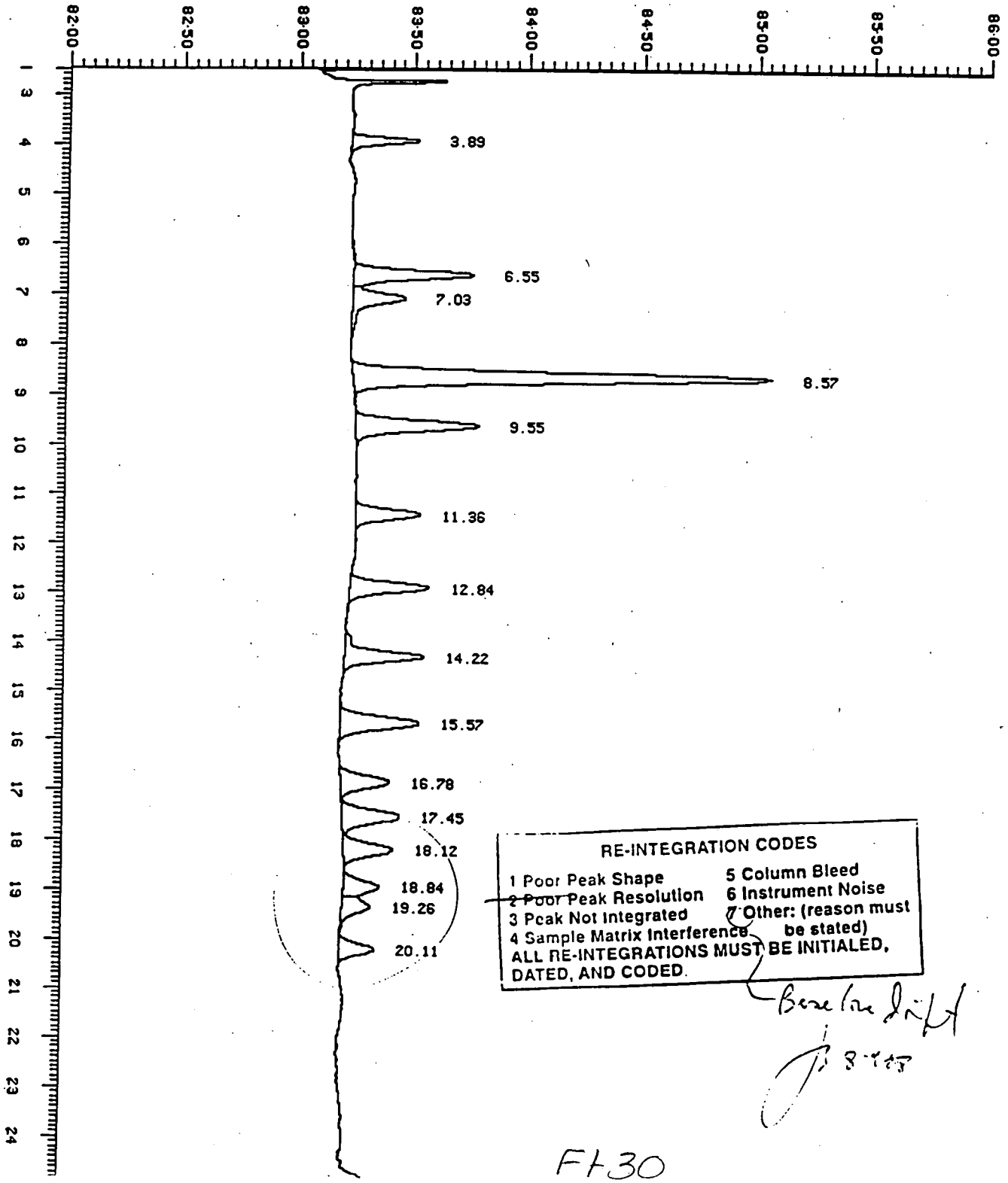
Method..... 8330_HY
Run sequence..... EXP4HY
Calibration..... 03AUG98
External standard calibration using height
Calibration last modified on 4-AUG-1998 at 16:39
Calibration initialised on 12-DEC-1997 at 21:47
Method last modified on 4-AUG-1998 at 16:38

Uncalibrated peaks use user factor (0.0000)

Quanterra, Inc (West Sacramento)

[AUG_SV] 109 4PC04AUG980849,5,1

E070898F 25/100ng/mL STD=I. Amount : 1.000.
Varian 5560/UV9050 @250nm flow=1.5 injection volume=200uL
Acquired on 4-AUG-1998 at 11:30
Reported on 4-AUG-1998 at 16:39
Box 1 (of 1)



RE-INTEGRATION CODES

1 Poor Peak Shape
2 Poor Peak Resolution
3 Peak Not Integrated
4 Sample Matrix Interference
5 Column Bleed
6 Instrument Noise
7 Other: (reason must be stated)

ALL RE-INTEGRATIONS MUST BE INITIALED, DATED, AND CODED.

Baseline drift

8-7-98

FH30

Injection Report

Acquired on 4-AUG-1998 at 12:06

Quanterra, Inc (West Sacramento)

Analyst Name : LC4UV
Lims Id : 109LC4UV9050
Comment : 75:25:0/2-22-50:00:50/2" H2O:2%THF/MeOH:Methanol (Curve)
Method Title : Var-LC5500 UV Hypersil-C8 150x4.6mm 5um SN251486 8330M
Sample Name : E070898C 2.5/5.0ng/ml STD=I
Sample Id :
Sample Type : Standard Amount=1.00000
Bottle No : 8

PEAK INFORMATION

Peak	RT mins	RT Exp	Hght uV	ppb	Peak name	Width
1	3.867	3.769	47	3.93875	HMX	8.5
2	6.542	6.493	68	3.17024	1,3,5-Trinitrobenzene	11.2
[illegible]	6.969	6.907	35	3.96672	RDX	11.2
[illegible]	8.551	8.476	95	5.28418	2,4-Dinitrofluorobenzene(Surr)	13.9
5	9.538	9.444	56	2.58955	1,3-Dinitrobenzene	12.8
6	11.333	11.271	29	2.65410	Nitrobenzene	12.8
7	12.809	12.729	43	3.22501	2,4,6-Trinitrotoluene (TNT)	12.8
8	14.196	14.078	36	2.66488	TETRYL	12.3
9	15.564	15.413	35	2.46364	2,4-Dinitrotoluene	14.9
10	16.711	16.576	16	1.97965	2,6-Dinitrotoluene	12.3
11	17.431	0.000	26	0.00000		14.9
12	18.071	17.836	34	3.85652	4-AM-2,6-Dinitrotoluene	19.7
13	20.062	19.942	13	2.34294	3-Nitrotoluene	9.1
14	24.747	0.000	60	0.00000		16.0

Totals		
Unknowns	86	0.00000
Named	507	38.13620
Grand Total	593	38.13620

ANALYSIS SUMMARY

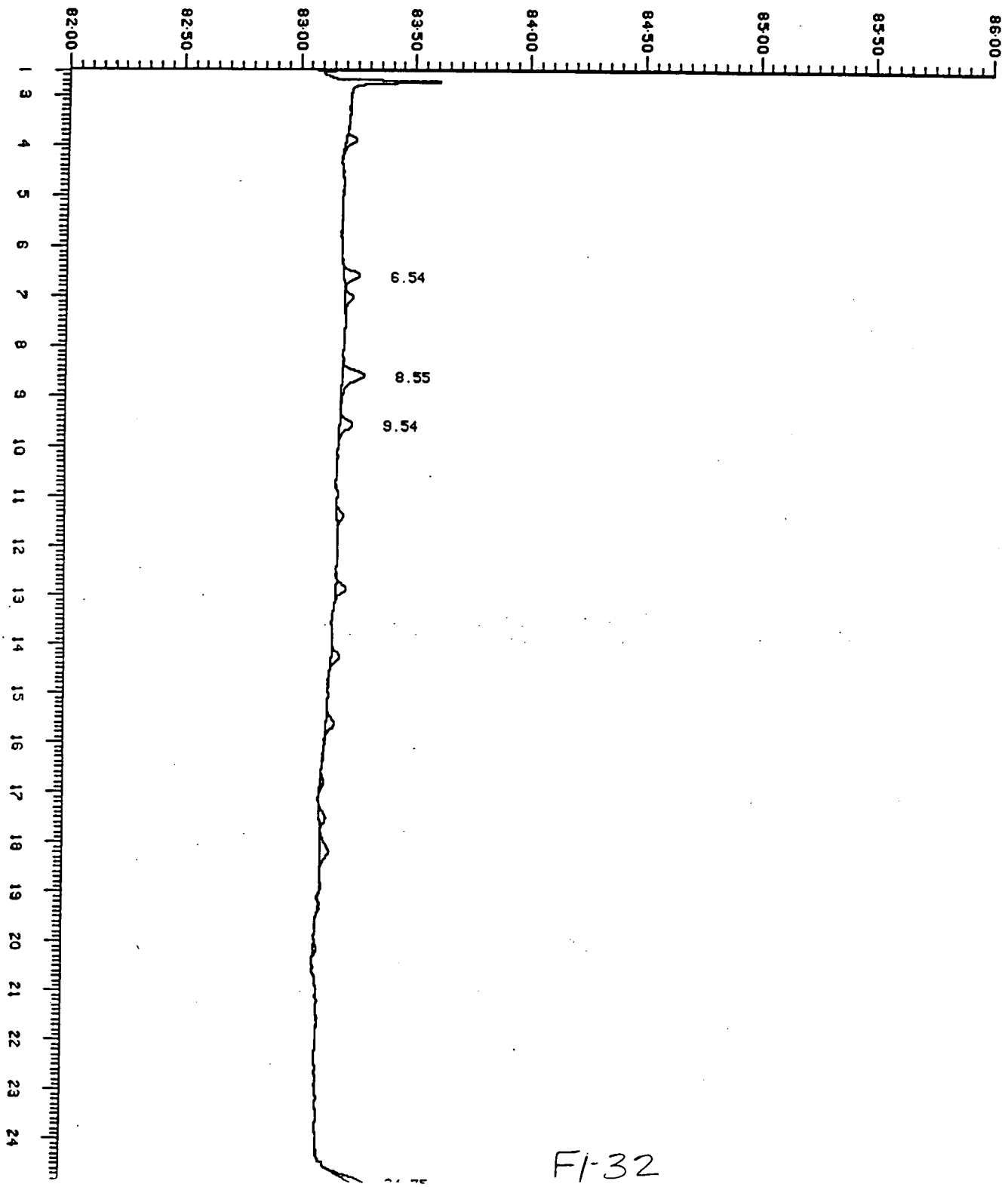
Method..... 8330 HY
Run sequence..... EXP4HY
Calibration..... 03AUG98
External standard calibration using height
Calibration last modified on 4-AUG-1998 at 15:50
Calibration initialised on 12-DEC-1997 at 21:47
Method last modified on 4-AUG-1998 at 15:54

Uncalibrated peaks use user factor (0.0000)

Quanterra, Inc (West Sacramento)

[AUG_SV] 109 4PC04AUG980849,6,1

E070898C 2.5/5.0ng/ml STD=I. Amount : 1.000.
Varian 5560/UV9050 @250nm flow=1.5 injection volume=200uL
Acquired on 4-AUG-1998 at 12:06
Reported on 4-AUG-1998 at 15:55
Box 1 (of 1)



F1-32

Injection Report

Acquired on 4-AUG-1998 at 12:43

Quanterra, Inc (West Sacramento)

Analyst Name : LC4UV
Lims Id : 109LC4UV9050
Comment : 75:25:0/2-22-50:00:50/2" H2O:2%THF/MeOH:Methanol (Curve)
Method Title : Var-LC5500 UV Hypersil-C8 150x4.6mm 5um SN251486 8330M
Sample Name : E070898D 5.0/25ng/mL STD=I
Sample Id :
Sample Type : Standard Amount=1.00000
Bottle No : 8

PEAK INFORMATION

Peak	RT mins	RT Exp	Hght uV	ppb	Peak name	Width
1	3.849	3.769	59	4.90791	HMX	8.5
2	6.542	6.493	95	4.41476	1,3,5-Trinitrobenzene	11.7
3	6.978	6.907	43	4.80423	RDX	12.3
4	8.542	8.476	422	23.45408	2,4-Dinitrofluorobenzene(Surr)	12.8
5	9.538	9.444	94	4.37793	1,3-Dinitrobenzene	13.9
6	10.596	0.000	10	0.00000		9.6
7	11.316	11.271	48	4.42673	Nitrobenzene	12.3
8	12.800	12.729	60	4.48856	2,4,6-Trinitrotoluene (TNT)	12.8
9	14.196	14.078	70	5.27121	TETRYL	12.3
10	15.538	15.413	58	4.15513	2,4-Dinitrotoluene	17.1
11	16.711	16.576	43	5.19907	2,6-Dinitrotoluene	16.0
12	17.378	17.178	60	5.85455	2-AM-4,6-Dinitrotoluene	18.1
13	18.000	17.836	49	5.63678	4-AM-2,6-Dinitrotoluene	19.2
14	18.782	18.687	29	5.30190	2-Nitrotoluene	10.7A
15	19.218	19.080	30	6.84271	4-Nitrotoluene	21.9
16	20.062	19.942	31	5.40084	3-Nitrotoluene	13.9
17	20.747	0.000	11	0.00000		8.5
18	24.720	0.000	46	0.00000		15.5

Totals		
Unknowns	67	0.00000
Named	1192	94.53639
Grand Total	1259	94.53639

ANALYSIS SUMMARY

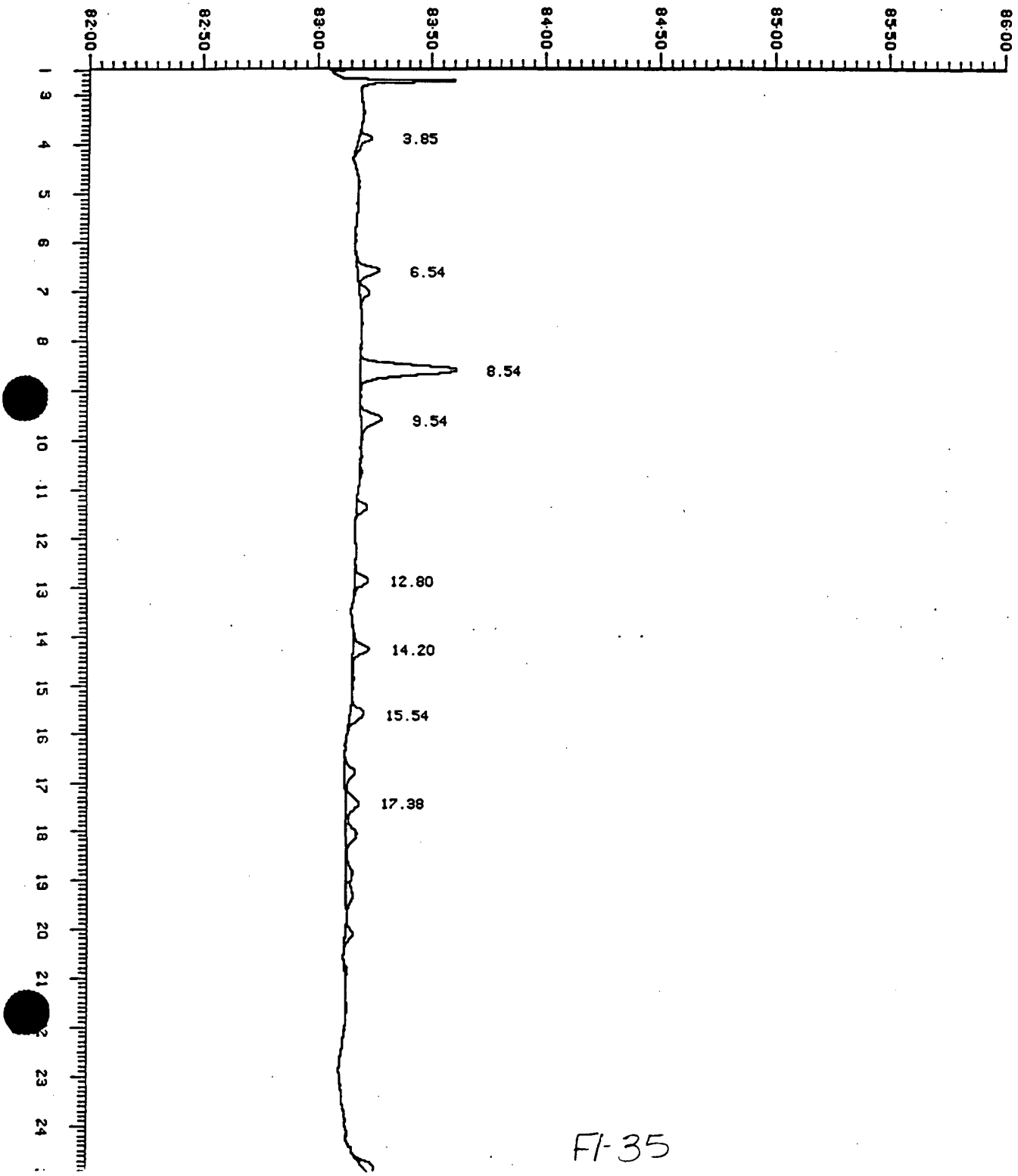
Method..... 8330_HY
Run sequence..... EXP4HY
Calibration..... 03AUG98
External standard calibration using height
Calibration last modified on 4-AUG-1998 at 15:50
Calibration initialised on 12-DEC-1997 at 21:47
Method last modified on 4-AUG-1998 at 15:54

Uncalibrated peaks use user factor (0.0000)

Quanterra, Inc (West Sacramento)

[AUG_SV] 109 4PC04AUG980849,7,1

E070898D 5.0/25ng/mL STD=I. Amount : 1.000.
Varian 5560/UV9050 @250nm flow=1.5 injection volume=200uL
Acquired on 4-AUG-1998 at 12:43
Reported on 4-AUG-1998 at 15:56
Box 1 (of 1)



F1-35

Injection Report

Acquired on 4-AUG-1998 at 13:19

Quanterra, Inc (West Sacramento)

Analyst Name : LC4UV
Lims Id : 109LC4UV9050
Comment : 75:25:0/2-22-50:00:50/2" H2O:2%THF/MeOH:Methanol (Curve)
Method Title : Var-LC5500 UV Hypersil-C8 150x4.6mm 5um SN251486 8330M
Sample Name : E070898E 10/50ng/ml STD=I
Sample Id :
Sample Type : Standard Amount=1.00000
Bottle No : 8

PEAK INFORMATION

Peak	RT mins	RT Exp	Hght uV	ppb	Peak name	Width
1	3.849	3.769	123	10.23578	HMX	8.0
2	6.524	6.493	202	9.43180	1,3,5-Trinitrobenzene	11.7
3	6.969	6.907	86	9.66013	RDX	11.7
4	8.524	8.476	914	50.75963	2,4-Dinitrofluorobenzene(Surr)	12.8
5	9.484	9.444	200	9.26140	1,3-Dinitrobenzene	13.3
6	11.307	11.271	109	10.07566	Nitrobenzene	12.3
7	12.782	12.729	126	9.36288	2,4,6-Trinitrotoluene (TNT)	12.8
8	14.151	14.078	131	9.83814	TETRYL	12.3
9	15.493	15.413	141	10.05330	2,4-Dinitrotoluene	16.0
10	16.667	16.576	80	9.70008	2,6-Dinitrotoluene	16.5
11	17.333	17.178	98	9.53755	2-AM-4,6-Dinitrotoluene	16.5
12	17.956	17.836	94	10.82101	4-AM-2,6-Dinitrotoluene	19.7
13	18.773	18.687	57	10.37658	2-Nitrotoluene	18.1
14	19.164	19.080	41	9.30460	4-Nitrotoluene	20.3
15	20.009	19.942	54	9.55937	3-Nitrotoluene	14.9
16	20.684	0.000	13	0.00000		6.9
17	24.773	0.000	61	0.00000		16.5

Totals		
Unknowns	74	0.00000
Named	2456	187.97791
Grand Total	2530	187.97791

ANALYSIS SUMMARY

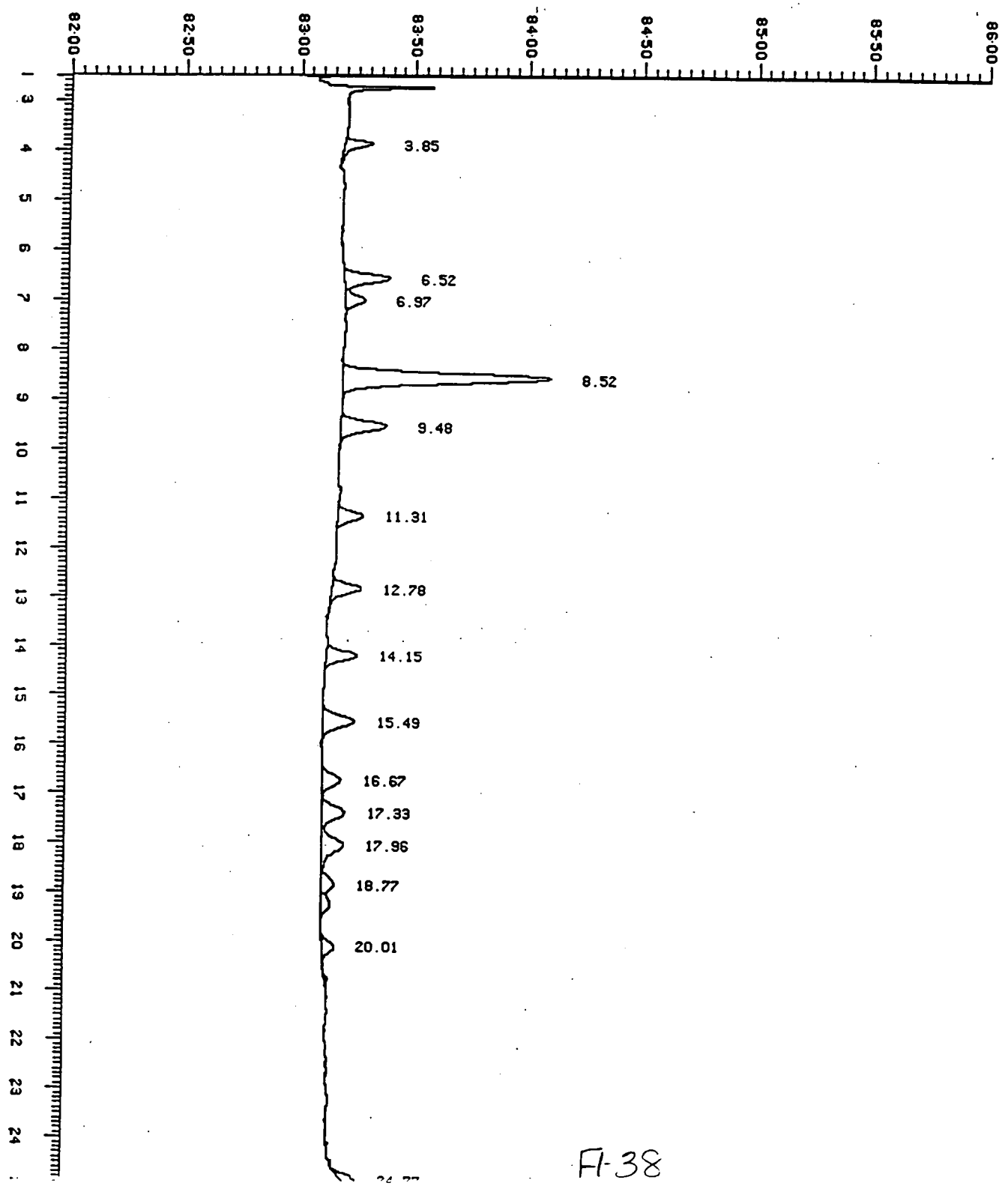
Method..... 8330 HY
Run sequence..... EXP4HY
Calibration..... 03AUG98
External standard calibration using height
Calibration last modified on 4-AUG-1998 at 15:50
Calibration initialised on 12-DEC-1997 at 21:47
Method last modified on 4-AUG-1998 at 15:54

Uncalibrated peaks use user factor (0.0000)

Quanterra, Inc (West Sacramento)

[AUG_SV] 109 4PC04AUG980849,8,1

E070898E 10/50ng/ml STD=I. Amount : 1.000.
Varian 5560/UV9050 @250nm flow=1.5 injection volume=200uL
Acquired on 4-AUG-1998 at 13:19
Reported on 4-AUG-1998 at 15:56
Box 1 (of 1)



F1-38

Injection Report

Acquired on 4-AUG-1998 at 13:55

Quanterra, Inc (West Sacramento)

Analyst Name : LC4UV
Lims Id : 109LC4UV9050
Comment : 75:25:0/2-22-50:00:50/2" H2O:2%THF/MeOH:Methanol (Curve)
Method Title : Var-LC5500 UV Hypersil-C8 150x4.6mm 5um SN251486 8330M
Sample Name : E070898G 100/150 STD=I
Sample Id :
Sample Type : Standard Amount=1.00000
Bottle No : 8

PEAK INFORMATION

Peak	RT mins	RT Exp	Hght uV	ppb	Peak name	Width
1	3.804	3.769	1131	94.28961	HMX	8.0
2	6.489	6.493	2070	96.59766	1,3,5-Trinitrobenzene	11.7
3	6.907	6.907	814	91.05035	RDX	13.3
4	8.471	8.476	2625	145.84637	2,4-Dinitrofluorobenzene(Surr)	12.8
5	9.431	9.444	2058	95.48882	1,3-Dinitrobenzene	13.3
6	11.244	11.271	1071	99.19094	Nitrobenzene	13.3
7	12.729	12.729	1370	101.77075	2,4,6-Trinitrotoluene (TNT)	12.8
8	14.098	14.078	1336	100.02416	TETRYL	13.3
9	15.413	15.413	1429	101.91945	2,4-Dinitrotoluene	16.0
10	16.569	16.576	804	98.04065	2,6-Dinitrotoluene	16.0
11	17.182	17.178	969	93.84743	2-AM-4,6-Dinitrotoluene	17.1
12	17.840	17.836	785	90.17307	4-AM-2,6-Dinitrotoluene	17.1
13	18.640	18.687	560	101.57875	2-Nitrotoluene	19.2
14	19.031	19.080	443	100.18039	4-Nitrotoluene	19.2A
15	19.911	19.942	606	106.75216	3-Nitrotoluene	16.0
16	21.591	0.000	23	0.00000		48.5
17	24.747	0.000	57	0.00000		16.5

Totals		
Unknowns	80	0.00000
Named	18071	1516.75061
Grand Total	18151	1516.75061

ANALYSIS SUMMARY

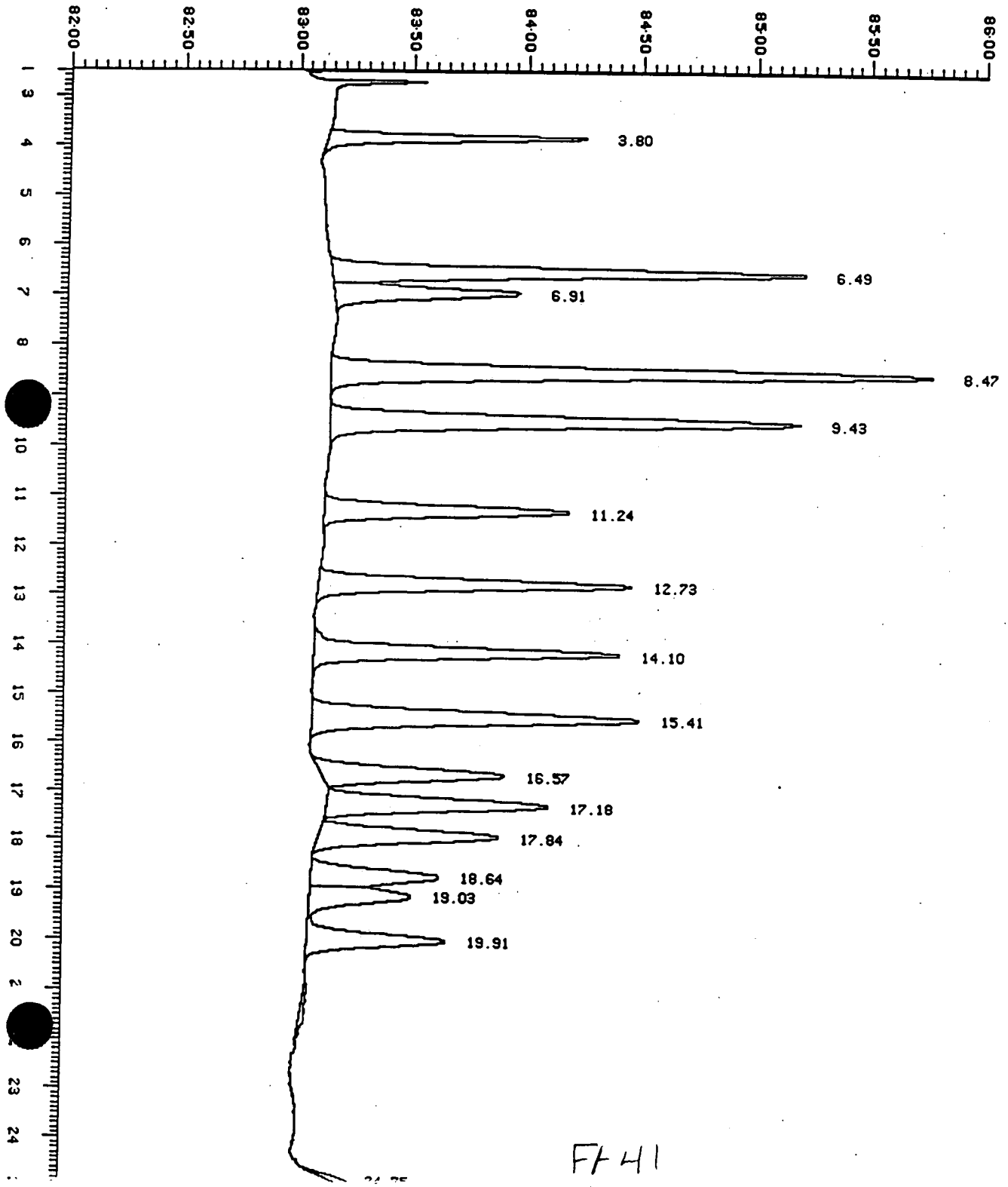
Method..... 8330_HY
Run sequence..... EXP4HY
Calibration..... 03AUG98
External standard calibration using height
Calibration last modified on 4-AUG-1998 at 15:50
Calibration initialised on 12-DEC-1997 at 21:47
Method last modified on 4-AUG-1998 at 15:54

Uncalibrated peaks use user factor (0.0000)

Quanterra, Inc (West Sacramento)

[AUG_SV] 109 4PC04AUG980849,9,1

E070898G 100/150 STD=I. Amount : 1.000.
Varian 5560/UV9050 @250nm flow=1.5 injection volume=200uL
Acquired on 4-AUG-1998 at 13:55
Reported on 4-AUG-1998 at 15:56
Box 1 (of 1)



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Injection Report

Acquired on 4-AUG-1998 at 14:31

Quanterra, Inc (West Sacramento)

Analyst Name : LC4UV
Lims Id : 109LC4UV9050
Comment : 75:25:0/2-22-50:00:50/2" H2O:2%THF/MeOH:Methanol (Curve)
Method Title : Var-LC5500 UV Hypersil-C8 150x4.6mm 5um SN251486 8330M
Sample Name : E070898H 500 ng/mL 8330 Mix STD=I
Sample Id :
Sample Type : Standard Amount=1.00000
Bottle No : 8

PEAK INFORMATION

Peak	RT mins	RT Exp	Hght uV	ppb	Peak name	Width
1	3.467	0.000	14	0.00000		6.9
2	3.769	3.769	6251	521.19122	HMX	7.5
3	6.507	6.493	11941	557.29205	1,3,5-Trinitrobenzene	10.1
4	6.924	6.907	4756	531.95862	RDX	11.2
5	8.480	8.476	18	1.00468	2,4-Dinitrofluorobenzene(Surr)	10.7
6	9.467	9.444	11898	552.18610	1,3-Dinitrobenzene	12.3
7	11.298	11.271	5658	523.81244	Nitrobenzene	12.3
8	12.747	12.729	7206	535.29248	2,4,6-Trinitrotoluene (TNT)	12.8
9	13.556	0.000	40	0.00000		12.3
10	14.089	14.078	6529	488.65332	TETRYL	13.3
11	15.431	15.413	7648	545.52191	2,4-Dinitrotoluene	16.0
12	16.596	16.576	4274	521.40314	2,6-Dinitrotoluene	16.5
13	17.200	17.178	5364	519.43842	2-AM-4,6-Dinitrotoluene	17.6
14	17.858	17.836	4187	480.72772	4-AM-2,6-Dinitrotoluene	17.6
15	18.720	18.687	2377	431.51663	2-Nitrotoluene	18.1
16	19.102	19.080	2032	459.88449	4-Nitrotoluene	18.1A
17	19.964	19.942	2715	478.07458	3-Nitrotoluene	16.0
18	21.520	0.000	10	0.00000		13.9
19	24.036	0.000	12	0.00000		9.1
20	24.373	0.000	18	0.00000		4.3A
21	24.889	0.000	65	0.00000		4.8

Totals		
Unknowns	160	0.00000
Named	82855	7147.95752
Grand Total	83014	7147.95752

ANALYSIS SUMMARY

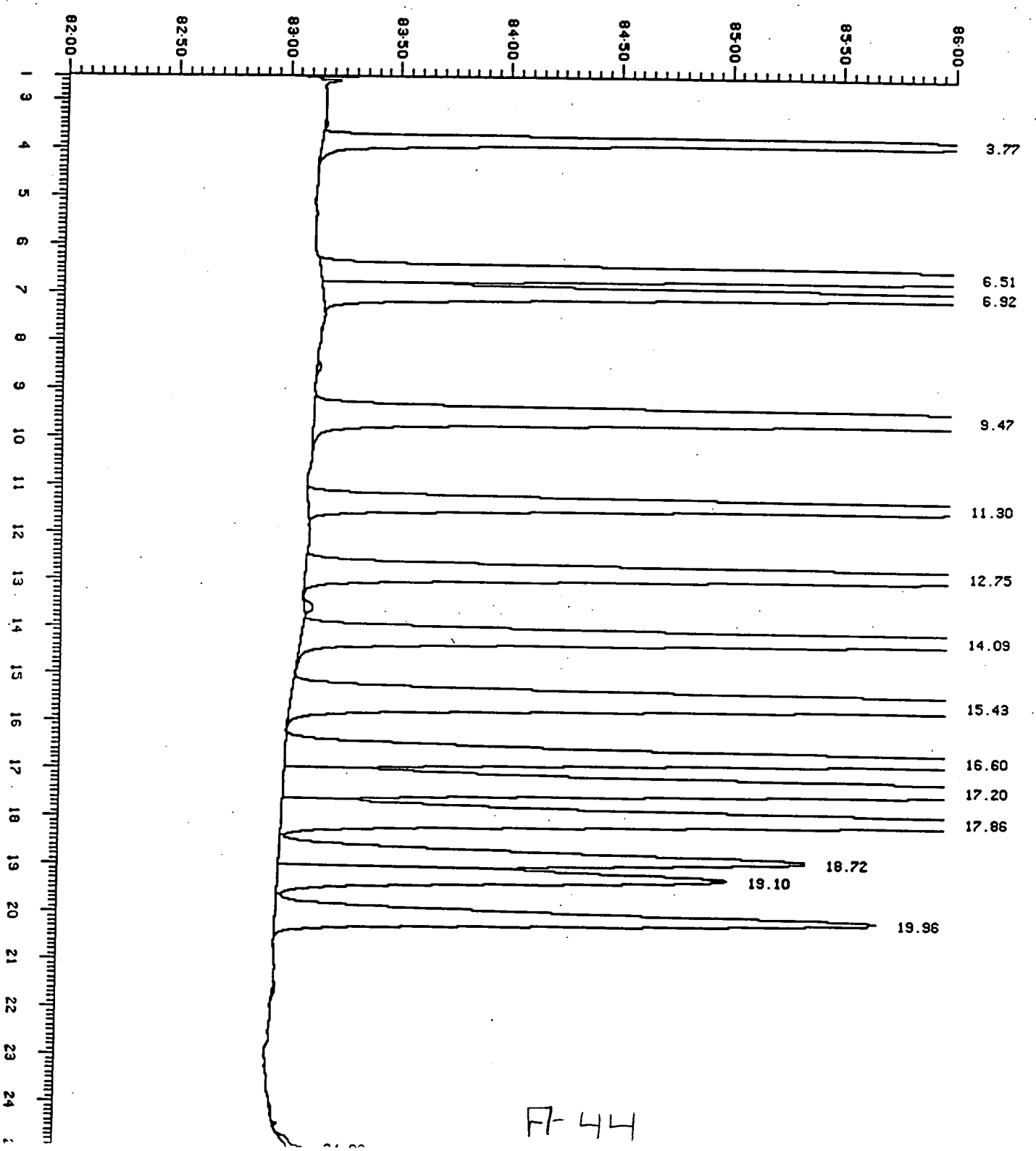
Method..... 8330_HY
Run sequence..... EXP4HY
Calibration..... 03AUG98
External standard calibration using height
Calibration last modified on 4-AUG-1998 at 15:50
Calibration initialised on 12-DEC-1997 at 21:47
Method last modified on 4-AUG-1998 at 15:54

Uncalibrated peaks use user factor (0.0000)

Quanterra, Inc (West Sacramento)

[AUG_SV] 109 4PC04AUG980849,10,1

E070898H 500 ng/mL 8330 Mix STD=I. Amount : 1.000.
Varian 5560/UV9050 @250nm flow=1.5 injection volume=200uL
Acquired on 4-AUG-1998 at 14:31
Reported on 4-AUG-1998 at 15:56
Box 1 (of 1)



A-44

Injection Report

Acquired on 4-AUG-1998 at 15:08

Quanterra, Inc (West Sacramento)

Analyst Name : LC4UV
Lims Id : 109LC4UV9050
Comment : 75:25:0/2-22-50:00:50/2" H2O:2%THF/MeOH:Methanol (Curve)
Method Title : Var-LC5500 UV Hypersil-C8 150x4.6mm 5um SN251486 8330M
Sample Name : E070898I 1000 ng/mL 8330 Mix STD=I
Sample Id :
Sample Type : Standard Amount=1.00000
Bottle No : 8

PEAK INFORMATION

Peak	RT mins	RT Exp	Hght uV	ppb	Peak name	Width
1	3.369	0.000	11	0.00000		5.9
2	3.751	3.769	12312	1026.58838	HMX	7.5
3	5.307	0.000	26	0.00000		8.5
4	6.489	6.493	23916	1116.17505	1,3,5-Trinitrobenzene	10.7
5	6.898	6.907	9538	1066.84058	RDX	11.2
6	9.440	9.444	23711	1100.43970	1,3-Dinitrobenzene	11.7
7	11.271	11.271	11128	1030.19116	Nitrobenzene	11.7
8	12.720	12.729	14256	1059.00940	2,4,6-Trinitrotoluene (TNT)	12.8
9	13.511	0.000	92	0.00000		12.8
10	14.062	14.078	12798	957.81213	TETRYL	13.3
11	15.404	15.413	15187	1083.23999	2,4-Dinitrotoluene	16.0
12	16.569	16.576	8445	1030.26306	2,6-Dinitrotoluene	17.1
13	17.164	17.178	10555	1022.03387	2-AM-4,6-Dinitrotoluene	17.6
14	17.822	17.836	8254	947.71387	4-AM-2,6-Dinitrotoluene	18.1
15	18.693	18.687	4573	830.23987	2-Nitrotoluene	18.7
16	19.093	19.080	3900	882.55566	4-Nitrotoluene	17.1A
17	19.947	19.942	5277	929.16205	3-Nitrotoluene	15.5
18	20.916	0.000	16	0.00000		14.4
19	21.538	0.000	20	0.00000		16.0
20	24.871	0.000	115	0.00000		4.3

Totals		
Unknowns	279	0.00000
Named	163850	14082.26563
Grand Total	164129	14082.26563

ANALYSIS SUMMARY

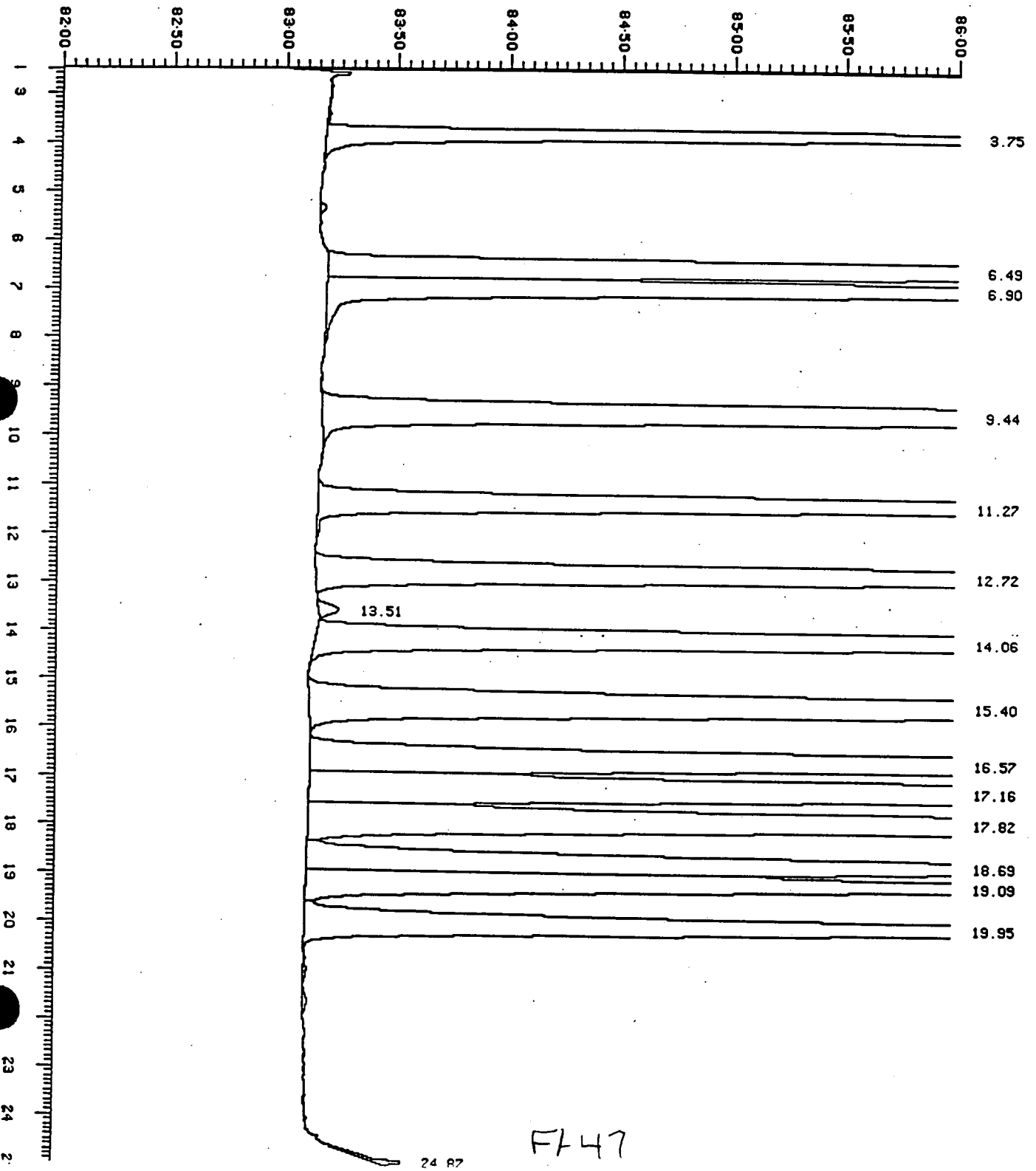
Method..... 8330_HY
Run sequence..... EXP4HY
Calibration..... 03AUG98
External standard calibration using height
Calibration last modified on 4-AUG-1998 at 15:50
Calibration initialised on 12-DEC-1997 at 21:47
Method last modified on 4-AUG-1998 at 15:54

Uncalibrated peaks use user factor (0.0000)

Quanterra, Inc (West Sacramento)

[AUG_SV] 109 4PC04AUG980849,11,1

E070898I 1000 ng/mL 8330 Mix STD=I. Amount : 1.000.
Varian 5560/UV9050 @250nm flow=1.5 injection volume=200uL
Acquired on 4-AUG-1998 at 15:08
Reported on 4-AUG-1998 at 15:56
Box 1 (of 1)



F147

Injection Report

Acquired on 4-AUG-1998 at 15:44

Quanterra, Inc (West Sacramento)

Analyst Name : LC4UV
Lims Id : 109LC4UV9050
Comment : 75:25:0/2-22-50:00:50/2" H2O:2%THF/MeOH:Methanol (Curve)
Method Title : Var-LC5500 UV Hypersil-C8 150x4.6mm 5um SN251486 8330M
Sample Name : Water blank
Sample Id :
Sample Type : Sample Amount=1.00000
Bottle No : 8

PEAK INFORMATION

Peak	RT mins	RT Exp	Hght uV	ppb	Peak name	Width
1	3.467	0.000	10	0.00000		4.3
2	20.942	0.000	13	0.00000		14.4
3	24.782	0.000	54	0.00000		16.0

Totals		
Unknowns	78	0.00000
Named	0	0.00000
Grand Total	78	0.00000

ANALYSIS SUMMARY

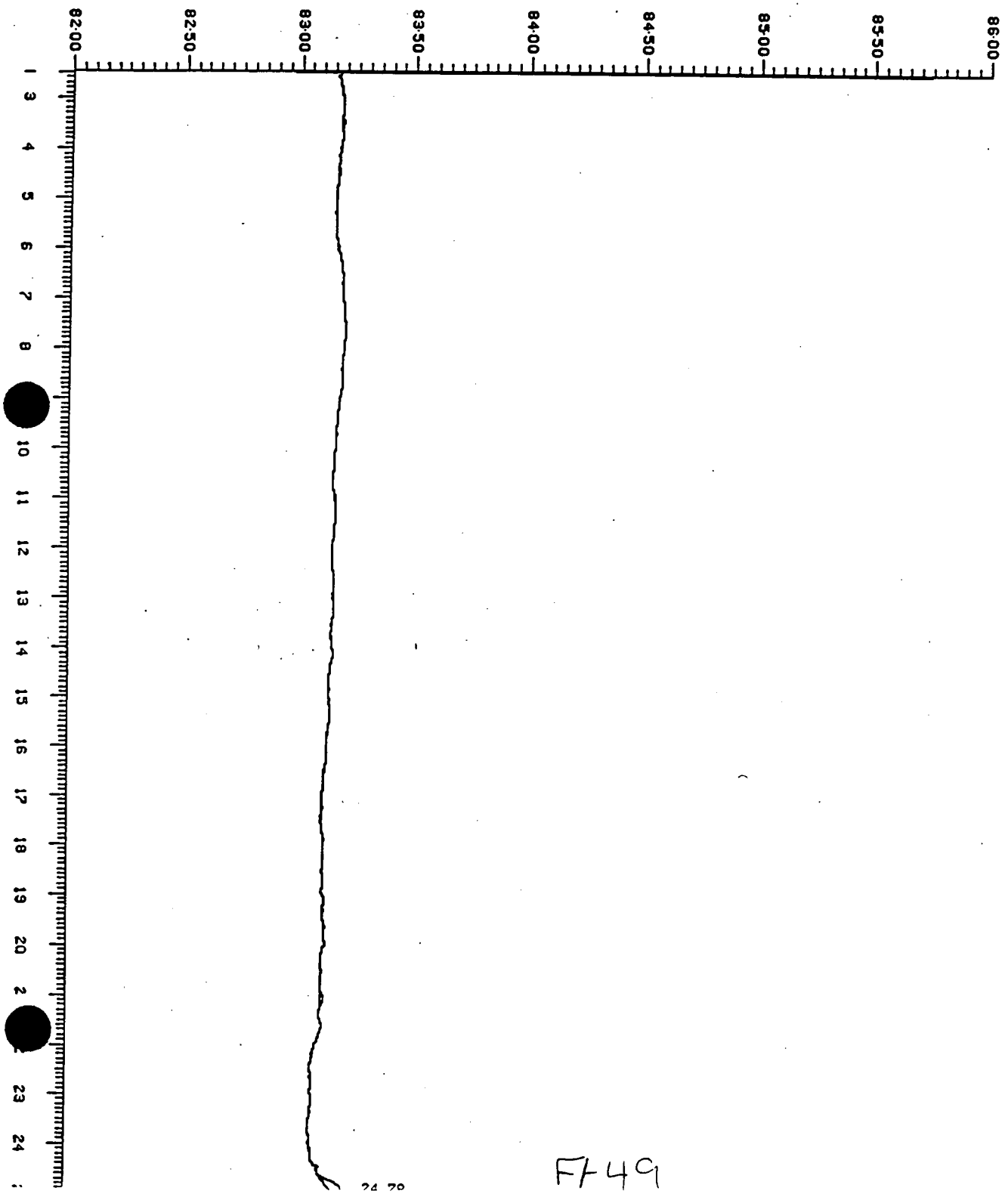
Method..... 8330_HY
Run sequence..... EXP4HY
Calibration..... 03AUG98
External standard calibration using height
Calibration last modified on 4-AUG-1998 at 16:07
Calibration initialised on 12-DEC-1997 at 21:47
Method last modified on 4-AUG-1998 at 15:54

Uncalibrated peaks use user factor (0.0000)

Quanterra, Inc (West Sacramento)

[AUG_SV] 109 4PC04AUG980849,12,1

Water blank. Amount : 1.000.
Varian 5560/UV9050 @250nm flow=1.5 injection volume=200uL
Acquired on 4-AUG-1998 at 15:44
Reported on 4-AUG-1998 at 16:09
Box 1 (of 1)



F-49

Continuing Calibration

Injection Report

Acquired on 4-AUG-1998 at 16:47

Quanterra, Inc (West Sacramento)

Analyst Name : LC4UV
Lims Id : 109LC4UV9050
Comment : 75:25:0/2-22-50:00:50/2"- H2O:2%THF/MeOH:Methanol (Analysis)
Method Title : Var-LC5500 UV Hypersil-C8 150x4.6mm 5um SN251486 8330M
Sample Name : E070898F 25/100 8330MIX CV
Sample Id :
Sample Type : Standard Amount=1.00000
Bottle No : 8

PEAK INFORMATION

Peak	RT mins	RT Exp	Hght uV	ppb	Peak name	Width
1	3.804	3.769	290	24.19226	HMX	8.0
2	6.498	6.493	510	23.80516	1,3,5-Trinitrobenzene	11.2
3	6.889	6.907	205	22.94697	RDX	13.3
4	8.480	8.476	1796	99.80174	2,4-Dinitrofluorobenzene(Surr)	13.3
5	9.422	9.444	514	23.86339	1,3-Dinitrobenzene	13.3
6	11.244	11.271	277	25.63266	Nitrobenzene	12.8
7	12.738	12.729	328	24.36662	2,4,6-Trinitrotoluene (TNT)	12.8
8	14.062	14.078	331	24.80697	TETRYL	13.3
9	15.387	15.413	349	24.86063	2,4-Dinitrotoluene	16.5
10	16.551	16.576	190	22.81024	2,6-Dinitrotoluene	17.1
11	17.147	17.178	234	22.29460	2-AM-4,6-Dinitrotoluene	17.6
12	17.822	17.836	190	21.85193	4-AM-2,6-Dinitrotoluene	18.1
13	18.667	18.687	141	26.30777	2-Nitrotoluene	18.1
14	19.022	19.080	110	25.64306	4-Nitrotoluene	20.3A
15	19.902	19.942	155	27.52489	3-Nitrotoluene	16.0
16	24.382	0.000	47	0.00000		10.1

Totals		
Unknowns	47	0.00000
Named	5621	440.70889
Grand Total	5668	440.70889

ANALYSIS SUMMARY

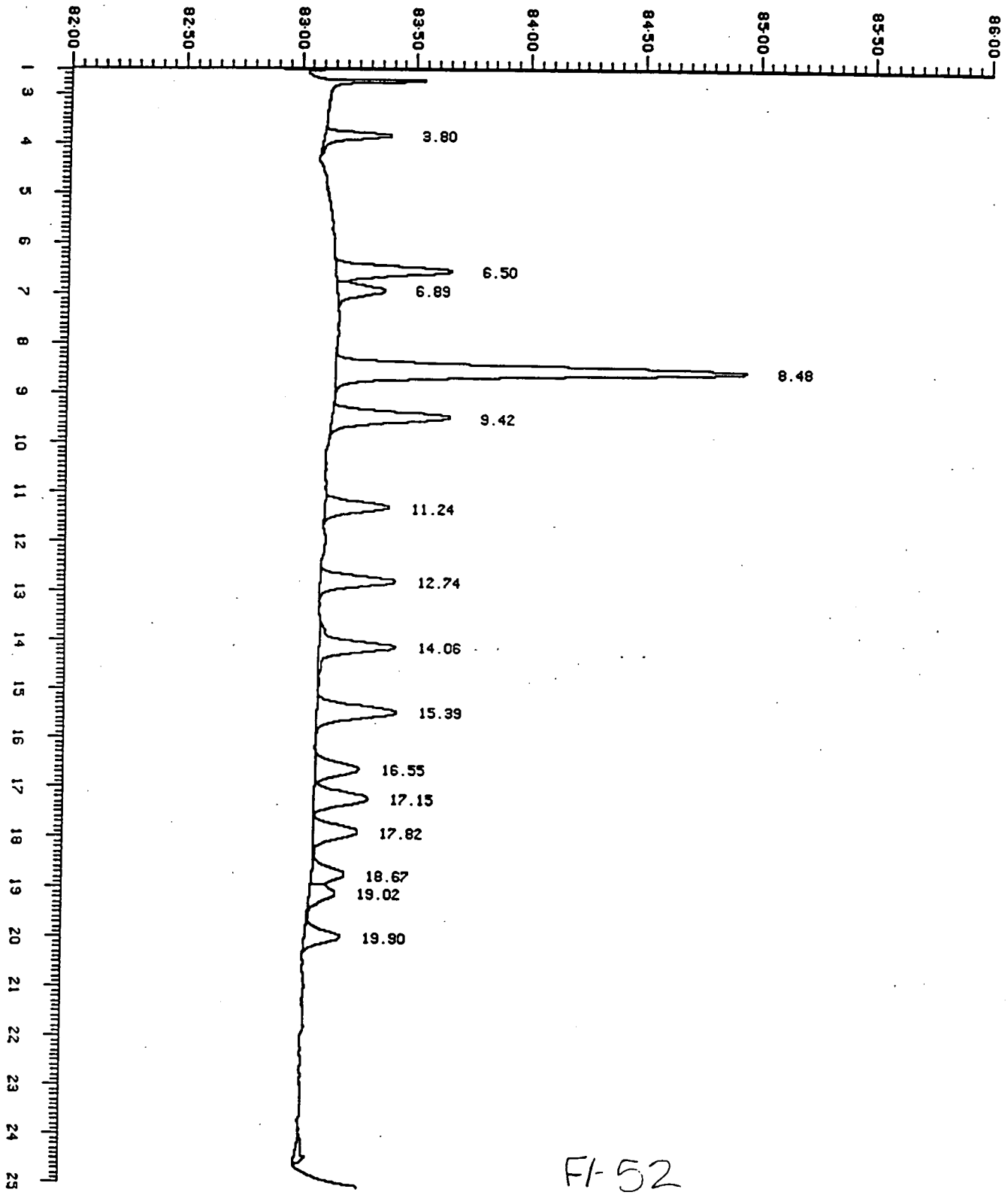
Method..... 8330 HY
Run sequence..... EXP4HY1
Calibration..... 03AUG98
External standard calibration using height
Calibration last modified on 4-AUG-1998 at 17:12
Calibration initialised on 12-DEC-1997 at 21:47
Method last modified on 4-AUG-1998 at 16:40

Uncalibrated peaks use user factor (0.0000)

Quanterra, Inc (West Sacramento)

[AUG_SV] 109 4H_04AUG981450,1,1

E070898F 25/100 8330MIX CV. Amount : 1.000.
Varian 5560/UV9050 @250nm flow=1.5 injection volume=200uL
Acquired on 4-AUG-1998 at 16:47
Reported on 4-AUG-1998 at 17:12
Box 1 (of 1)



F1-52

Quanterra / W. Sacramento **Analysis ID:** [AUG_SV] 109 4H_04AUG981450, 1
Chromatography Summary **Acquired:** HPLC #4 4-AUG-1998 at 16:47

Sample: E070898F 25/100 8330MIX CV **Mult:** 1.00 **Dilf:** 1.00 **Div:** 1.000
Method File: 8330_HY 75:25:0/2-22-50:00:50/2"- H2O:2%THF/MeOH:Methanol (Analysis)
Calibration: 03AUG98 8330H 2.5-1000ng/mL Phenomenex Hypersil C8 SN251486

Calibration Check

Channel 109 Var-LC5500 UV
Hypersil-C8
150x4.6mm 5um SN251486 8330M

	Peak Name	RT	True	Calc.	%D	-Limits-
1	HMX	3.804	25.00	24.1923	-3.2	(15)
2	1,3,5-Trinitrobenz	6.498	25.00	23.8052	-4.8	(15)
3	RDX	6.889	25.00	22.9470	-8.2	(15)
4	2,4-Dinitrofluorob	8.480	100.0	99.802	-0.20	(15)
5	1,3-Dinitrobenzene	9.422	25.00	23.8634	-4.5	(15)
6	Nitrobenzene	11.244	25.00	25.6327	2.5	(15)
7	2,4,6-Trinitrotolu	12.738	25.00	24.3666	-2.5	(15)
8	TETRYL	14.062	25.00	24.8070	-0.77	(15)
9	2,4-Dinitrotoluene	15.387	25.00	24.8606	-0.56	(15)
10	2,6-Dinitrotoluene	16.551	25.00	22.8102	-8.8	(15)
11	2-AM-4,6-Dinitroto	17.147	25.00	22.2946	-11	(15)
12	4-AM-2,6-Dinitroto	17.822	25.00	21.8519	-13	(15)
13	2-Nitrotoluene	18.667	25.00	26.3078	5.2	(15)
14	4-Nitrotoluene	19.022	25.00	25.6431	2.6	(15)
15	3-Nitrotoluene	19.902	25.00	27.5249	10	(15)

Notes:
No limits found 15% used

Average %D = 5.2

Q/WS SUM 3.29A 8-5-98 **Reviewer:** G **Date:** 8/5/98
Printed: 08/04/98 17:13:03

F1-53

Injection Report

Acquired on 5-AUG-1998 at 00:18

Quanterra, Inc (West Sacramento)

Analyst Name : LC4UV
Lims Id : 109LC4UV9050
Comment : 75:25:0/2-22-50:00:50/2"- H2O:2%THF/MeOH:Methanol (Analysis)
Method Title : Var-LC5500 UV Hypersil-C8 150x4.6mm 5um SN251486 8330M
Sample Name : E070898F 25/100 8330MIX CV
Sample Id :
Sample Type : Standard Amount=1.00000
Bottle No : 8

PEAK INFORMATION

Peak	RT mins	RT Exp	Hght uV	ppb	Peak name	Width
1	3.893	3.890	293	24.43827	HMX	7.5
2	6.551	6.550	503	23.45500	1,3,5-Trinitrobenzene	11.7
3	7.076	7.080	215	24.01365	RDX	13.3
4	8.596	8.600	1752	97.34324	2,4-Dinitrofluorobenzene(Surr)	13.3
5	9.591	9.590	521	24.15938	1,3-Dinitrobenzene	13.3
6	11.396	11.400	269	24.93740	Nitrobenzene	13.3
7	12.898	12.900	323	23.97477	2,4,6-Trinitrotoluene (TNT)	12.8
8	14.338	14.340	340	25.44824	TETRYL	13.3
9	15.680	15.680	342	24.39936	2,4-Dinitrotoluene	16.5
10	16.889	16.890	217	26.12652	2,6-Dinitrotoluene	18.1
11	17.618	17.620	277	26.40487	2-AM-4,6-Dinitrotoluene	17.6
12	18.276	18.280	212	24.36284	4-AM-2,6-Dinitrotoluene	17.6
13	18.987	18.990	151	28.17546	2-Nitrotoluene	17.1A
14	19.360	19.360	129	29.93246	4-Nitrotoluene	20.3A
15	20.187	20.190	166	29.48434	3-Nitrotoluene	16.0
16	22.098	0.000	13	0.00000		14.9
17	22.311	0.000	19	0.00000		12.8
18	24.373	0.000	69	0.00000		3.2
19	24.702	0.000	59	0.00000		18.1

see Reintegration

JRS 5-5-97

Totals		
Unknowns	160	0.00000
Named	5710	456.65582
Grand Total	5870	456.65582

ANALYSIS SUMMARY

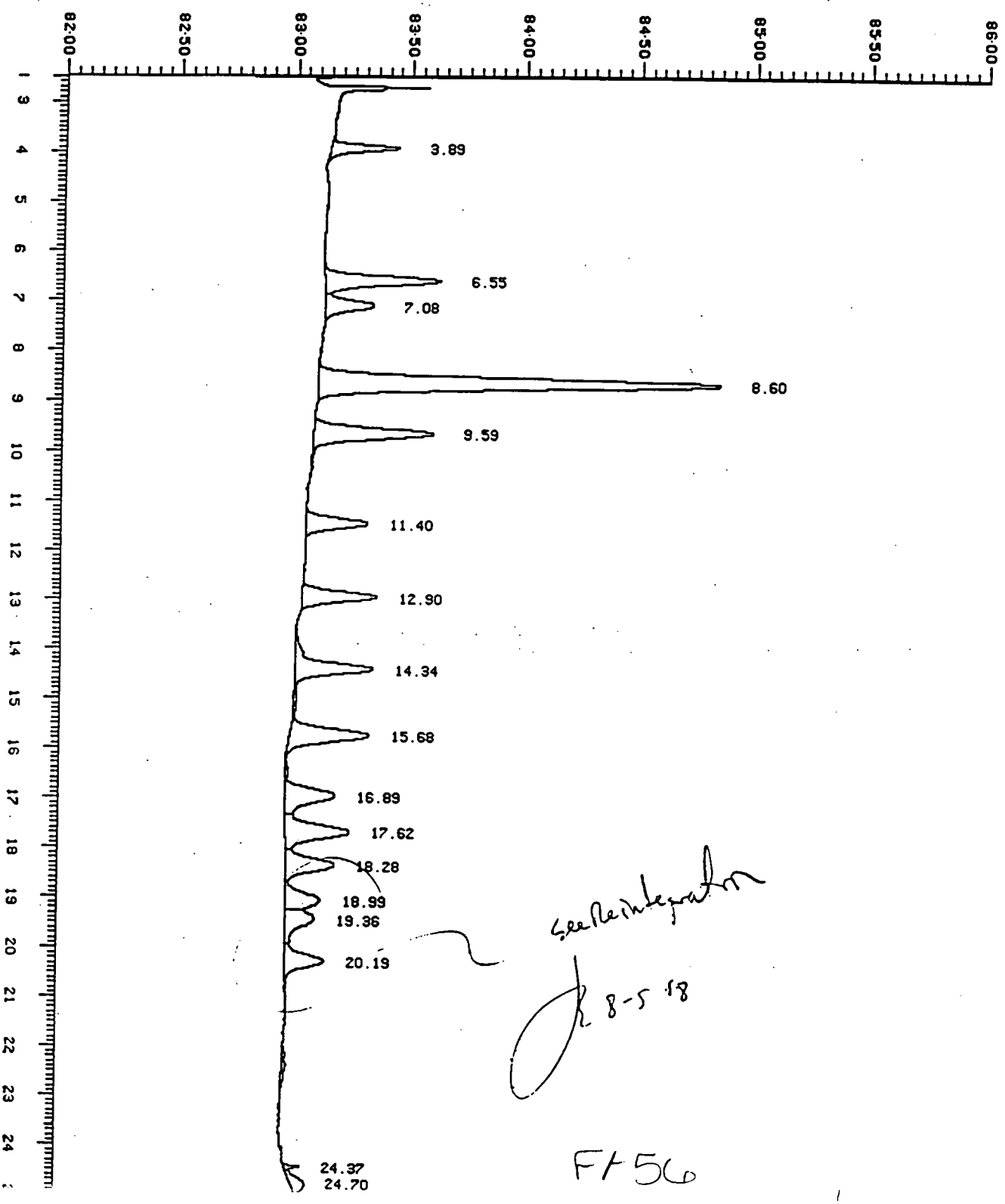
Method..... 8330_HY
Run sequence..... EXP4HY1
Calibration..... 03AUG98
External standard calibration using height
Calibration last modified on 5-AUG-1998 at 14:13
Calibration initialised on 12-DEC-1997 at 21:47
Method last modified on 4-AUG-1998 at 16:40

Uncalibrated peaks use user factor (0.0000)

Quanterra, Inc (West Sacramento)

[AUG_SV] 109 4H_04AUG981450,12,1

E070898F 25/100 8330MIX CV. Amount : 1.000.
Varian 5560/UV9050 @250nm flow=1.5 injection volume=200uL
Acquired on 5-AUG-1998 at 00:18
Reported on 5-AUG-1998 at 14:13
Box 1 (of 1)



See Reintegration
8-5-98

F/56

Quanterra / W. Sacramento
Chromatography Summary

Analysis ID: [AUG_SV] 109 4H_04AUG981450,12
Acquired: HPLC #4 5-AUG-1998 at 00:18

Sample: E070898F 25/100 8330MIX CV **Mult:** 1.00 **Dilf:** 1.00 **Div:** 1.000
Method File: 8330_HY 75:25:0/2-22-50:00:50/2"- H2O:2%THF/MeOH:Methanol (Analysis)
Calibration: 03AUG98 8330H 2.5-1000ng/mL Phenomenex Hypersil C8 SN251486

Calibration Check

Channel 109 Var-LC5500 UV
Hypersil-C8
150x4.6mm 5um SN251486 8330M

	Peak Name	RT	True	Calc.	%D	-Limits-
1	HMX	3.893	25.00	24.4383	-2.2	(15)
2	1,3,5-Trinitrobenz	6.551	25.00	23.4550	-6.2	(15)
3	RDX	7.076	25.00	24.0136	-3.9	(15)
4	2,4-Dinitrofluorob	8.596	100.0	97.3432	-2.7	(15)
5	1,3-Dinitrobenzene	9.591	25.00	24.1594	-3.4	(15)
6	Nitrobenzene	11.396	25.00	24.9374	-0.25	(15)
7	2,4,6-Trinitrotolu	12.898	25.00	23.9748	-4.1	(15)
8	TETRYL	14.338	25.00	25.4482	1.8	(15)
9	2,4-Dinitrotoluene	15.680	25.00	24.3994	-2.4	(15)
10	2,6-Dinitrotoluene	16.889	25.00	26.1265	4.5	(15)
11	2-AM-4,6-Dinitroto	17.618	25.00	26.4049	5.6	(15)
12	4-AM-2,6-Dinitroto	18.276	25.00	24.3628	-2.5	(15)
13	2-Nitrotoluene	18.987	25.00	28.1755	13	(15)
14	4-Nitrotoluene	19.360	25.00	29.9325	20 *	(15)
15	3-Nitrotoluene	20.187	25.00	29.4843	18 *	(15)

see file integration
5-5-98

Notes:
No limits found 15% used

Average %D = 6.0

Q/WS SUM 3.29A 8-5-98

Reviewer: _____ Date: _____
Printed: 08/05/98 14:13:16

Ft 57

Injection Report

Acquired on 5-AUG-1998 at 00:18

Quanterra, Inc (West Sacramento)

Analyst Name : LC4UV
Lims Id : 109LC4UV9050
Comment : 75:25:0/2-22-50:00:50/2"- H2O:2%THF/MeOH:Methanol (Analysis)
Method Title : Var-LC5500 UV Hypersil-C8 150x4.6mm 5um SN251486 8330M
Sample Name : E070898F 25/100 8330MIX CV
Sample Id :
Sample Type : Standard Amount=1.00000
Bottle No : 8

PEAK INFORMATION

Peak	RT mins	RT Exp	Hght uV	ppb	Peak name	Width
1	3.893	3.890	293	24.43827	HMX	7.5
2	6.551	6.550	503	23.45500	1,3,5-Trinitrobenzene	11.7
3	7.076	7.080	215	24.01365	RDX	13.3
4	8.596	8.600	1752	97.34324	2,4-Dinitrofluorobenzene(Surr)	13.3
5	9.591	9.590	521	24.15938	1,3-Dinitrobenzene	13.3
6	11.396	11.400	269	24.93740	Nitrobenzene	13.3
7	12.898	12.900	323	23.97477	2,4,6-Trinitrotoluene (TNT)	12.8
8	14.338	14.340	340	25.44824	TETRYL	13.3
9	15.680	15.680	342	24.39936	2,4-Dinitrotoluene	16.5
10	16.889	16.890	214	25.76401	2,6-Dinitrotoluene	18.1
11	17.618	17.620	271	25.80247	2-AM-4,6-Dinitrotoluene	17.6
12	18.276	18.280	203	23.29481	4-AM-2,6-Dinitrotoluene	17.1
13	18.987	18.990	138	25.83562	2-Nitrotoluene	19.2
14	19.360	19.360	115	26.63412	4-Nitrotoluene	18.1A
15	20.196	20.190	155	27.40993	3-Nitrotoluene	15.5
16	22.098	0.000	13	0.00000		14.9
17	22.311	0.000	19	0.00000		12.8
18	24.373	0.000	69	0.00000		3.2
19	24.702	0.000	59	0.00000		18.1

Totals		
Unknowns	160	0.00000
Named	5653	446.91028
Grand Total	5813	446.91028

RE-INTEGRATION CODES

1 Poor Peak Shape
2 Poor Peak Resolution
3 Peak Not Integrated
4 Sample Matrix Interference
5 Column Bleed
6 Instrument Noise
7 Other: (reason must be stated)

ALL RE-INTEGRATIONS MUST BE INITIALED, DATED, AND CODED.

Baseline drift
8-5-98

FI-58

ANALYSIS SUMMARY

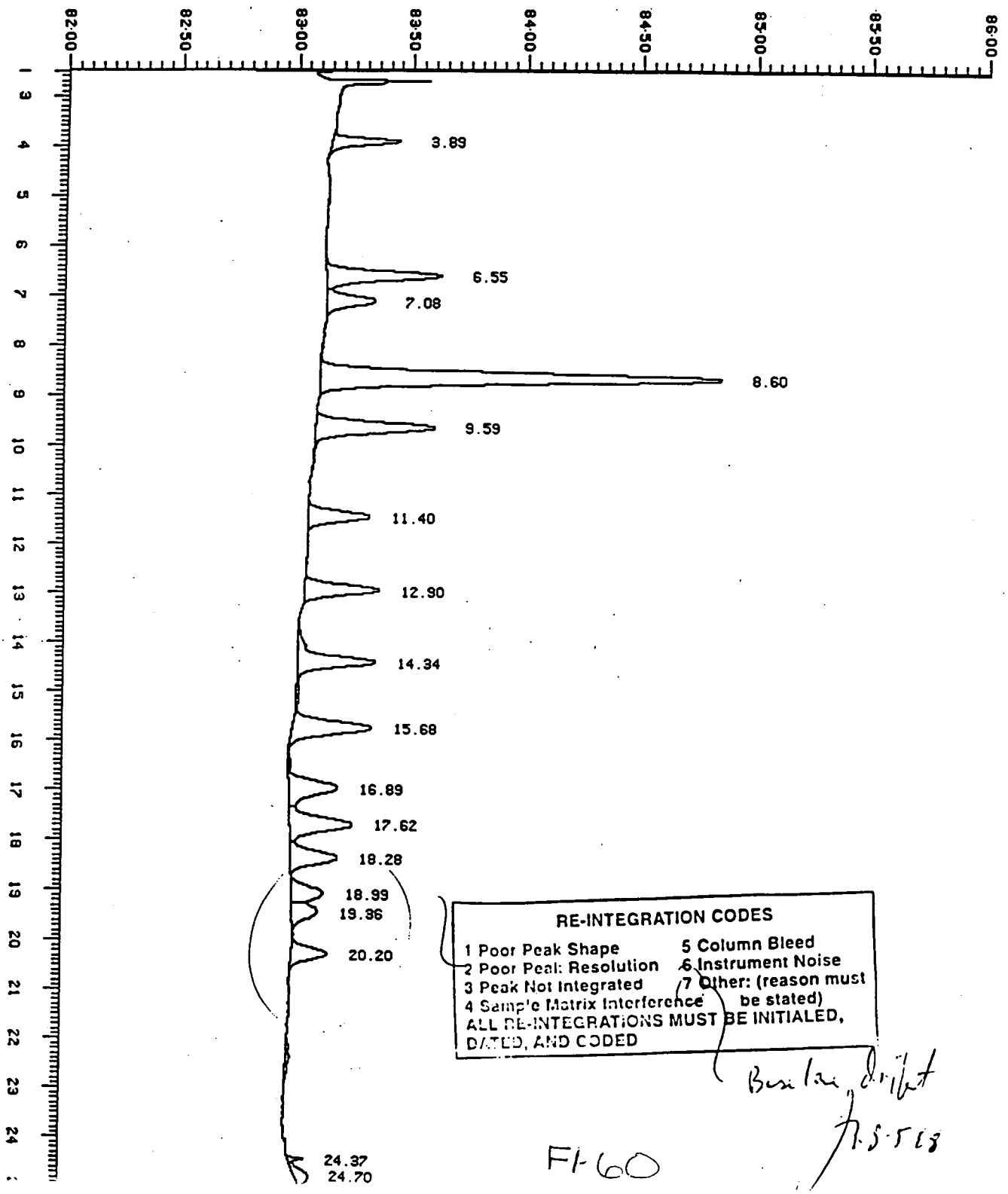
Method..... 8330_HY
Run sequence..... EXP4HY1
Calibration..... 03AUG98
External standard calibration using height
Calibration last modified on 5-AUG-1998 at 14:16
Calibration initialised on 12-DEC-1997 at 21:47
Method last modified on 5-AUG-1998 at 14:15

Uncalibrated peaks use user factor (0.0000)

Quanterra, Inc (West Sacramento)

[AUG_SV] 109 4H_04AUG981450,12,1

E070898F 25/100 8330MIX CV. Amount : 1.000.
Varian 5560/UV9050 @250nm flow=1.5 injection volume=200uL
Acquired on 5-AUG-1998 at 00:18
Reported on 5-AUG-1998 at 14:16
Box 1 (of 1)



RE-INTEGRATION CODES

1 Poor Peak Shape
2 Poor Peak Resolution
3 Peak Not Integrated
4 Sample Matrix Interference
5 Column Bleed
6 Instrument Noise
7 Other: (reason must be stated)

ALL RE-INTEGRATIONS MUST BE INITIALED, DATED, AND CODED

Baseline drift
8-5-98

FI-60

Quanterra / W. Sacramento **Analysis ID:** [AUG_SV] 109 4H_04AUG981450,12
Chromatography Summary **Acquired:** HPLC #4 5-AUG-1998 at 00:18

Sample: E070898F 25/100 8330MIX CV **Mult:** 1.00 **Dilf:** 1.00 **Div:** 1.000
Method File: 8330_HY 75:25:0/2-22-50:00:50/2"- H2O:2%THF/MeOH:Methanol (Analysis)
Calibration: 03AUG98 8330H 2.5-1000ng/mL Phenomenex Hypersil C8 SN251486

Calibration Check

Channel 109 Var-LC5500 UV
Hypersil-C8
150x4.6mm 5um SN251486 8330M

	Peak Name	RT	True	Calc.	%D	-Limits-
1	HMX	3.893	25.00	24.4383	-2.2	(15)
2	1,3,5-Trinitrobenz	6.551	25.00	23.4550	-6.2	(15)
3	RDX	7.076	25.00	24.0136	-3.9	(15)
4	2,4-Dinitrofluorob	8.596	100.0	97.3432	-2.7	(15)
5	1,3-Dinitrobenzene	9.591	25.00	24.1594	-3.4	(15)
6	Nitrobenzene	11.396	25.00	24.9374	-0.25	(15)
7	2,4,6-Trinitrotolu	12.898	25.00	23.9748	-4.1	(15)
8	TETRYL	14.338	25.00	25.4482	1.8	(15)
9	2,4-Dinitrotoluene	15.680	25.00	24.3994	-2.4	(15)
10	2,6-Dinitrotoluene	16.889	25.00	25.7640	3.1	(15)
11	2-AM-4,6-Dinitroto	17.618	25.00	25.8025	3.2	(15)
12	4-AM-2,6-Dinitroto	18.276	25.00	23.2948	-6.8	(15)
13	2-Nitrotoluene	18.987	25.00	25.8356	3.3	(15)
14	4-Nitrotoluene	19.360	25.00	26.6341	6.5	(15)
15	3-Nitrotoluene	20.196	25.00	27.4099	9.6	(15)

RE-INTEGRATION CODES
Peak Shape
Resolution
Integrated
Interfe.
CODED

Baseline drift
9-5-98

Notes:
No limits found 15% used

Average %D = 4.0

Q/WS SUM 3.29A

9-5-98

Reviewer: a Date: 8/5/98
Printed: 08/05/98 14:16:11

FF61

Method Blank (w/SCS)

F-62

METHOD BLANK REPORT
HPLC Analysis Area
Project: 300612

Test: 8330-IRPMS-1C-A Nitroaromatics and Nitramines by HPLC
Method: 8330
Matrix: AQUEOUS
QC Lot: 28 JUL 98-13D QC Run: 28 JUL 98-13D
Analyzed: 04 AUG 98 Time: 18:16

Analyte	Result	Units	Reporting Limit	Qualifier
RDX	ND	ug/L	5.0	
1,3-Dinitrobenzene	ND	ug/L	5.0	
Nitrobenzene	ND	ug/L	5.0	
2,4,6-Trinitrotoluene	ND	ug/L	5.0	
Tetryl	ND	ug/L	5.0	
2,4-Dinitrotoluene	ND	ug/L	5.0	
2,6-Dinitrotoluene	ND	ug/L	5.0	
2-Am-DNT	ND	ug/L	5.0	
4-Am-DNT	ND	ug/L	5.0	
2-Nitrotoluene	ND	ug/L	10	
4-Nitrotoluene	ND	ug/L	8.5	
3-Nitrotoluene	ND	ug/L	7.9	

Surrogate	% Recovery	Acceptable Range
2,4-Dinitrofluorobenzene	89	65 -135

ND = Not Detected

Injection Report

Acquired on 4-AUG-1998 at 18:16

Quanterra, Inc (West Sacramento)

Analyst Name : LC4UV
Lims Id : 109LC4UV9050
Comment : 75:25:0/2-22-50:00:50/2"- H2O:2%THF/MeOH:Methanol (Analysis)
Method Title : Var-LC5500 UV Hypersil-C8 150x4.6mm 5um SN251486 8330M
Sample Name : MB 28JUL98-13D 770ML/20ML
Sample Id :
Sample Type : Sample Amount=1.00000
Bottle No : 8

PEAK INFORMATION

Peak	RT mins	RT Exp	Hght uV	ppb	Peak name	Width
1	8.507	8.476	1595	2.30113	2,4-Dinitrofluorobenzene(Surr)	12.8
2	13.876	14.078	34	0.06702	TETRYL	12.3
3	21.618	0.000	27	0.00000		38.4
4	22.649	0.000	14	0.00000		12.3
5	24.311	0.000	59	0.00000		23.5A
6	24.400	0.000	109	0.00000		8.5A
7	24.658	0.000	32	0.00000		4.8

Totals		
Unknowns	241	0.00000
Named	1629	2.36814
Grand Total	1870	2.36814

ANALYSIS SUMMARY

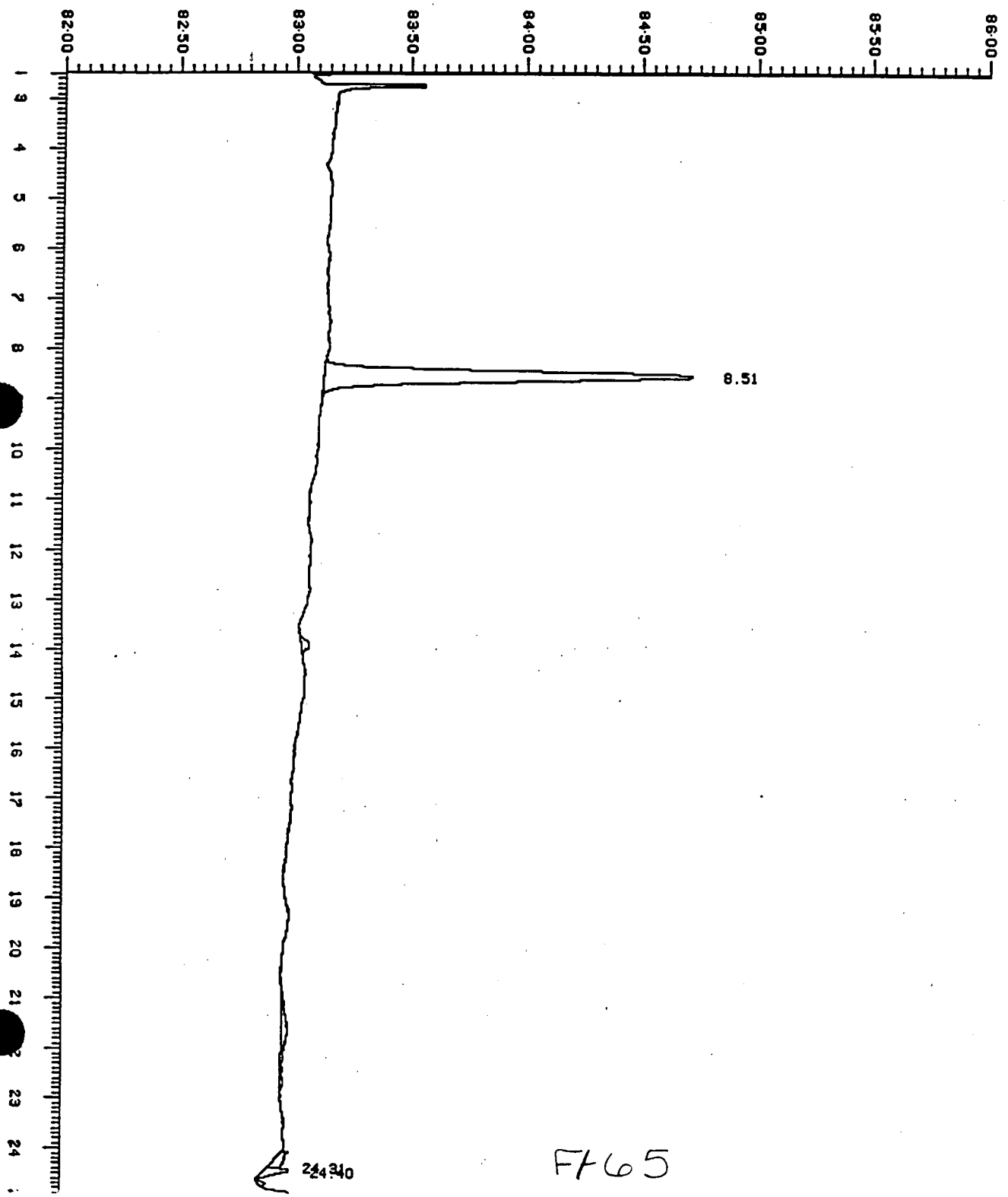
Method..... 8330 HY
Run sequence..... EXP4HY1
Calibration..... 03AUG98
External standard calibration using height
Calibration last modified on 4-AUG-1998 at 17:13
Calibration initialised on 12-DEC-1997 at 21:47
Method last modified on 4-AUG-1998 at 16:40

Uncalibrated peaks use user factor (0.0000)

Quanterra, Inc (West Sacramento)

[AUG_SV] 109 4H_04AUG981450,2,1

MB 28JUL98-13D 770ML/20ML. Amount : 1.000.
Varian 5560/UV9050 @250nm flow=1.5 injection volume=200uL
Acquired on 4-AUG-1998 at 18:16
Reported on 4-AUG-1998 at 18:41
Box 1 (of 1)



F465

Quanterra / W. Sacramento **Analysis ID:** [AUG_SV] 109 4H_04AUG981450, 2
Chromatography Summary **Acquired:** HPLC #4 4-AUG-1998 at 18:16

Sample: MB 28JUL98-13D 770ML/20ML **Mult:** 20.00 **Dilf:** 1.00 **Div:** 770.0
Method File: 8330_HY 75:25:0/2-22-50:00:50/2"- H2O:2%THF/MeOH:Methanol (Analysis)
Calibration: 03AUG98 8330H 2.5-1000ng/mL Phenomenex Hypersil C8 SN251486

Target Analyte Results

Channel 109 Var-LC5500 UV
Hypersil-C8
150x4.6mm 5um SN251486 8330M

	Peak Name	RT	Diff	Height	ppb	R	P	RL
1	HMX							1.00
2	1,3,5-Trinitrobenz							0.300
3	RDX							0.800
4	2,4-Dinitrofluorob	8.507	+0.03	1595	2.3011		F	
5	1,3-Dinitrobenzene							0.100
6	Nitrobenzene							1.00
7	2,4,6-Trinitrotolu							0.130
8	TETRYL	13.876	-0.20	34	0.0670	U	F	1.00
9	2,4-Dinitrotoluene							0.100
10	2,6-Dinitrotoluene							0.300
11	2-AM-4,6-Dinitroto							0.130
12	4-AM-2,6-Dinitroto							0.130
13	2-Nitrotoluene							1.00
14	4-Nitrotoluene							1.00
15	3-Nitrotoluene							1.00

Surrogate Results

	Peak Name	Amount Spiked	Amount Found	%Rec	-Limits-
4	2,4-Dinitrofluorob	2.60	2.3011	89	(65 - 135)

Notes:
8330
Quantitation limits exceed RL

Range Flags (R)
U - Under calibration Range
O - Over calibration Range
X - Not calibrated

Peak Type Flags (P)
* - Flat top Peak
F - Forced baseline
M - Modified baseline

Q/WS SUM 3.29A

5-5-98

Reviewer: a Date: 8/5/98
Printed: 08/04/98 18:41:36

FH66

Laboratory/Duplicate Control Sample

DUPLICATE CONTROL SAMPLE REPORT
HPLC Analysis Area
Project: 300612

Category: 8330-COE-A Explosives by HPLC
Testcode: 8330-IRPMS-1C-A
Matrix: AQUEOUS
QC Lot: 28 JUL 98-13D

Method: 8330
Concentration Units: ug/L
Analyzed Date: 04 AUG 98 Time: 19:28

Analyte	Concentration Spiked	Concentration Measured DCS1	Concentration Measured DCS2	Accuracy (%) DCS1	Accuracy (%) DCS2	Limits	Precision (RPD) DCS	Precision (RPD) Limit
HMX	0.500	0.468	0.473	94	95	65-135	1.2	35
1,3,5-Trinitrobenzene	0.500	0.468	0.470	94	94	65-135	0.51	35
RDX	0.500	0.432	0.453	86	91	65-135	4.6	35
1,3-Dinitrobenzene	0.500	0.425	0.461	85	92	65-135	8.1	35
Nitrobenzene	0.500	0.369	0.458	74	92	65-135	22	35
2,4,6-Trinitrotoluene	0.500	0.511	0.495	102	99	65-135	3.2	35
Tetryl	0.500	0.510	0.512	102	102	65-135	0.47	35
2,4-Dinitrotoluene	0.500	0.486	0.485	97	97	65-135	0.12	35
2,6-Dinitrotoluene	0.500	0.432	0.487	86	97	65-135	12	35
2-Am-DNT	0.500	0.453	0.478	91	96	65-135	5.2	35
4-Am-DNT	0.500	0.482	0.456	96	91	65-135	5.5	35
2-Nitrotoluene	0.500	0.414	0.504	83	101	65-135	19	35
4-Nitrotoluene	0.500	0.413	0.552	83	110	65-135	29	35
3-Nitrotoluene	0.500	0.430	0.523	86	105	65-135	20	35

Surrogate	Concentration Spiked	Concentration Measured DCS1	Concentration Measured DCS2	Accuracy(%) DCS1	Accuracy(%) DCS2	Limits
2,4-Dinitrofluorobenzene	2.6	2.4	2.4	90	92	65-135

Calculations are performed before rounding to avoid round-off errors in calculated resul

Injection Report

Acquired on 4-AUG-1998 at 18:52

Quanterra, Inc (West Sacramento)

Analyst Name : LC4UV
Lims Id : 109LC4UV9050
Comment : 75:25:0/2-22-50:00:50/2"- H2O:2%THF/MeOH:Methanol (Analysis)
Method Title : Var-LC5500 UV Hypersil-C8 150x4.6mm 5um SN251486 8330M
Sample Name : DCS1 28JUL98-13D 770ML/20ML
Sample Id :
Sample Type : Sample Amount=1.00000
Bottle No : 8

PEAK INFORMATION

Peak	RT mins	RT Exp	Hght uV	ppb	Peak name	Width
1	3.804	3.769	216	0.46767	HMX	8.0
2	6.480	6.493	386	0.46791	1,3,5-Trinitrobenzene	11.2
3	6.880	6.907	149	0.43213	RDX	12.8
4	8.462	8.476	1629	2.35050	2,4-Dinitrofluorobenzene(Surr)	12.3
5	9.422	9.444	352	0.42461	1,3-Dinitrobenzene	13.3
6	10.276	0.000	12	0.00000		14.9
7	11.253	11.271	153	0.36882	Nitrobenzene	12.3
8	12.711	12.729	265	0.51105	2,4,6-Trinitrotoluene (TNT)	12.8
9	14.036	14.078	262	0.50981	TETRYL	12.3
10	15.369	15.413	262	0.48551	2,4-Dinitrotoluene	16.5
11	16.516	16.576	139	0.43250	2,6-Dinitrotoluene	17.1
12	17.129	17.178	183	0.45330	2-AM-4,6-Dinitrotoluene	16.5
13	17.778	17.836	161	0.48171	4-AM-2,6-Dinitrotoluene	17.1
14	18.622	18.687	85	0.41414	2-Nitrotoluene	18.7
15	19.022	19.080	69	0.41340	4-Nitrotoluene	19.7
16	19.902	19.942	93	0.43015	3-Nitrotoluene	15.5
17	24.382	0.000	59	0.00000		3.2
18	24.773	0.000	98	0.00000		12.3

Totals		
Unknowns	169	0.00000
Named	4405	8.64320
Grand Total	4574	8.64320

F469

ANALYSIS SUMMARY

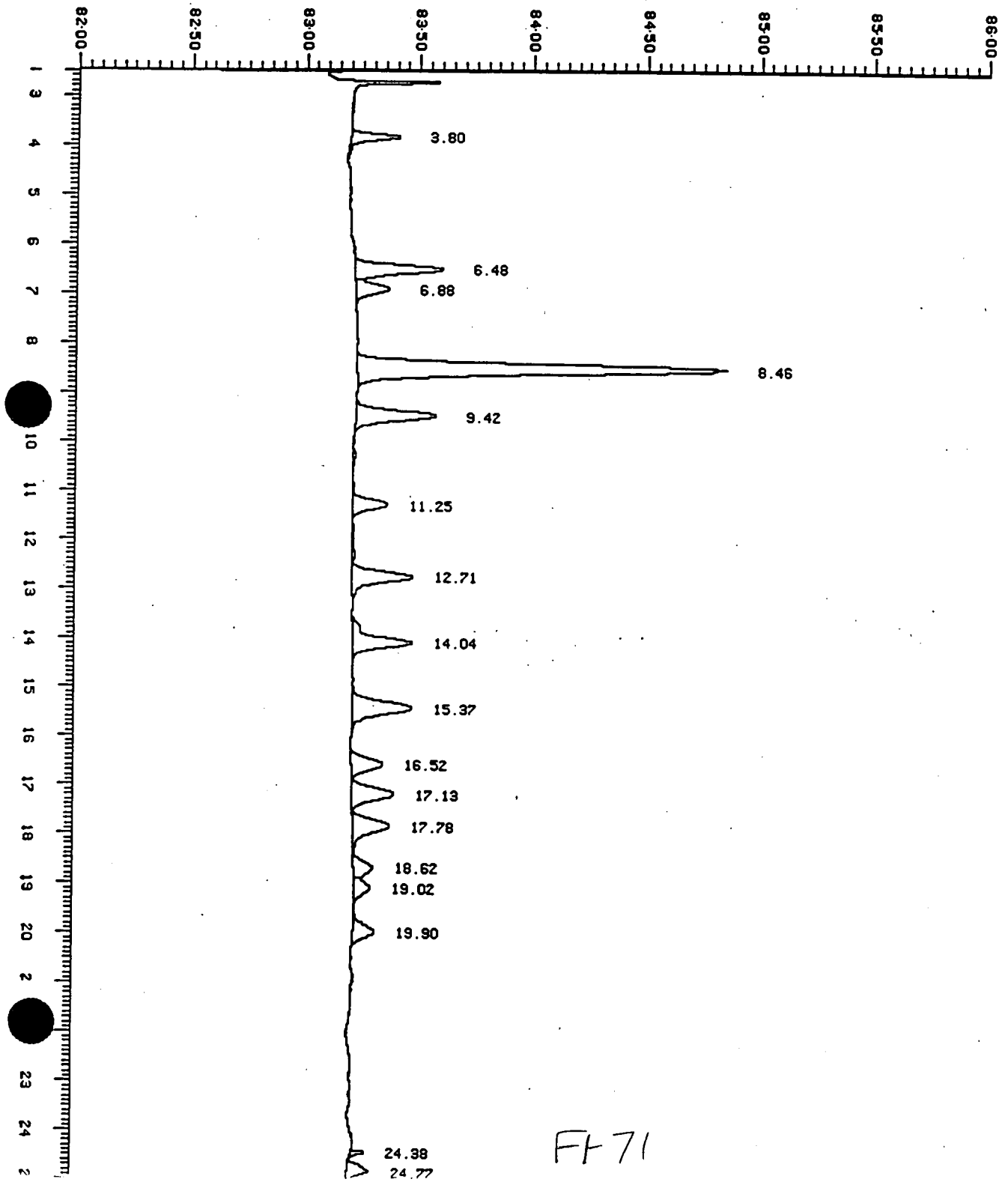
Method..... 8330_HY
Run sequence..... EXP4HY1
Calibration..... 03AUG98
External standard calibration using height
Calibration last modified on 4-AUG-1998 at 18:41
Calibration initialised on 12-DEC-1997 at 21:47
Method last modified on 4-AUG-1998 at 16:40

Uncalibrated peaks use user factor (0.0000)

Quanterra, Inc (West Sacramento)

[AUG_SV] 109 4H_04AUG981450,3,1

DCS1 28JUL98-13D 770ML/20ML. Amount : 1.000.
Varian 5560/UV9050 @250nm flow=1.5 injection volume=200uL
Acquired on 4-AUG-1998 at 18:52
Reported on 4-AUG-1998 at 19:17
Box 1 (of 1)



FH71

Quanterra / W. Sacramento
Chromatography Summary

Analysis ID: [AUG_SV] 109 4H_04AUG981450, 3
Acquired: HPLC #4 4-AUG-1998 at 18:52

Sample: DCS1 28JUL98-13D 770ML/20ML **Mult:** 20.00 **Dilf:** 1.00 **Div:** 770.0
Method File: 8330_HY 75:25:0/2-22-50:00:50/2"- H2O:2%THF/MeOH:Methanol (Analysis)
Calibration: 03AUG98 8330H 2.5-1000ng/mL Phenomenex Hypersil C8 SN251486

Channel 109 Var-LC5500 UV

Laboratory Control Sample Hypersil-C8
150x4.6mm 5um SN251486 8330M

	Peak Name	RT	Spiked	Found	Rec	-Limits-
1	HMX	3.804	0.500	0.4677	94	(65 - 135)
2	1,3,5-Trinitrobenz	6.480	0.500	0.4679	94	(65 - 135)
3	RDX	6.880	0.500	0.4321	86	(65 - 135)
4	2,4-Dinitrofluorob	8.462	2.60	2.3505	90	(65 - 135)
5	1,3-Dinitrobenzene	9.422	0.500	0.4246	85	(65 - 135)
6	Nitrobenzene	11.253	0.500	0.3688	74	(65 - 135)
7	2,4,6-Trinitrotolu	12.711	0.500	0.5111	102	(65 - 135)
8	TETRYL	14.036	0.500	0.5098	102	(65 - 135)
9	2,4-Dinitrotoluene	15.369	0.500	0.4855	97	(65 - 135)
10	2,6-Dinitrotoluene	16.516	0.500	0.4325	86	(65 - 135)
11	2-AM-4,6-Dinitroto	17.129	0.500	0.4533	91	(65 - 135)
12	4-AM-2,6-Dinitroto	17.778	0.500	0.4817	96	(65 - 135)
13	2-Nitrotoluene	18.622	0.500	0.4141	83	(65 - 135)
14	4-Nitrotoluene	19.022	0.500	0.4134	83	(65 - 135)
15	3-Nitrotoluene	19.902	0.500	0.4302	86	(65 - 135)

Surrogate Results

	Peak Name	Amount Spiked	Amount Found	%Rec	-Limits-
4	2,4-Dinitrofluorob	2.60	2.3505	90	(65 - 135)

Notes:
8330

Q/WS SUM 3.29A

Reviewer: a Date: 8/8/98
Printed: 08/05/98 17:04:06

FF72

Injection Report

Acquired on 4-AUG-1998 at 19:28

Quanterra, Inc (West Sacramento)

Analyst Name : LC4UV
Lims Id : 109LC4UV9050
Comment : 75:25:0/2-22-50:00:50/2"- H2O:2%THF/MeOH:Methanol (Analysis)
Method Title : Var-LC5500 UV Hypersil-C8 150x4.6mm 5um SN251486 8330M
Sample Name : DCS2 28JUL98-13D 770ML/20ML
Sample Id :
Sample Type : Sample Amount=1.00000
Bottle No : 8

PEAK INFORMATION

Peak	RT mins	RT Exp	Hght uV	ppb	Peak name	Width
1	3.813	3.769	219	0.47340	HMX	8.0
2	6.489	6.493	388	0.47031	1,3,5-Trinitrobenzene	11.2
3	6.889	6.907	156	0.45265	RDX	12.8
4	8.471	8.476	1663	2.39968	2,4-Dinitrofluorobenzene(Surr)	12.3
5	9.431	9.444	382	0.46064	1,3-Dinitrobenzene	12.8
6	10.276	0.000	13	0.00000		9.6
7	11.262	11.271	190	0.45786	Nitrobenzene	12.3
8	12.720	12.729	256	0.49485	2,4,6-Trinitrotoluene (TNT)	12.8
9	14.062	14.078	263	0.51216	TETRYL	13.9
10	15.396	15.413	262	0.48493	2,4-Dinitrotoluene	16.0
11	16.569	16.576	156	0.48725	2,6-Dinitrotoluene	17.1
12	17.173	17.178	193	0.47763	2-AM-4,6-Dinitrotoluene	17.1
13	17.849	17.836	153	0.45595	4-AM-2,6-Dinitrotoluene	17.1
14	18.676	18.687	104	0.50345	2-Nitrotoluene	13.9A
15	19.084	19.080	92	0.55199	4-Nitrotoluene	21.3A
16	19.956	19.942	114	0.52330	3-Nitrotoluene	15.5
17	24.382	0.000	64	0.00000		3.2
18	24.773	0.000	84	0.00000		11.2

Totals		
Unknowns	161	0.00000
Named	4590	9.20605
Grand Total	4751	9.20605

ANALYSIS SUMMARY

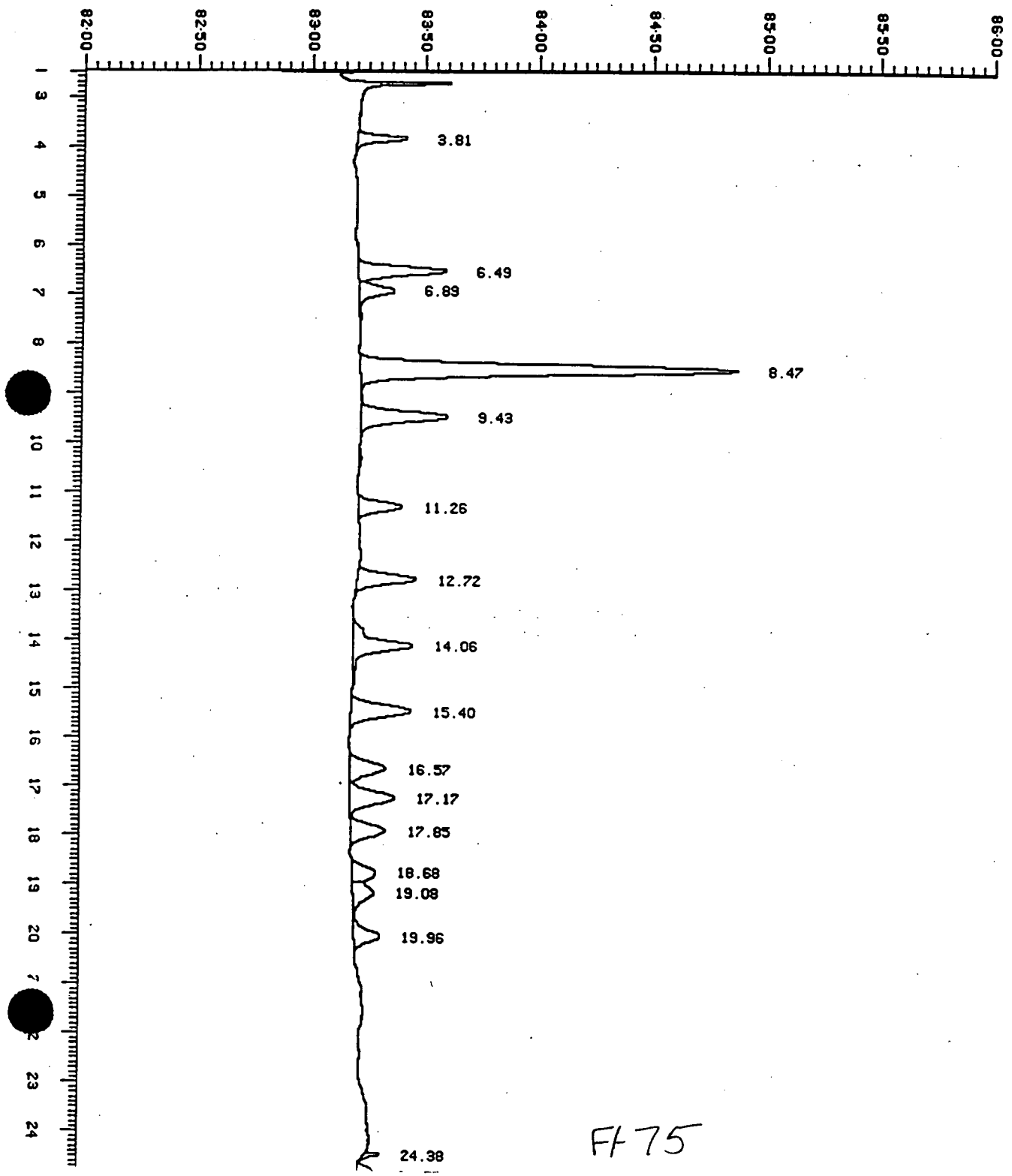
Method..... 8330_HY
Run sequence..... EXP4HY1
Calibration..... 03AUG98
External standard calibration using height
Calibration last modified on 4-AUG-1998 at 19:17
Calibration initialised on 12-DEC-1997 at 21:47
Method last modified on 4-AUG-1998 at 16:40

Uncalibrated peaks use user factor (0.0000)

Quanterra, Inc (West Sacramento)

[AUG_SV] 109 4H_04AUG981450,4,1

DCS2 28JUL98-13D 770ML/20ML. Amount : 1.000.
Varian 5560/UV9050 @250nm flow=1.5 injection volume=200uL
Acquired on 4-AUG-1998 at 19:28
Reported on 4-AUG-1998 at 19:54
Box 1 (of 1)



F475

Quanterra / W. Sacramento
Chromatography Summary

Analysis ID: [AUG_SV] 109 4H_04AUG981450, 4
Acquired: HPLC #4 4-AUG-1998 at 19:28

Sample: DCS2 28JUL98-13D 770ML/20ML **Mult:** 20.00 **Dilf:** 1.00 **Div:** 770.0
Method File: 8330_HY 75:25:0/2-22-50:00:50/2"- H2O:2%THF/MeOH:Methanol (Analysis)
Calibration: 03AUG98 8330H 2.5-1000ng/mL Phenomenex Hypersil C8 SN251486

Channel 109 Var-LC5500 UV

Laboratory Control Sample Hypersil-C8
150x4.6mm 5um SN251486 8330M

	Peak Name	RT	Spiked	Found	Rec	-Limits-
1	HMX	3.813	0.500	0.4734	95	(65 - 135)
2	1,3,5-Trinitrobenz	6.489	0.500	0.4703	94	(65 - 135)
3	RDX	6.889	0.500	0.4526	91	(65 - 135)
4	2,4-Dinitrofluorob	8.471	2.60	2.3997	92	(65 - 135)
5	1,3-Dinitrobenzene	9.431	0.500	0.4606	92	(65 - 135)
6	Nitrobenzene	11.262	0.500	0.4579	92	(65 - 135)
7	2,4,6-Trinitrotolu	12.720	0.500	0.4949	99	(65 - 135)
8	TETRYL	14.062	0.500	0.5122	102	(65 - 135)
9	2,4-Dinitrotoluene	15.396	0.500	0.4849	97	(65 - 135)
10	2,6-Dinitrotoluene	16.569	0.500	0.4872	97	(65 - 135)
11	2-AM-4,6-Dinitroto	17.173	0.500	0.4776	96	(65 - 135)
12	4-AM-2,6-Dinitroto	17.849	0.500	0.4560	91	(65 - 135)
13	2-Nitrotoluene	18.676	0.500	0.5035	101	(65 - 135)
14	4-Nitrotoluene	19.084	0.500	0.5520	110	(65 - 135)
15	3-Nitrotoluene	19.956	0.500	0.5233	105	(65 - 135)

Surrogate Results

	Peak Name	Amount Spiked	Amount Found	%Rec	-Limits-
4	2,4-Dinitrofluorob	2.60	2.3997	92	(65 - 135)

Notes:
8330

Q/WS SUM 3.29A

Reviewer: a Date: 8/5/98
Printed: 08/05/98 17:05:40

FF 76

Analysis Log Copies

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Quanterra / W. Sacramento
CHROMATOGRAPHY RUN LOG

Analysis: HPLC #4 [AUG_SV] 4PC04AUG980849

Channel **Column** **Detector**
109 / Hypersil-C8 / UV

Method File: 8330_HY
Calib File: 03AUG98
8330H 2.5-1000ng/mL Phenomenex Hypersil C8 SN251486

Analysis Info: 75:25:0/2-22-50:00:50/2" H2O:2%THF/MeOH:Methanol (Curve)

#	SAMPLE NAME	MULT	DIVS	DATE	TIME	LVL
1	PRIMER	1.00	1.000	4-AUG-1998	09:05	
2	PRIMER	1.00	1.000	4-AUG-1998	09:42	
3	Water blank	1.00	1.000	4-AUG-1998	10:18	
4	Water blank	1.00	1.000	4-AUG-1998	10:54	
5	E070898F 25/100ng/mL STD=I	1.00	1.000	4-AUG-1998	11:30	3
6	E070898C 2.5/5.0ng/ml STD=I	1.00	1.000	4-AUG-1998	12:06	T1
7	E070898D 5.0/25ng/mL STD=I	1.00	1.000	4-AUG-1998	12:43	1
8	E070898E 10/50ng/ml STD=I	1.00	1.000	4-AUG-1998	13:19	2
9	E070898G 100/150 STD=I	1.00	1.000	4-AUG-1998	13:55	5
10	E070898H 500 ng/mL 8330 Mix STD=I	1.00	1.000	4-AUG-1998	14:31	6
11	E070898I 1000 ng/mL 8330 Mix STD=I	1.00	1.000	4-AUG-1998	15:08	7
12	Water blank	1.00	1.000	4-AUG-1998	15:44	

QESS Elog 2.0

Analyst: _____ Date: 8-4-98 Page: 148
Printed: 04-AUG-1998 16:48:58

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Quanterra / W. Sacramento
CHROMATOGRAPHY RUN LOG

Analysis: HPLC #4 [AUG_SV] 4H_04AUG981450

Channel Column Detector
109 / Hypersil-C8 / UV

Method File: 8330_HY
Calib File: 03AUG98
8330H 2.5-1000ng/mL Phenomenex Hypersil C8 SN251486

Analysis Info: 75:25:0/2-22-50:00:50/2"- H2O:2%THF/MeOH:Methanol (Analysis)

#	SAMPLE NAME	MULT	DIVS	DATE	TIME	LVL
1	E070898F 25/100 8330MIX CV	1.00	1.000	4-AUG-1998	16:47	3
2	MB 28JUL98-13D 770ML/20ML	20.0	770.0	4-AUG-1998	18:16	LA
3	DCS1 28JUL98-13D 770ML/20ML	20.0	770.0	4-AUG-1998	18:52	LA
4	DCS2 28JUL98-13D 770ML/20ML	20.0	770.0	4-AUG-1998	19:28	LA
5	300592-2 770ML/20ML	20.0	770.0	4-AUG-1998	20:04	LA
6	300602-1 770ML/20ML	20.0	770.0	4-AUG-1998	20:41	LA
7	300602-2 770ML/20ML	20.0	770.0	4-AUG-1998	21:17	LA
8	300602-2-MS 770ML/20ML	20.0	770.0	4-AUG-1998	21:53	LA
9	300602-2-SD 770ML/20ML	20.0	770.0	4-AUG-1998	22:29	LA
10	300612-1 770ML/20ML	20.0	770.0	4-AUG-1998	23:06	LA
11	300551-11 770ML/20ML	20.0	770.0	4-AUG-1998	23:42	LA
12	E070898F 25/100 8330MIX CV	1.00	1.000	5-AUG-1998	00:18	3
13	MB 29JUL98-11D 770ML/20ML	20.0	770.0	5-AUG-1998	00:54	LA
14	DCS1 29JUL98-11D 770ML/20ML	20.0	770.0	5-AUG-1998	01:31	LA
15	DCS2 29JUL98-11D 770ML/20ML	20.0	770.0	5-AUG-1998	02:07	LA
16	300575-1 770ML/20ML	20.0	770.0	5-AUG-1998	02:43	LA
17	300575-2 770ML/20ML	20.0	770.0	5-AUG-1998	03:19	LA
18	MB 28JUL98-11D 0.4G/8ML	8.00	0.400	5-AUG-1998	03:55	LS
19	DCS1 28JUL98-11D 0.4G/8ML	8.00	0.400	5-AUG-1998	04:32	LS
20	DCS2 28JUL98-11D 0.4G/8ML	8.00	0.400	5-AUG-1998	05:08	LS
21	132219-1 0.4G/8ML	8.00	0.400	5-AUG-1998	05:44	LS
22	132219-2 0.4G/8ML	8.00	0.400	5-AUG-1998	06:20	LS
23	E070898F 25/100 8330MIX CV	1.00	1.000	5-AUG-1998	06:57	3
24	132219-3 0.4G/8ML	8.00	0.400	5-AUG-1998	07:33	LS
25	132219-4 0.4G/8ML	8.00	0.400	5-AUG-1998	08:09	LS
26	132219-5 0.4G/8ML	8.00	0.400	5-AUG-1998	08:45	LS
27	132219-6 0.4G/8ML	8.00	0.400	5-AUG-1998	09:21	LS
28	132219-7 0.4G/8ML	8.00	0.400	5-AUG-1998	09:58	LS
29	132219-7-MS 0.4G/8ML	8.00	0.400	5-AUG-1998	10:34	LS
30	132219-7-SD 0.4G/8ML	8.00	0.400	5-AUG-1998	11:10	LS
31	MB 28JUL98-12D 0.4G/8ML	8.00	0.400	5-AUG-1998	11:46	LS
32	LCS 28JUL98-12D 0.4G/8ML	8.00	0.400	5-AUG-1998	12:23	LS
33	300551-1 0.4G/8ML	8.00	0.400	5-AUG-1998	12:59	LS
34	E070898F 25/100 8330MIX CV	1.00	1.000	5-AUG-1998	13:35	3
35	300551-1-MS 0.4G/8ML	8.00	0.400	5-AUG-1998	14:11	LS

***** Printed before sequence complete *****

Analyst: [signature] Date: 5-5-98 Page: 149
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Sample Extraction /Preparation Log Copies

QUANTERRA INCORPORATED

West Sacramento

DUE DATE: 7-28

PROJECT ANOMALY RECORD AND DATA CHECKLIST

PROJECT # 300612 *TEST DESC.* 8330 - IRPMS - KC-A

DATA CHECKLIST

CALIBRATION
__ICAL or ICAL Summary & ICV/CCV included.
__ICAL, ICV/CCV criteria met.
__Standards labeled or correctly identified by data system
__Copy of logbook for ICAL included.
__Tune criteria met and copy included (GCMS only).
__Method number is identified on data.

QAQC VERIFICATION
__Method blank and DCS/LCS frequencies were met.
__DCS/LCS and MBcopies are included if applicable.
__DCS/LCS and MBdata are within control limits.
__If the batch QC data did not meet criteria, appropriate comments were made.
__MS/MSD data complete if applicable.
__Holding times were met.
__All samples within tune time (GCMS only).

SAMPLE ANALYSIS
__Logbook copies for all injections made, including ICV/CCV standards and ICAL.
__Logbooks/Prep bench sheets are properly filled out.
__Manual integrations are reviewed.
__All raw data is included.
__All analytes are reported correctly.
__Correct reporting limits were used.
__Spectra are present for all positive analytes (GCMS only).

LIMS
__Results were entered into LIMS correctly.
__The prepared and analytical dates are correct.
__The correct MB/DCS/LCS data were entered.
__The correct footnotes were used.
__The data sheets are complete and included
__Method blanks are included with correct prep and analyzed dates
__Anomalies are written and entered.

SIGNATURES BELOW INDICATE THE ABOVE CRITERIA HAVE BEEN MET.

CHEMIST [signature] DATE 8 5 98
REVIEWER Emily Lobelhoer DATE 8/5/98

SEE ELECTRONIC ANOMALY: X

NO ANOMALIES: _____

COMMENTS:

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QUANTERRA-CALLAB LABORATORY
Chromatography Assignment Sheet

Project #: 300612
Analysis: P-8330-COE-A Ver: 1
Client: [redacted]
PA: DBROOKS
Delivery Accepted: MGARCIA 25 JUL 98
Number of Samples: 1
Program: Explosives

Due Date: 06 AUG 98
Receipt Date: 25 JUL 98
Log Released: 27 JUL 98
Turnaround: RUSH
Storage Location: WR1G
Earliest Holding Time: 31 JUL 98

Project Description:
Aqueous for 8330-Total.

5-day TAT for 8330 level 2 and 10-day TAT for level 3.

Low PQLs for the aqueous sample - J values
IRPMS entry needed

Full raw data package - blue sheet order

Test Modified: N
Sample Instructions: N
Test Instructions: N
Group Instructions: N
QAS #: _____

Date Delivered to PA: _____

Comments:

__ No Problems Occurred with Analysis

__ See Anomaly Sheet

QUANTERRA-CALLAB LABORATORY
Bottle Descriptions

Project #: 300612 Test: P-8330-COE-A

0001SA - 2-AGB

QUANTERRA-CALLAB LABORATORY
Analytical Bench Sheet

Project Number: 300612 Date: 27 JUL 98

Test Code: P-8330-COE-A Test Description: Prep - Explosives by HPLC

Turnaround Status: RUSH Sample Matrix: AQUEOUS Date Assigned:_____ Prep Completion Due:_____

SOP-Section/Page (Revision):_____

Extractions: [ ] Separatory Funnel [ ] Continuous Liquid/Liquid [ ] Sonication [ ] Soxhlet [ ] KD

[ ] Roto Vap [ ] Shaker [ ] Other_____

Samp ID	Holding Time	Sample Size	Extractn Date/ Initials	Init. pH	Adj pH Date/ Initials	Hydrolysis Date/ Initials	Acid Ext Date/ Initials	Conc Date/ Initials	CH2N2 Date/ Initials	N2 Date/ Initials	Final Sample Concentn
0001SA	31 JUL 98										

Project Due: 06 AUG 98 DCS With Project:_____ DCS Code:_____

LCS With Project:_____ LCS Code:_____

Method Blank With Project:_____ Extraction Includes Project(s):_____

Notes/Comments:
** PLEASE PUT COMMENTS ON ANOMALY SHEET ** MSQC with Project: _____ MSQC Code: _____

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Date To Instruments: By: Peer Approval By:

QUANTERRA-CALLAB LABORATORY
Quality Assurance Sheet

Project #: 300612 Test: P-8330-COE-A

Quality Assurance/Quality Control:

Sample ID:_____ Spiking Standard Name:_____

Spike Code ID:_____ Volume: _____ Conc: _____

Spiked By:_____ Witness:_____

Sample ID:_____ Spiking Standard Name:_____

Spike Code ID:_____ Volume: _____ Conc: _____

Spiked By:_____ Witness:_____

Sample ID:_____ Spiking Standard Name:_____

Spike Code ID:_____ Volume: _____ Conc: _____

Spiked By:_____ Witness:_____

Sample ID:_____ Spiking Standard Name:_____

Spike Code ID:_____ Volume: _____ Conc: _____

Spiked By:_____ Witness:_____

Solvent: Manufacturer And Lot Number:

Extracting Solvent:_____

Solvent Keeper:_____

Transfer Solvent:_____

Derivatizing Reagent:_____

Final Solvent:_____

Other:_____

_____

Was an aliquot taken? Y / N

If yes, how much was aliquoted?_____mL _____mL

What was the sample concentration before aliquot?_____

_____

Why was the aliquot taken?_____

_____

_____

_____

QUANTERRA-CALLAB LABORATORY
Instruction Sheet

Project #: 300612 Test: P-8330-COE-A

Sample Instructions:

None.

Test Instructions:

None.

Group Instructions:

None.

QUANTERRA-CALLAB LABORATORY
Anomaly Report Form

Project #: 300612 Test: P-8330-COE-A

Anomalies:	Sample(s) Affected:
__ Sample broken during shipping.	_____
__ Limited sample volume.	_____
__ Matrix required a subsample to facilitate extraction.	_____
__ pH of sample required excess acid/base (please circle).	_____
__ Solvent and sample were miscible.	_____
__ Emulsion problems.	_____
__ Emulsion problem required centrifuging.	_____
__ Sample lost during preparation. Re-extraction performed? Yes/No	_____
__ Concentration volume went lower than specified in the SOP.	_____
__ Concentration time longer than normal.	_____
__ Extraction solvent saturated with organic material, therefore sample volume was not concentrated but adjusted based on the screening.	_____
__ Final concentration greater than specified in the SOP due to the screening results.	_____
__ Precipitate observed in the extract.	_____
__ Other, please comment below.	_____

Comments:

Submitted By:_____ Date:_____

QUANTERRA-CALLAB LABORATORY
Prep (Extraction) Check List

Project #: 300612 Test: P-8330-COE-A

Chemist: _____ Date: _____

Reviewer: _____ Date: _____

Number of samples: 1 Number of extracts: _____

Benchsheet:	Initiated		Reviewed	
SOP section and page number filled out properly.	Y___	N___	Y___	N___
Each step of extraction procedure initial'ed and dated.	Y___	N___	Y___	N___
Anomalies or deviations from SOP documented.	Y___	N___	Y___	N___
pH adjustments noted.	Y___	N___	Y___	N___
Solvent manufacturer and lot number completed.	Y___	N___	Y___	N___
Volumes and weights of samples to two sig. figs.	Y___	N___	Y___	N___

QA/QC:	Initiated		Reviewed	
DCS/MS forms filled out properly.	Y___	N___	Y___	N___
Spike information correct on bench sheet.	Y___	N___	Y___	N___
Spike witness?	Y___	N___	Y___	N___
If not spiked per SOP, were spike amounts verified?	Y___	N___	Y___	N___

LIMS:	Initiated		Reviewed	
Extraction date (complete date) correct.	Y___	N___	Y___	N___
QC lot number present and correct.	Y___	N___	Y___	N___
QC Run number present and correct.	Y___	N___	Y___	N___
Prep released (date extract goes to Inst.).	Y___	N___	Y___	N___

Extracts:	Initiated		Reviewed	
Number of extracts agree with benchsheet.	Y___	N___	Y___	N___
Concentrations of extract agree with benchsheet.	Y___	N___	Y___	N___
Testtube rack labelled with project id and test.	Y___	N___	Y___	N___

Comments:

QUANTERRA-CALLAB LABORATORY
Client Descriptions

Project #: 300612 Test: P-8330-COE-A

0001SA - [redacted]

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QUANTERRA / W. SACRAMENTO

PROJECT QC LIMITS

The S.M. Stoller Corporation

Project: CALLAB-300612
Matrix: AQUEOUS

Test Code: 8330-IRPMS-1C-A
Version: 3
QC Category: 8330-COE-A

S# NA		RL	Surrogate/SCS			Lab Control (LCS)			Lab Control (DCS)				Matrix Spike			
Modified Date:		25 JUL 98	24 OCT 95			24 OCT 95			24 OCT 95				20 OCT 94			
Component	CASN	ug/L	ug/L	LCL	UCL	ug/L	LCL	UCL	ug/L	LCL	UCL	RPD	ug/L	LCL	UCL	RPD
2,4-Dinitrofluorobenzene	70348	*	100	65	135	100	65	135	100	65	135	35				
HMX	2691410					50	65	135	50	65	135	35	50	65	135	35
1,3,5-Trinitrobenzene	99354					50	65	135	50	65	135	35	50	65	135	35
RDX	121824	5				50	65	135	50	65	135	35	50	65	135	35
1,3-Dinitrobenzene	99650	5.0				50	65	135	50	65	135	35	50	65	135	35
Nitrobenzene	98953	5.0				50	65	135	50	65	135	35	50	65	135	35
2,4,6-Trinitrotoluene	118967	5.0				50	65	135	50	65	135	35	50	65	135	35
Tetryl	479458	5.0				50	65	135	50	65	135	35	50	65	135	35
2,4-Dinitrotoluene	121142	5.0				50	65	135	50	65	135	35	50	65	135	35
2,6-Dinitrotoluene	606202	5.0				50	65	135	50	65	135	35	50	65	135	35
2-Am-DNT	35572782	5.0				50	65	135	50	65	135	35	50	65	135	35
4-Am-DNT	1946510	5.00				50	65	135	50	65	135	35	50	65	135	35
2-Nitrotoluene	88722	10.0				50	65	135	50	65	135	35	50	65	135	35
4-Nitrotoluene	99990	8.5				50	65	135	50	65	135	35	50	65	135	35
3-Nitrotoluene	99081	7.9				50	65	135	50	65	135	35	50	65	135	35

17-90

300551 300612

PROJECT NO. : 300592 300602 Page 1 of 1

METHOD NO. : SW846 8330 SOIL SMC
SAMPLE MATRIX: AQ
COMPOUND(S) : 8330 by HPLC/UV

MASTER SHEET

Holding time Due: 7-29-98

PROJECT Due: 7-28-98

INITIATED BY: B DANIELS DATE: 7-28-98
COMPLETED BY: DATE:
REVIEWED BY : DATE:
APPROVED BY : DATE:

STORAGE	Weigh	Fortification	Extract		Filter	Dilute		
WR1C W19C WR1C W24A	770gm into 1L vol. flask BAL QA051 BD 7-28-98	By BD 7-28-98 Witness [illegible] 7-28-98	+251.4 NACL +164 ml ACN Stir, Stand decant. +8ml ACN, Stir, Stand, decant BD 7-28-98	+85 ml SALT H2O Stir Stand, decant +2ml ACN Stir, Stand, decant	Bring to 5.0ml with ACN. +5ug Acetic Acid. Filter thru 25mm nylon disk BD 7-29-98	1ml extract 3ml HPLC grade H2O BD 7-29-98		

Comments:

CAL ID/SAMPLE DATA	SAMPLE SIZE (gm)	FORTIFICATION - STANDARD DATA PPB	COMPOUND(S)	ALIQUOT	STD. CONC.	STD. NO.	(g/ml) Conc.	(g/ml) Dilution	
300592-MB	770						770/5	770/20	8330 Surrogate
28Jul98 13D	770	0.5	8330 mix	770	5.0ug/ml	E070898A	770/5	770/20	added to all
↓ 13 DD	770	0.5	↓	770	↓		770/5	770/20	40ul of 50ug/ml
300592-2	770						770/5	770/20	to yield 2.6ppb
300602-1	770						770/5	770/20	# E060898C
~~2X~~	770						770/5	770/20	
~~2MS~~	770	0.5	8330 mix	770	5.0ug/ml	E070898A	770/5	770/20	
✓ ~~2SD~~	770	0.5	↓	770	↓	↓	770/5	770/20	
300612-1	770						770/5	770/20	
300551-11	770						770/5	770/20	

EXACT COPY OF ORIGINAL

BOILER 5-26-94.

F-91

APPENDIX G

TOXICITY TESTING AND LEACHING PROCEDURES

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1.0
INTRODUCTION

This appendix will define the testing of compost material from the Naval Surface Warfare Center (NSWC), Crane Division, Crane, Indiana to determine the toxic affects of the treated compost material when in the environment of terrestrial invertebrates. This appendix will supplement the approved *Quality Assurance Project Plan for Full-Scale Operations.* [MK, 1998]

In this study, five compost samples will be collected from one windrow on day final of composting activities. The windrow selected for sampling will contain the highest contaminant levels of explosives based on baseline (day 0 of the treatment cycle) sample results from the first set of full scale windrows for that SWMU. This pile will be representative for the SWMU and represent the worst-case scenario for treated compost material based on the selection criteria utilized.

The treated soil/compost blend from each of the four Solid Waste Management Units (SWMUs 3/10, 10/15, 12/14, and 13/14) will be sampled and analyzed to document that the treated soil meets toxicity remedial goals. This appendix provides the following information:

- Remedial Goals for Toxicity Tests
- Laboratory Standard Operating Procedures
- Laboratory Contacts and Addresses
- Quality Control Procedures
- Test report preparation

Refer to Appendix G of the approved *Full-Scale Operational Plan* [MK, 1998] for sample location and frequency information, collection and splitting procedures, sample handling, and test method descriptions.

In addition to sampling the soil/compost blend from each of the SWMUs, a control pile will be constructed for each SWMU to provide material control samples. The results from control samples will be utilized to aid in the development of background levels for the compost material. In addition, the control pile will provide a basis for determining if the amendment ratios are toxic. Appendix G of the *Full-Scale Operational Plan* [MK, 1998] Section 2.0 describes how the control windrow pile will be created.

2.0
TEST METHODS

Table G-1 summarizes the testing methods.

TABLE G-1 TOXICITY AND LEACHABILITY TESTING SUMMARY				
Test and Test Method	**Frequency**	**Holding Time**	**Preservative**	**Sample Size/Container**
Microtox (Proprietary method of AZUR Environmental)	5 per SWMU + 1 control	14 days	4°C	8 ounce glass
Earthworm Toxicity (EPA Method 600/3-88/029)	5 per SWMU + 1 control	3 days	4°C	3.5 gallon HDPE plastic container lined with 2 plastic bags
Pathogen Testing (40 CFR 503.32)	5 per SWMU + 1 control	3 days	4°C	8 ounce glass
Toxicity Characteristic Leaching Procedure (TCLP) (EPA Method 1311) and Explosives Analysis (EPA Method 8330)	5 per SWMU	14 days for extraction; 40 days from extraction to analysis	4°C	8 ounce glass
Synthetic Precipitation Leaching Procedure (SPLP) (EPA Method 1312) and Explosives Analysis (EPA Method 8330)	5 per SWMU	14 days for extraction; 40 days from extraction to analysis	4°C	8 ounce glass

Samples will be maintained from collection through receipt until testing at 4°C and then brought to 20°C immediately prior to testing.

Tox G001

3.0
REMEDIAL GOALS AND OBJECTIVES

This appendix provides the design and specifications of the toxicity testing to be performed at NSWC Crane. The testing is intended to show the toxic effects that the treated compost material may have on the environment. These effects will be studied using two toxicity methods: Microtox and Earthworm Toxicity, two pathogen tests: fecal coliform, and salmonella, and two leaching procedures: TCLP and SPLP.

For each SWMU a single sample from the control windrow will be collected to provide background data for comparison and interpretation. Toxicity data of the samples collected from the treated contaminated soil windrow will be directly compared to the background sample to determine if there is an elevation in toxicity levels due to the treated explosive contaminants.

The objective of the toxicity testing is to assess the acute toxicity of the compost to worms and the bacteria. For the Microtox assays, the EC-50 will be estimated to determine the elutriate compost concentration which causes an 50% decrease in the illuminance of the test organisms. The EC-50 for the five samples will be averaged and compared to the EC-50 for the control pile to show a comparable or lower toxicity in the compost samples than in the control pile. For the earthworm toxicity the LC-50 will be estimated to determine the compost concentration which causes 50% mortality of earthworms. The LC-50 for the five samples will be averaged and compared to the LC-50 for the control pile to show comparable or lower toxicity in the compost samples than in the control pile.

Pathogen objectives were based on specifications found in 40 CFR 503.32 which pertains to the disposal of sewage sludge.. These specifications show that sewage sludge may be prepared for sale or given away as an application to the land if the sludge meets the requirements of fecal coliform and salmonella. The treated compost is intended to be returned to the land for similar purpose, and therefore these same standards will be applied to the compost material. Objectives for the density of fecal coliform shall be less than 1000 Most Probable Number per gram of dry solids, and Salmonella shall be less than 3 Most Probable Number per 4 grams of dry solids.

Two tests will be performed to demonstrate the leachability of any SWMU specific contaminant that was identified during initial excavation of the soil used to create the windrow. Toxicity Characteristic Leaching Procedure (TCLP) will simulate the leachability of contamination expected in a sanitary landfill while Synthetic Precipitation Leaching Procedure (SPLP) will simulate the leachabililty of contamination expected in a natural environment. The limits of leachability for all SWMU contaminants are defined in Section 1.0 page 10 of 44 Table 1-4C of this QAPP.

TABLE G-2 TOXICITY AND LEACHABILITY OBJECTIVE SUMMARY			
Test and Test Method	**Result to be Obtained**	**Objective**	**Result will demonstrate**
Microtox (Proprietary method of AZUR Environmental)	EC-50 for each sample and average EC-50	Average EC-50 less than EC-50 of control pile	Compost piles are no more toxic than uncontaminated soil from a borrow
Earthworm Toxicity (EPA Method 600/3-88/029)	LC-50 for each sample and average LC-50	Average LC-50 less than LC-50 of control pile	Compost piles are no more toxic than uncontaminated soil from a borrow
Pathogen Testing (40 CFR 503.32)	Fecal Coliform Salmonella	Less than 1000 Most Probable Number/g dry solids Less than 3 MPN/4g dry solids	Compost piles are no more toxic than sewage sludge
Toxicity Characteristic Leaching Procedure (TCLP) (EPA Method 1311) and Explosives Analysis (EPA Method 8330)	TCLP results for all SWMU specific contaminants	Less than leachate limits presented in Table 1-4C in Section 1.0 of the QAPP	Compost piles do not show leachability greater than Regulatory Limits
Synthetic Precipitation Leaching Procedure (SPLP) (EPA Method 1312) and Explosives Analysis (EPA Method 8330)	SPLP results for all SWMU specific contaminants	SPLP results less than leachate limits presented in Table 1-4C.	Compost piles do not show leachability greater than Regulatory Limits

4.0
QUALITY CONTROL PROCEDURES

4.1 FIELD QUALITY CONTROL

A small control compost windrow will be constructed for each SWMU using the same amendment recipe as the full scale windrows. Clean, analyzed soil from a documented borrow source selected to be of similar soil characteristics to the contaminated soil will be used to construct the control windrow which will be approximately 10 x 10 x 5 feet high located in the same building as other windrow piles. This windrow will be constructed during the first set of full-scale windrows and maintained to simulate the full-scale windrows. This windrow will be turned at the same frequency, temperature, oxygen, and moisture content of the control windrow will be measured and recorded at the same frequency as the full-scale windrows. This control will be utilized to aid in the determination of background toxicity levels as well to serve as a guide to determine if the amendment ratios are toxic. In addition, the control pile will be analyzed for explosives at the end of cycle to verify that there is no cross contamination from other windrow piles. End of cycle for control pile will be the same number of days that the pile chosen for toxicity sample analysis was processed. Other QC field procedures for decontamination of equipment is detailed in Section 5.0 of Appendix G of the *Full-Scale Operational Plan* [MK, 1998]

The collection of field blanks, rinse water samples, and field duplicates will be performed at the same frequency as other compost samples. The specifications for compost quality control is specified in Section 4.6 on page 8 through 12 of 18 of the QAPP.

4.2 ANALYTICAL TESTING QUALITY CONTROL

Detailed standard operating procedures for each analytical test are included in Attachment A of this Appendix. These procedures outline the specific quality control measures required by the analytical method.

4.2.1 Earthworm Toxicity

Earthworm analysis will be performed using the test organism, *Eisenia Foetida* supplied from Carolina Biological Supply Company or other documented source. The condition and environment of the worms will be documented on the livestock record from receipt in the laboratory until use in this project. Worms used in this project will not have been previously submitted to any other testing. The worms will be uniform in size, at least 60 days of age, and weigh approximately 300 - 500 mg. Worms will be maintained at a temperature of 20 $\pm$ 2°C and acclimated for a period of five days prior to testing. During this time worms will be fed with Magic Worm Food. Feedings will be documented on the livestock record. Initiation of the test will begin after the five day stabilization has occurred. Ten worms will be placed in the testing vessels by sequential randomization, where no more than 2 worms will be added into each vessel at a time. Testing vessels will be one liter borosilicate glass containers with screw top

covers with small holes punched in the covers to allow air to enter exposure vessels. During the duration of the analysis, the worms will not be fed.

Each vessel will contain 200 grams of testing material (compost/artificial soil). Artificial soil prepared from a homogenous mixture of 10% sphagnum peat, 20% colloidal kaolinite clay and 70% grade 70 silica sand will be used as a blank and also as a dilutant to the compost mixture. Five varying concentrations of artificial soil and compost will be homogenized using a grinder. The concentration ranges of 6.25%, 12,5%, 25%, 50%, and 100% will be created for this analysis. In addition, three replicates for each concentration of compost will analyzed be performed to determine the LC-50 (the compost concentration that kills 50% of the worms). The LC-50 will be calculated with a computer program developed by C.E. Stephans and ASTM.

Additional QC includes the use KCl as a reference toxicant. The KCl will be utilized as a positive control to evaluate the sensitivity of each batch of organisms. Varying concentrations of KCl contaminated soil will be prepared to determine the LC-50 of the KCl contaminated soil. This value will represent the concentration of KCl which results in 50% mortality. The LC-50 will be compared to published acceptance limits as well as previous laboratory data to verify that the worms reacted at similar sensitivities as other batches of *Eisenia Foetida.* The artificial soil prepared in the lab will also be analyzed to verify that less than 10% of the worms died in the blank. These QC checks will be analyzed with three replicates for each concentration also.

Initially, and again at the completion of this analysis, the pH of the testing material will be recorded for each testing vessel. The pH will be documented and measured with a calibrated meter. The temperature of the test will be monitored continually during the analysis. The temperature should remain in the window of 22± 2 degrees Centigrade and will not fluctuate more than 3 degrees in any twenty four hour period. Testing temperatures will be documented and measured with a thermometer that has been calibrated to an ASTM traceable thermometer. Earthworm analysis will be performed by Aqua Survey, Inc.

4.2.2 Pathogen Analysis

Pathogen analysis will be performed according to the laboratory standard operating procedure. Each sample is prepared with three dilution series, each with five replicate tubes for Salmonella and four dilution series each with five replicate tubes for Fecal Coliform as stated in the laboratory SOP. All data from the various replicates will be evaluated from the raw data. The pathogen analysis will include an empty bottle shipped to the site and returned as a blank control to verify that contamination has not occurred during transport. Pathogen analysis will be performed by Pure Earth Environmental Laboratory.

4.2.3 Microtox Analysis

The EC-50 value will be determined as the concentration of elutriate compost material which causes the effect of a 50% decrease in the light output of the microtox reagent organisms created from a strain of the marine bacterium, *Photobacterium phosphoreum.* The microtox test will be performed according to the specifications of

the proprietary method from Azur Environmental with the assistance of a computer program from Azur. The microtox test will utilize a blank to be analyzed with each assay. The blank will have a ratio of 0.75 or greater. A computer program will be utilized to determine the EC-50. This value must fall within the range of the test concentrations, and the confidence limits must closely bracket the EC-50 value. In addition, the correlation coefficient of the five data points must be greater than 0.9. If any of these values fail, the assay will be re-analyzed. Microtox tests will be performed by Aqua Survey, Inc.

4.2.4 TCLP and SPLP Analysis

The TCLP and SPLP test will utilize a blank for every twenty extractions that are conducted in each extraction vessel. In addition, a matrix spike will be performed with each analytical batch. Matrix spike recoveries will be compared to the analytical procedure for limits for acceptability. Both TCLP and SPLP procedures will be performed by Southwest Laboratories.

5.0
LABORATORY INFORMATION

5.1 STANDARD OPERATING PROCEDURES

Refer to Attachment A of this Appendix for the standard operating procedures for each test performed for the toxicity analysis.

5.2 LABORATORY SHIPMENTS AND CONTACTS

Samples for the analysis of TCLP and SPLP will be shipped to Southwest Laboratories at the following location:

Mr. Keith Sims
Sample Receiving
1700 West Albany
Broken Arrow, OK 74012-1421

Samples for the analysis of Microtox and Earthworm Toxicity will be shipped to Aqua Survey, Inc., at the following location:

Mr. James Todd
Sample Receiving
499 Point Breeze Road
Flemington, NJ 08822

Samples for the analysis of Pathogens will be shipped to Pure Earth Environmental Laboratory at the following address:

Dr. Ted Passon
Sample Receiving
7184 North Park Drive
Pennsauken, NJ 08110

6.0
REFERENCES

MK, 1998. *Full-Scale Operational Plan* - Soils Bioremediation Facility - NSWC Crane, Crane, Indiana. Delivery Order Number 0009, Contract Number N62467-93-D-1106. Prepared by Morrison Knudsen Corporation, March 12, 1998.

ATTACHMENT A
LABORATORY STANDARD OPERATING PROCEDURES

STANDARD OPERATING PROCEDURE

SOP NO. SOP/PRP/004
REVISION NO. 002*
COPY NO. Original
DATE 6/29/98
* Modified for project #4324-0009-07-35

TITLE: **PREPARATION OF SEDIMENT POREWATER SAMPLES FOR USE IN TOXICITY TESTS OR MONITORING POREWATER AMMONIA IN CONCENTRATIONS IN TEST SEDIMENTS**

CONTENTS **PAGE**

I. **Objective** 1
II. **Materials and Equipment** 2
III. **Procedure** 2
Preparation 2
Procedure 2
Appendix I Benchsheet. 4

I. OBJECTIVE

The objective of this procedure is to provide a standard method for the preparation of porewater samples for use in toxicity tests, also for monitoring porewater ammonia concentrations during purging for solid phase toxicity tests. (See SOP/SED/purging tests SOPs.)

II. MATERIALS and EQUIPMENT

1. Centrifuge jugs, quantity according to need.
2. Sorvall RC-5B Centrifuge with rotor models GSA and HS-4
3. Top load balance (readability ± 0.1 g)
4. 250 mL polycarbonate centrifuge bottles (Thomas #2625 - G40)

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III. PROCEDURE

1. Preparation

1.1 If the porewater extraction is for a toxicity test, the sample can be taken directly from the receiving container. If necessary, the sample can be the supernatant prepared for an elutriate test according to SOP/PRP/005

1.2 If the porewater extraction is for the purpose of monitoring concentrations of ammonia in the porewater, the sample(s) must be treated as regular test samples and allowed to settle overnight before monitoring is initiated. A set of "dummy" chambers are set-up for monitoring purposes.

1.3 If standing water is present in the sample, pour this water off before mixing. Completely homogenize the sample using a spoon, plastic rod, stainless steel trowel or equivalent.

1.4 Determine the amount of porewater sample required. Assuming approximately 25% water, calculate the amount of sediment needed to spin down. If moisture values are available these may be helpful.

2. Procedure (Consult SOP/INS110 for detailed centrifuge operation)

2.1 Fill the centrifuge bottles with compost and weigh to the nearest 0.1 gram with a top loading balance (or equivalent). Ensure that the same mass is placed in the opposing jugs so that the rotor remains balanced.

2.2 Place cups in the rotor.

2.3 Tighten down rotor cover, close door and set temperature to the test temperature.

2.4 Spin at 2480 rpm for 30 minutes with rotor GSA or 3225 rpm for 30 minutes with rotor HS-4.

2.5 Remove the samples from the centrifuge.

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2.5.1 If the sample is for toxicity testing, remove sample from the centrifuge and pour off the supernatant into a suitable container. The sample must be used the same day that it is prepared, but if it is not to be used immediately store at the test temperature.

2.5.2 If the sample is for monitoring sediment porewater ammonia, place 10 mL into a small sample cup and measure the ammonia concentration immediately. The remaining supernatant sample is used to determine porewater salinity and pH.

VI. APPROVAL SIGNATURES

Prepared By G. Stephen Hornberger — 12/3/97
G. Stephen Hornberger — Date

Approved By: _____ — 12/17/97
James Todd
Vice President — Date

Approved By: _____ — 12/17/97
York Terrell
Laboratory Manager — Date

Approved By: _____ — 12/17/97
Jane Norvell
Quality Assurance Office — Date

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DETERMINATION OF WATER HOLDING CAPACITY OF SOILS AND SEDIMENTS

OBJECTIVE

The objective of this procedure is to provide a standard methodology for the determination of water holding capacity of soil and sediment samples.

MATERIALS

1. Weigh boats
2. Drying oven
3. Crucible tongs or flat-tipped forceps
4. Desiccator
5. Drierite® or similar drying agent
6. 250-mL glass beakers
7. Triple Beam balance
8. Glass stir rod
9. 100-mm diameter x 95-mm overall length glass funnels
10. 185-mm diameter, coarse porosity, qualitative crepe filter paper
11. 500-mL Erlenmeyer flasks
12. Aluminum foil

PROCEDURES

1. Preparation

1.1 Prepare and label a set of weigh boats equal in number to the number of samples to be analyzed.

1.2 Place the weigh boats in a 100 ± 5° C drying oven for a minimum of 2 hours. From this point on, DO NOT manipulate the weigh boats by hand as natural skin oils will adhere to the boats and bias the weights.

1.3 Using a crucible tongs or flat-tipped forceps, transfer the boats from the oven to a desiccator containing fresh drying agent. Allow to cool to room temperature.

1.4 Calibrate the Triple Beam balance as per SOP/INS/01 .

1.5 Weigh enough sample into the weigh boats to allow a final sample dry weight of at least 100 g. The amount of sample necessary will depend on the particle size and degree of hydration of the samples.

1.6 Using a crucible tongs or flat-tipped forceps, transfer the weigh boats with sample to the drying oven. Dry the samples at 100 ± 5° C for 24 hours.

1.7 Using a crucible tongs or flat-tipped forceps, remove the samples from the oven to the desiccator and cool to room temperature.

1.8 Calibrate the Triple Beam balance as per SOP/INS/01 .

1.9 With a forceps, remove the weigh boats with sample from the desiccator. Determine and record, as the final dry weight, the weight of each sample to the nearest gram.

2. Rehydration and Analysis

2.1 Weigh 100 g dried sample into a 250-mL glass beaker.

2.2 Measure 100 mL deionized water into the beaker. Thoroughly mix the sample and water with a glass stir rod, ensuring that all sample particles are wetted, to create a homogeneous slurry.

2.3 Fold a circle of filter paper in half and then in half again. Pull open one side of the folded paper to form a cone.

2.4 Set the cone of filter paper into a glass funnel. The edges of the paper shpuld be even with the top of the funnel walls.

2.5 Using a pipet, slowly add 9 mL deionized water to the filter paper so as to wet its entire surface.

2.6 Determine and record, to the nearest 0.1 g, the combined weight of the funnel with the wetted paper. Add the weight of the dried sample (100 g) to the weight of the funnel and paper and record as the initial weight.

2.7 Set the funnel into the mouth of an Erlenmeyer flask. Slowly add the slurry of sample to the filter paper and rinse into the funnel any sample remaining in the beaker or on the stir rod with deionized water. Use as little rinse water as necessary.

2.8 Cover the funnel tightly with aluminum foil and allow the sample to drain for 3 hours at room temperature.

2.9 Determine and record, to the nearest 0.1 g, the weight of the funnel and sample as the final weight.

3. Calculation of Water Holding Capacity

3.1 To determine the sample water holding capacity as mL water per 100 g sample, subtract the initial weight from the final weight, as per the following example:

- Water Holding Capacity (mL/100 g) = [Final Weight] - [Initial Weight]
- Water Holding Capacity (mL/100 g) = [297] - [115]
- Water Holding Capacity (mL/100 g) = 182

STANDARD OPERATING PROCEDURE

SOP NO. SOP/PRP/005
REVISION NO. 000
COPY NO. ~~Original~~ 401-7
DATE 07/25/94

TITLE: PREPARATION OF SUSPENDED PARTICULATE PHASE ELUTRIATES FOR USE IN TOXICITY TESTS

CONTENTS

		PAGE
I.	Objective	1
II.	Materials and Equipment	1
III.	Procedure	2
IV.	Miscellaneous	2
V.	Approval Signatures	3
VI.	Appendix	

I. OBJECTIVE

The objective of this procedure is to provide direction for the preparation of elutriates for use in toxicity tests. This procedure is primarily designed for use in the dredge sediment program under the NYACE but can be modified for use in other areas.

II. MATERIALS AND EQUIPMENT

1. New HDPE mixing chambers, sized according to need
2. Stainless steel mixers (for larger volumes)
3. Stir Bars (for small volumes)
4. Stir Plate
5. Site water, near subsurface, quantity according to need
6. Lab coat, gloves, glasses. Nitrile gloves and tyvek for dioxin.

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A-3d

III. PROCEDURE

1. Obtain homogenized and composited test sediment.

2. Obtain site water, near subsurface.

3. Bring both to room temperature.

4. Mix together, 4 parts water to 1 part sediment, by volume. Break up clods and clay balls by hand, remove large debris and discard appropriately.

5. Mix with stir bar and plate, or stainless steel mixer, as appropriate for 30 minutes. Allow to settle for at least one hour.

6. Decant carefully to another clean HDPE container, avoid pouring settled sediment.

7. This mixture must be used the same day it is prepared.

IV. MISCELLANEOUS

Not applicable at this time.

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A-3e

V. APPROVAL SIGNATURES

Prepared By: _____ Date 7/25/94
Kenneth R. Hayes
Laboratory Director

Approved By: _____ Date 7/25/94
W. Scott Douglas
Project Manager

Approved By: _____ Date 7/25/94
James M. Todd
Director of Operations

Approved By: _____ Date 7/25/94
Jane Norvell
Quality Assurance Officer

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AQUA SURVEY, INC.
499 Point Breeze Road
Flemington, NJ 08822

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PROTOCOL: The Acute Soil Testing Toxicity Using the Earthworm, *Eisenia foetida*.

TEST SUBSTANCE: Soil collected from NSWC Crane

ASI NO. 98164

APPROVED BY: _____ _____
STUDY DIRECTOR DATE

APPROVED BY: Ala M Tosdick 9/21/99
SPONSOR DATE

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Approved: York Terrell — 9-15-99
York Terrell
GLP Testing Manager
Date

Approved: Jane Dowell — 9/16/99
Quality Assurance Unit
Date

Approved: — 9/16/99
James Todd
Vice President
Date

THE ACUTE SOIL TOXICITY TEST USING THE EARTHWORM, *EISENIA FOETIDA*.

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I. Purpose/Objective

This test is designed to determine the acute effect of contaminated soils on the earthworm, *Eisenia foetida*. Test concentrations will be prepared by the addition of the test soil to artificial soil. The concentration of the test substance which is lethal to half of the organisms in a concentration (LC_{50}) series during a 7-day and 14-day exposure will be calculated from observations of mortality. When no adverse effects are noted below a test concentration, the results are also expressed as a highest acute No Observed Effect Concentration (NOEC). The information from this response test can be used to predict the likelihood of an ecosystem effect if the test substance enters the terrestrial environment.

II. Justification

Evaluating contaminants for their adverse impact on the terrestrial environment is an important component of environmental hazard assessments. The present procedure evaluates the potential of contaminated soils to cause acute (14 days or less) adverse effects on the earthworm, *Eisenia foetida*. This potential is expressed as a 7-day LC_{50} and a 14-day LC_{50} value and/or as the highest acute No Observed Effect Concentration (NOEC). The effect of the test substance on the earthworm is considered indicative of its potential to affect other terrestrial organisms. The procedure described herein is similar to standard and/or generally acceptable procedures such as those described by the US FDA[1], ASTM[2], US EPA[3], OECD[4] and APHA/AWWA/WPCF[5].

III. Test Administration

A. Testing Facility

Aqua Survey, Inc.
499 Point Breeze Road
Flemington, NJ 08822

B. Sponsor

Morrison Knudsen Corporation
1500 West Third Street
Cleveland, OH 44113-1406

C. Dates of Experiment

Estimated Test Substance Exposure Initiation Date:

Estimated Test Substance Exposure Completion Date:

Estimated Study Completion Date:

D. Test Characteristics

1. ____ Range-Finding/ __X__ Definitive

2. __X__ Nominal/ ____ Measured Test Substance Concentrations

4. __3__ Number of Replicates

IV. Test and Control Substances

A. Test Substance

1. Identity:

2. ASI Number:
Batch/Lot Number:
CAS Registry No.:

B. Control Substance(s)

1. Negative Control: Untreated artificial soil

2. Reference Toxicant Test: A reference toxicant test with potassium chloride (KCL) will be performed concurrently with the test substance exposures.

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V. Test System

A. Test Organism

1. The earthworm, *Eisenia Foetida*, will be used for this test; only adult worms will be used. Adult worms weigh approximately 300 milligrams (mg) wet weight and have a distinct, fully developed clitellum.

2. Source of organism: Earthworms used for these tests will be obtained from a commercial supplier. An organism Chain-of-Custody, as well as any culturing procedures, accompany each batch of organisms and are kept on file in the archives of Non-Test Data. In addition, the receipt date, lot or batch number, condition of shipping container, worms, temperature and soil, as well as taxonomic verification of the test species, will be documented upon receipt of organisms. The following taxonomic characterization applies to the earthworm, *Eisenia foetida*:

Phylum - Annelida
Class - Oligochaeta
Order - Lumbricina
Family - Lumbricidae
Genus - *Eisenia*
Species - *foetida*

3. Rearing and Maintenance Regime: Earthworms are reared within the laboratory in plastic trays measuring approximately 34 by 28 by 14 cm. Each tray is provided with a bedding of sphagnum peat moss pH adjusted to approximately 7.0 with pure calcium carbonate (99%). Seven hundred grams (dry weight)of peat moss is added to each tray and hydrated to approximately 75% of its water holding capacity with approximately 2300 mL of deionized water. The number of earthworms that can be maintained in each tray is determined by the age and size of the worms. Seven hundred grams of bedding holds a population of approximately 350 adult worms. The trays are covered with plastic to prevent drying, however, several 1/8" air holes are punched into the plastic for air circulation. Moisture content is monitored on a weekly basis and hydrated water is added as needed. Bedding is changed every 2-3 months. Fluorescent lighting above the trays provides illumination (50-100fc) on a continuous basis. Bedding temperature is maintained at 20 ± 2°C.

Organisms are observed for signs of disease, stress, injury and mortality once each week by hand turning the soil. All observations and operations such as feeding and maintenance procedures and schedules, clinical observations, mortality and critical rearing parameter (e.g., temperature, pH, etc.) are documented in culture systems log books.

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4. Diet: Earthworms are maintained on a diet of magic worm food, obtained from Magic Products, Inc., Amhurst Jct., Wisconsin, 54407. Food is prepared as a slurry by adding deionized water to the magic worm food using approximately a 1:1 v/w ratio. This slurry is applied to the top of the bedding of rows of about 1/8" thick, using a 60 mL disposable syringe. The cultures are usually fed twice each week but if the number of worms in a tray is low, feeding is reduced accordingly. Food remaining on the surface of the bedding from a previous feeding is removed and discarded. An estimate of the amount of food discarded will be used in deciding what level of feeding is adequate for successful growth of earthworms.

5. Acclimation: Organisms are held at test conditions (e.g., temperature, pH, illumination) during culture so that a period of acclimation is not necessary. However, if worms are obtained from an outside source just prior to testing, that will be held at test conditions for a minimum of 5 days.

B. Artificial Soil

1. Source: Soil used as substrate in this test will be artificial. It has an adsorptive capacity resembling typical loamy soils. The substrate is prepared in the laboratory from a mixture of commercially available constituents. The following constituents are mixed on a dry weight basis, to produce the artificial soil: 10% sphagnum peat moss (that portion passing through a 2.36 mm screen); 20% Kaolin clay (greater than 50% kaolinite with particle sizes under 40 mm): 70% silica sand (97.1% particle size of 0.053 - 0.300 mm). After the sphagnum, clay and sand are mixed, calcium carbonate equal to 0.39% of the weight of the mixture is added to adjust the pH to 6.5 ± 1.5 su at which time the artificial soil is homogenized in a 5-gallon plastic bucket, using a hand drill fitted with a stainless steel mixing blade.

2. Quality: The use of artificial soil virtually eliminates the problem of variability between different natural soil types and should contain no contaminants that might interfere with the outcome of the study.

VI. Test Procedure

A. Preparation of Test Concentrations

Test soils are screened through a one-quarter inch stainless steel screen. The screened soil is then mixed in a 5-gallon plastic bucket using a hand drill filtered with a stainless steel mixing blade until the sample is fully homogenized. Seven hundred grams of each test concentration is prepared by adding appropriate amounts of test soil with artificial soil in a geometric series in which the ratio is 2.0 (e.g., 100, 50, 25, 12.5 and 6.25 dry weight/dry weight). Negative controls (100% artificial soil only) are also prepared. To ensure even distribution of the test

soils with artificial soil, the total amount (700g) for each concentration is homogenized using a blender. Two hundred grams of soil (dry weight) from each concentration is used for each of three replicate test concentrations and control, at which time the soil is hydrated to 75% of its water holding capacity using deionized water. Hydration is accomplished by using the moisture fraction and water holding capacity or both the test and artificial soil, then using a computer program developed by this laboratory to calculate the required volume of hydrated water. The computer program requires the following information:

THW_{ts} (test soil water hydration required in mL/100g) = PHYD [(PAS*WHC_{as}) + (PWS*WHC_{ws})

EHW_{ts} (existing test soil hydration water in mL/100 g) = [(PAS*MF_{as}) + (PWS*MF_{ws})] X 100

Amount of hydration water to be added (mL/100 g of soil) = THW_{ts} - EHW_{ts}

Where PHYD = proportion of hydration required (constant = 0.75)
PAS = proportion of artificial soil in test soil
WHC_{as} = water holding capacity of the artificial soil in mL/100 g
PWS = proportion of waste sample in the test soil
WHC_{ws} = water holding capacity of the waste sample in mL/100g
MF_{as} = moisture fraction of the artificial soil
MF_{ws} = moisture fraction of the waste sample

B. Soil Moisture Content and Water Holding Capacity

The moisture content of soil is determined by pre weighing an empty aluminum weigh boat or crystallizing dish. Appropriate amount of soil is placed in the weigh boat in order to obtain a minimum of 100 g dry weight soil. The combined weight of the boat and sample equal the initial wet weight and is recorded to 0.1 g. The wet weight is calculated by subtracting the weight of the empty weigh boat from the combined weight. The weigh boat and sample are placed in a drying oven at 100±5°C for 24 hours at which time it is removed and allowed to cool in a desiccator. The weight of the boat and the dried sample are recorded as the final dry weight. The moisture content of the sample is determined according to the following formula:

$$\text{Percent moisture} = \frac{(\text{wet weight + pan (g) - dry weight + pan})}{(\text{Wet weight + pan (g) - pan})} \times 100$$

After the moisture content has been calculated the water holding capacity is determined by placing 100 G) of dry soil into a 500-mL glass beaker. One hundred mLs of deionized water is added to the sample and mixed thoroughly with a glass rod to ensure that all particles are wet and a slurry of soil and water exists. A circle of coarse porosity, qualitative, crepe filter paper is folded into eighths and placed in a 100-mm glass funnel. The filter paper is moistened with approximately 10 mL deionized water at which time the weight of the funnel including the wet filter paper is determined. Next, the slurry of soil and water is poured in the funnel; all

remaining soil in the beaker and on the stirring rod is rinsed into the funnel with deionized water. The funnel is covered tightly with aluminum foil and allowed to drain for a minimum of three (3) hours at room temperature. The time of drainage will be documented.
After the drying period the foil is removed and the funnel with drained soil is weighed to find the final weight. The final weight minus the initial weight is the number of mLs of water held by the soil. The water held, when related to the amount of soil, is the water-holding capacity and is expressed as g water/100g dry weight soil.

C, Preparation of Test Vessels

One liter borosilicate glass containers with screw top covers with small holes punched in the covers to allow air to enter the exposure vessels. The side of each replicate jar is labeled with the site soil concentration and the replicate jar number. Permanent felt-tip markers are used for labeling.

D. Collection of Organisms for Testing

Earthworms of relatively uniform size and age are collected from a single, established stock culture and transferred to intermediate holding vessels. Sequential randomization is accomplished by allocating to each holding vessel no more than 20 percent of anyone set of test organisms at a time.

E. Number of Organisms

Ten (10) organisms per replicate test vessel will be used at each concentration and for each control. All controls and samples will be analyzed in triplicate.

F. Characterization of Size, Age, or Absorption of Test Substance

No attempt is made to characterize the test organisms as to exact age or sex. Adult earthworms which weigh approximately 300 mg wet weight and have a distinct fully developed clitellum will be used to characterize the age of the worms. The degree of absorption of the test substance by the test system will not be determined in this test. Mean weights will be determined by weighing a surrogate container of earthworms from the same lot as used for testing. Average weights are calculated and these values are used as mean test population characteristics. No other weights will be determined during this study.

G. Test Endpoint

The 14-day LC_{50} with its associated 95 percent confidence limits, and, when sufficient mortality data are noted, the 7-day LC_{50} values and their confidence limits, will be calculated. The LC_{50} value is the median lethal concentration (i.e., the concentration, in terms of initial or measured values, of test substance in substrate that results in the death of 50 percent of the test organisms within a specified time interval (e.g., 14 days). Death is defined as lack of observable movement

by a test organism when gentle mechanical stimulation is applied to the anterior end (FDA[1]). A computer program developed by C.E. Stephan[6] and ASTM[7] will be used to calculate the LC_{50} value and the associated confidence limits for each concentration series as described in Section VIII.

When possible, the no observed effect concentration, or NOEC, (the highest concentration tested at which there were no observable effects) will be determined through visual inspection of the data. However, appropriate statistical analyses (i.e., analysis of variance [ANOVA], group mean comparison, etc.) may be used.

H. Test Duration

The test will be conducted for a period of 14 days.

I. Control Vessels

Artificial soil (negative control) vessels will be prepared, in addition a positive control containing KCl as a toxicant with artificial soil will be prepared. All positive and negative controls will be analyzed in triplicate.

J. Feeding

The organisms will not be fed during this test.

K. Loading

The biological loading should not exceed one (1) organism mass per 20 g of soil.

L. Temperature

The temperature is maintained at 20 ± 2°C via an environmental chamber and is continuously measured (hourly) using a Ryan tempmonitor®. Fluorescent lighting above sample vessels provides illumination (50-100 fc) on a continuous basis.

M. Soil pH/Adjustment

Initial pH values are measured and recorded on the Earthworm Survival Data Sheet. The pH values should fall within the range of 5.0 and 9.0 SU for initiation of the test. If the pH falls outside this range it may be necessary to adjust the pH within range as close to 7.0 as possible for the survival of the test organisms. pH adjustment may be done with 5N HCL or 5N NaOH. Other normality solutions may be used. If the soil needs to be pH adjusted, a 40-g slurry may be prepared and adjusted by adding 20g soil to 20 mL deionized water. The mixture is stirred for approximately 30 minutes at which time the mixture is allowed to settle, for about one hour. At this time the pH maybe measured from the supernatant. The pH is

adjusted by adding the appropriate volumes of HCL or NaOH to the mixture until the pH of the slurry is in an acceptable range. The volume of HCL or NaOH and the normality used to adjust the pH of the slurry are recorded and then used in calculating the amount needed for adjusting the pH of the calculated wet weight of soil needed for the test.

N. Reference Toxicant

A reference toxicant test with KCL will be performed to assess the adequacy of the test conditions and to monitor the sensitivity of the test organisms. A stock solution containing 50,000 ppm of KCL is prepared by adding 10 grams of KCL in a total volume of 200 mL using DI water. Test concentrations of 625, 1250, 2500, 5000 and 10,000 ppm is prepared by placing 200 grams dry weight of artificial soil in each of three replicate test vessels and control vessels. A magnetic stir bar is used to stir the KCL until it is completely dissolved. Appropriate volumes of stock solution (i.e., 2.5, 5.0, 10.0, 20.0 and 40.0 mL for the 625, 1250, 2500, 5000 and 10,000 ppm test concentration, respectively) is added as part of the total volume of water used to hydrate the artificial soil). The total volume of water used for hydration is determined as per section VI.A. The acceptable ranges for this reference toxicant is 4321 to 8081 ppm.

O. Mortality of Negative Control

Mortality or abnormal behavior in the 100% artificial soil should not exceed 10 percent.

P. Observations

Observations performed after 24 hours of exposure is optional but may be conducted to determine if earthworms are burrowing and if any deaths have occurred on the test soil surface. Lack of burrowing in the presence of continuous light is considered a behavioral response to the toxicants and will be noted.

After seven days of exposure, the number of live worms is counted and observations of behavioral and pathological changes are recorded. The test jars are removed from the environmental chamber and emptied one at a time on a counting tray. During the worm count detailed observations are made regarding: (1) number of percentage of worms that did not burrow, (2) ulceration (3) coiling (4) "balling" together of worms (5) contraction (6) rigidity (7) elongation (8) mid segmental swelling (9) segmental constriction and (10) segmental loss. All observations are recorded on the Test Effects Report forms. Each replicate is counted and the number of mortalities is entered on the live count sheet. A worm is considered dead if it does not respond to a gentle mechanical stimulus to its anterior end. Worms decay rapidly in the moist testing environment. If worms cannot be found they are considered to have died and decomposed completely. After worms are counted, the soil is replaced in test vessels and worms are placed on the surface of the soil. After 14 days of exposure the worms are observed and counted as per day seven. Negative and positive control worms are counted on day seven and 14 as per test organisms.

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VII. Chemical Analysis

A. Measurements of Physical and Chemical Parameters

1. Temperature: The temperature of the exposure environmental chamber should be held at 20 ± 2° C. Measurements should be made continuously (hourly) during the entire study period.

2. pH: The soil pH measured in water should be between 5 and 9 su. Measurements are made at test initiation and termination. If the initial pH of the soil falls outside the 5-9 range, soils pH may be adjusted using 5N NaOH or 5N HCL.

3. Moisture Content: Soil moisture content should be maintained at approximately 75 percent of water holding capacity for optimum condition. This is accomplished by covering the exposure vessels with lids after the initial hydration.

Physical and chemical measurements will be recorded on the Test Effects Report form.

VIII. Endpoint Calculation

A computer program developed by C.E. Stephan[6] and ASTM[7] is used to compute point and interval (i.e., 95 percent confidence interval) estimates of the 14-day LC_{50} value. The 7-day and 14-day values should be calculated whenever there are sufficient mortality data to determine such values. The program requires the following data: the concentration of the test substance, either nominal or measured (log_{10} transformed for all methods); the number of organisms exposed; and the number of organisms that die. Data for the positive KCl sample and the negative artificial soil sample vessels are not used because the program does not have the capability of adjusting for check standard mortality. The program computes point and interval estimates of the LC_{50} value using three approaches:

1. Nonlinear Interpolation, using the concentrations that bracket 50 percent mortality, to give an approximate point estimate; and the Binomial Method, to give a conservative 95 percent confidence interval. In the case of utilizing a single concentration, use of the Binomial Method is capable of indicating (at a conservative 95 percent confidence interval) whether or not the LC_{50} value lies above or below the test substance concentration.

2. The Moving Average Method, to give both a point and interval estimate. This output is included only when two consecutive concentrations exhibit an effect.

3. The Probit Method, to give both a point and interval estimate. This output is included only when two consecutive concentrations exhibit an effect. Additionally, when the number of test substance concentrations is at least three, a chi-square statistic is included to test the goodness-of-fit for the Probit Method.

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The no observed effect concentration (NOEC) will be determined through observation and when appropriate, statistical analyses (i.e., analysis of variance [ANOVA], group mean comparison, etc.).

IX. Criteria for Test Validity

A. Control Mortality

The negative control (100% artificial soil) mortality should not exceed 10 percent.

B. Mishaps

Abnormal occurrences (i.e., laboratory accidents) that might influence the outcome of the test will be considered in determining the validity of test results.

X. Reporting and Records

A. Reporting

The report should contain:

1. Name and address of testing facility.
2. Good Laboratory Practice compliance statement.
3. The Quality Assurance Inspection Statement.
4. Dates of Study.
5. Name of Study Director and list of scientists involved in the test.
6. Details documented in this protocol, details of solution preparation, all data, and any appropriate graphs or calculation summaries.
7. All observations and results as well as any protocol deviations.
8. The 14-day LC_{50} value, and when sufficient data have been generated, the 7-day LC_{50} values, the associated 95 percent confidence limits, and the method(s) of calculation.
9. When possible, the no observed effect concentration (NOEC).

B. Records

The following records will be maintained by ASI and are available upon request.

1. All raw data (or exact copies thereof) and summaries of data, final reports, protocols and amendments will be retained for a period of 5 years with a subsequent review.

2. All Standard Operating Procedures and all Summaries of Training and Experience for all scientists involved in the testing will be retained for a period of 10 years with a subsequent review.

3. The Archive of Non-Test Data shall contain:

   a. Taxonomic verification of the test species and chain-of-custody records.

   b. Chemical characterization of the substrate.

   c. Diet receipt records and characterization results.

   d. Rearing and maintenance records for the aquatic organisms.

   These records will be retained for a period of 10 years with a subsequent review.

XI. References

1. United States Food and Drug Administration (US FDA). 1987. Environmental Assessment Technical Assistance Handbook, PB87-175345, Document 4.12, "Earthworm Subacute Toxicity". March 1987.

2. American Society for Testing and Materials (ASTM). Annual Book of ASTM Standards. 1995. Section 11, Water and Environmental Technology. Volume 11.04: Pesticides; Resource Recovery; Hazardous Substances and Oil Spill Responses; Waste Disposal; Biological Effects. Standard Guide for Conducting A Laboratory Soil Toxicity Test with Lumbercid Earthworm, *Eisenia foetida* E1676-95; American Society for Testing and Materials, Philadelphia, PA, pp. 1125-1139.

3. United States Environmental Protection Agency (US EPA). 1988. Protocols for Short-Term Toxicity Screening of Hazardous Waste Sites (USEPA/600/3-88/024). A.8.5, Earthworm Survival. March 1988.

4. Earthworm, Acute Toxicity Tests, 1984. In OECD Guideline for Testing of Chemicals. No. 207, pp. 1-9.

5. American Public Health Association/American Water Works Association/Water Pollution Control Federation. 1989. Standard Methods for the Examination of Water and Wastewater, 17th ed. American Public Health Association, Washington, DC.

6. Stephan, C.E. 1977. Methods for Calculating an LC_{50}. In: F.L. Mayer and J.L. Hamelink, Eds., Aquatic Toxicology and Hazard Evaluation, Special Technical Publication No. 634, American Society for Testing and Materials, Philadelphia, PA, pp. 65-84.

7. American Society for Testing Materials. 1988. Proposed New Standard Practice for Using Probit Analysis. ASTM Subcommittee E-47.07. Draft No. 4, June, 1988.

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STANDARD OPERATING PROCEDURE

SOP NO.	TOX-1001
REVISION NO.	00
COPY NO.	Original
DATE	09/17/92

TITLE: MICROTOX® STANDARD ASSAY

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I. Introduction

The objective of this procedure is to determine the effective toxicity of a sample to the bioluminescent marine bacterium, *Photobacterium phosphoreum*, using the Microtox® test system. The concentration of the test substance which results in fifty percent (50%) inhibition of bioluminescence in a concentration series during a 15-minute exposure will be calculated from observations of light emission. When no observed effects are noted below a test concentration, the results are expressed as the highest acute No Observed Effect Concentration (NOEC). The information from this primary sensitivity test can be used to evaluate the likelihood of a secondary community effect if the test substance enters an aquatic environment.

The procedure described herein is similar to standard and/or generally accepted procedures such as those described by the Microbics Corporation[1,2,3].

II. Materials

1. Microtox® Model 500
2. Microtox® glass cuvettes

3. 10-μL automatic pipettor
4. 250-μL automatic pipettor
5. 500-μL automatic pipettor
6. 10-μL pipettor tips
7. 200/500-μL pipettor tips
8. Microtox® Reagent
9. Microtox® Reconstitution Solution (Recon Solution)
10. Microtox® Diluent
11. Microtox® Osmotic Adjusting Solution (MOAS)
12. Microtox® data reduction software

III. Test System

A. Test Organism

The Microtox® test system utilizes a specially developed strain of the bioluminescent marine bacterium, *Photobacterium phosphoreum*. The organisms are obtained as lyophilized cultures, each containing approximately one hundred million cells. Lyophilized cultures have a shelf life of one year when stored at minus 20° C.

B. Diluent

A standardized test system diluent, obtained from Microbics Corporation, Carlsbad, CA, is used for the Microtox® test system. Diluent is a two percent (2%) solution of sodium chloride in distilled water.

IV. Test Procedures

A. Preparation

Place a cuvette in the REAGENT well. Place a cuvette in each well of row A and of row B of the incubator block.

Using the 500-μL pipettor fitted with a clean pipettor tip, dispense 1 mL (1000 μL) Recon Solution into the cuvette in the REAGENT well.

Using the 500-μL pipettor, dispense 500-μL Diluent into each cuvette in wells B1 through B5.

Dispense 1 mL Diluent into each cuvette in wells A1 through A4. Do not add diluent to the cuvette in well A5.

Prepare the sample dilutions as follows:

1. Using the 250-µL pipettor fitted with a clean pipettor tip, dispense 250 µL MOAS into the cuvette in well A5. Discard the pipettor tip.

2. Using the 500-µL pipettor, add 2.5 mL (2500 µL) sample to well A5 and mix thoroughly by alternately aspirating and ejecting the sample with the pipettor.

3. Transfer 1 mL from A5 to A4 and thoroughly mix A4.

4. Transfer 1 mL from A4 to A3 and thoroughly mix A3.

5. Transfer 1 mL from A3 to A2 and thoroughly mix A2. Do not transfer from A2 to A1, as A1 will be the Blank, or control, for the assay. Discard the pipettor tip.

If the sample toxicity is unknown or is suspected of being high, perform a rangefinding assay. Where necessary, prepare a primary dilution of the sample in Microtox® Diluent and continue as in steps 1-5 above.

Allow five minutes for temperature equilibration.

Remove a vial of Reagent from frozen storage and quickly remove the seal and stopper from the vial, avoiding contact with the glass surface. If necessary, seat the reagent pellet on the bottom of the vial by gently tapping the vial.

The following functions MUST be performed as quickly and as carefully as possible to ensure the integrity of the reagent:

1. Remove the cuvette from the REAGENT well and, with the lip of the cuvette seated firmly on the rim of the vial, pour the contents of the cuvette into the Reagent vial.

2. Immediately swirl the contents of the vial, pour the reagent suspension into the cuvette and return the cuvette to the REAGENT well.

3. Using the 250-µL pipettor fitted with a clean pipettor tip, thoroughly mix the reconstituted Reagent suspension by alternately aspirating and ejecting. Discard the pipettor tip.

Using the 10-µL pipettor fitted with a clean pipettor tip, dispense 10 µL Reagent suspension into each cuvette in row B of the incubator block as follows:

1. Do not remove the cuvettes from the wells while dispensing Reagent.

2. Place the pipettor tip under the surface of the liquid but DO NOT set the tip on the bottom of the cuvette.

3. Rest the pipettor tip against the inside rim of the cuvette and slide the tip down until the ridge along its surface rests on the rim of the cuvette. Carry out pipettor functions with the pipette tip in this position.

Thoroughly mix the contents of the cuvettes in row B using one of the following methods:

1. Using the 250-μL pipettor fitted with a clean pipettor tip, aspirate and eject the contents of each cuvette 2 or 3 times; or

2. Working quickly, remove a cuvette from its well and, holding the cuvette firmly between thumb and index finger, tap the cuvette near its base with a second finger to create a vortex. Replace the cuvette in the well and continue until all cuvettes are mixed.

Wait 15 minutes for reagent stabilization.

B. Assay

Set a timer for 15 minutes but do not yet start the timer.

Take the cuvette from well B1 and set it in the READ turret well. Press the SET pad. Wait for the Ready green symbol to appear in the display. DO NOT press the SET pad again during the assay procedure.

Press the READ pad and wait for the light level, or "gamma", value to appear in the display. Read and record the value as the initial light level (I_0) for cuvette B1. Repeat for cuvettes B2, B3, B4 and B5, in that order.

Immediately start the timer.

Using the 500-μL pipettor fitted with a clean pipettor tip, make the following 500-μL transfers, mixing thoroughly after each transfer: From A1 to B1; from A2 to B2; from A3 to B3; from A4 to B4; from A5 to B5.

The resulting assay concentrations are, in respective order, 0.0 (Blank), 5.6, 11.3, 22.5 and 45.0 percent of the undiluted sample or primary dilution, as applicable. If the addition of MOAS was omitted, the resulting concentrations are 0.0, 6.3, 12.5, 25.0 and 50.0 percent of the primary dilution.

At 5 minutes and 15 minutes post timer initiation, read and record the light level readings for the 5-minute and the 15-minute exposures, respectively, from cuvettes B1 through B5.

Light readings may be determined for intervals other than, or in addition to, 5 and 15 minutes, as dictated by purpose. With samples of unknown toxicity, it is recommended that the 5-minute and 15-minute light values be determined.

Exposure to some toxicants, such as organic compounds, will result in immediate light loss followed by a period of recovery or stabilization. In such cases the 5-minute values prove most useful.

With other classes of toxicants, such as metals, the light loss from the bacterium may still be occurring, or has not yet begun at the 5 minute interval. In such cases, the 15-minute data may be more useful.

Reduce the data (see section VI., below).

C. Observations

All observations of organism light emission and any other observations will be recorded on a Microtox® Data Sheet or an equivalent data form.

V. Chemical Analysis

The test substance concentration will be determined by a suitable analytical technique unless specified otherwise in the study protocol. The analytical procedure will be described in the protocol specific for the test substance.

No measurements of test substance exposure solution parameters (e.g., temperature, pH, dissolved oxygen) will be made.

VI. Data Reduction

Working at a computer station, initiate the Microtox® data reduction program from the general menu by entering the program code. Refer to Appendix I for a complete set of sample program displays and output.

Upon initiating the program, the Master Menu Screen will be displayed. Choose the option, 4) INPUT DATA FROM KEYBOARD.

The program will prompt for a sample description. Enter a description consisting of at least the study number, sample identification and test date.

The program will then prompt for Blank values. Enter the initial (B0) and the final (BT) light readings for the Blank, (cuvette B1).

The program will then prompt for sample treatment data. Enter the initial (I0) and the final (IT) light readings for each sample concentration, in ascending concentration order, in accordance with the format in the display.

The normalized data will be displayed along with a prompt to continue. The ensuing display will reveal the results of the data reduction and a second prompt to continue.

The MICROTOX CALCULATIONS MENU will be displayed. To initiate a printout of the data analysis, choose option 2.....HARD COPY. The information will be stored in the printer.

Instructions for printing a graph of the reduced data will appear in the display along with a prompt to produce and display the graph. Initiate the procedure and the graph will be created in the display. Use the print screen key to print the graph and then press ENTER. A hard copy of the reduced data and the graph will be produced.

The calculations menu will appear in the display. If another data set is to be reduced, choose the option, 6) RUN NEW DATA SET, and repeat Section 3. If no more data is to be analyzed, enter E to exit the program, and return to the general menu.

Examine the data printout for the following:

1. Blank Ratio - This should be 0.75 or greater. If the ratio is less than 0.75, it is an indication of poor reagent quality or poor reagent transfer technique. Blank ratio is calculated as BO/BT.

2. EC 50 - The EC 50 value should fall within the range of the test concentration values. If the EC 50 value falls outside of the range of concentrations, the accuracy of the test results becomes questionable.

3. EC 50 Confidence Limits - Found in parentheses to the right of the EC 50 value, the confidence limits express the range of precision of the reported EC 50. As such, they should closely bracket the EC 50.

4. R Value - The R Value indicates the degree to which the distribution of the data points correlates to a straight line, and should be as close to a value of 1.0 as possible. If the value is less than 0.9, it is an indication of a poor correlation.

If any one of the above parameters appears questionable, the assay should be repeated.

VII. Responsibility

The name of the person primarily responsible (and that of an alternate) for ensuring adherence to all procedures described in this SOP are documented in records maintained by the Unit Leaders. These records are currently located in the central file in the main office of ASI.

VIII. References

1. Microbics Corporation. 1985. How to Run Toxicity Tests Using the Microtox® Model 500. Microbics Corporation, Carlsbad, California.

2. Microbics Corporation. 1989. Microtox® Data Capture and Reduction Program, An Interim Manual. Microbics Corporation, Carlsbad, California.

3. Microbics Corporation. 1991. Microtox® Manual, Model 500 Toxicity Test System. Microbics Corporation, Carlsbad, California.

Original Issue: 09/17/92

APPENDIX I

Data Reduction Software Displays and Printout

Display One

```
                    MICROBICS CORPORATION

     MASTER MENU SCREEN

1)   SELECT PROCEDURE.
2)   REVIEW/CHANGE PARAMETERS.
3)   START TESTING.
4)   INPUT DATA FROM KEYBOARD.
5)   DISPLAY PARAMETER FILES.
6)   TIMER.
7)   SOFTWARE TEST.
8)   EXIT PROGRAM.

     PLEASE ENTER CHOICE:
```

Display Two

MICROTOX CALCULATIONS

ENTER SAMPLE DESCRIPTION, DATE, ETC. (ONE LINE ONLY):

Display Three

```
                    MICROTOX CALCULATIONS

ENTER SAMPLE DESCRIPTION, DATE, ETC. (ONE LINE ONLY):
SAMPLE RUN, DATA REDUCTION PROGRAM

ENTER BLANKS -- BO,BT

? 100,88
```

Display Four

```
                    MICROTOX CALCULATIONS

ENTER SAMPLE DESCRIPTION, DATE, ETC. (ONE LINE ONLY):
SAMPLE RUN, DATA REDUCTION PROGRAM

ENTER BLANKS -- B0,BT

? 100,88

BLANK RATIO=    .88

ENTER DATA -- CONCENTRATION, I0, IT
(enter 0,0,0 as prompt when finished)

?
```

Display Five

```
                    MICROTOX CALCULATIONS

ENTER SAMPLE DESCRIPTION, DATE, ETC. (ONE LINE ONLY):
SAMPLE RUN, DATA REDUCTION PROGRAM

ENTER BLANKS -- B0,BT

? 100,88

BLANK RATIO=    .88

ENTER DATA -- CONCENTRATION, I0, IT
(enter 0,0,0 as prompt when finished)

? 5.6,98,74
? 11.3,99,66
? 22.5,98,53
? 45,97,41
? 0,0,0
```

Display Six

SAMPLE #	CONCENTRATION	GAMMA
1	5.6	0.165
2	11.3	0.320
3	22.5	0.627
4	45	1.082

TO CONTINUE PRESS ANY KEY

Display Seven

SAMPLE RUN, DATA REDUCTION PROGRAM

PAIR #	CONC.	G-OBS	G-EST
1	5.600	0.165	0.169
2	11.300	0.320	0.319
3	22.500	0.627	0.597
4	45.000	1.082	1.120

EC 50 = 39.652 (33.629 TO 46.755)

R =0.99898 SLOPE = 1.0981 INTERCEPT = +3.6802

TO CONTINUE PRESS ANY KEY

Display Eight

```
MICROTOX CALCULATIONS MENU

1.....REPEAT RESULTS
2.....HARD COPY
3.....DELETE DATA POINTS
4.....ADD DATA POINTS
5.....ABSORBANCE CORRECTIONS
6.....RUN NEW DATA SET
7.....PLOT DATA
8.....CALCULATE ECXX
9.....RETURN TO MAIN MENU

PLEASE ENTER CHOICE  (E TO EXIT)
```

Data as Sent to Printer

SAMPLE RUN, DATA REDUCTION PROGRAM

PAIR #	CONC.	G-OBS	G-EST
1	5.600	0.165	0.169
2	11.300	0.320	0.319
3	22.500	0.627	0.597
4	45.000	1.082	1.120

BLANK Bo/Bt= 100 / 88
BLANK RATIO= 0.8800

EC 50 = 39.652 (33.629 TO 46.755)

R =0.99898 SLOPE = 1.0981 INTERCEPT = +3.6802

Display Nine

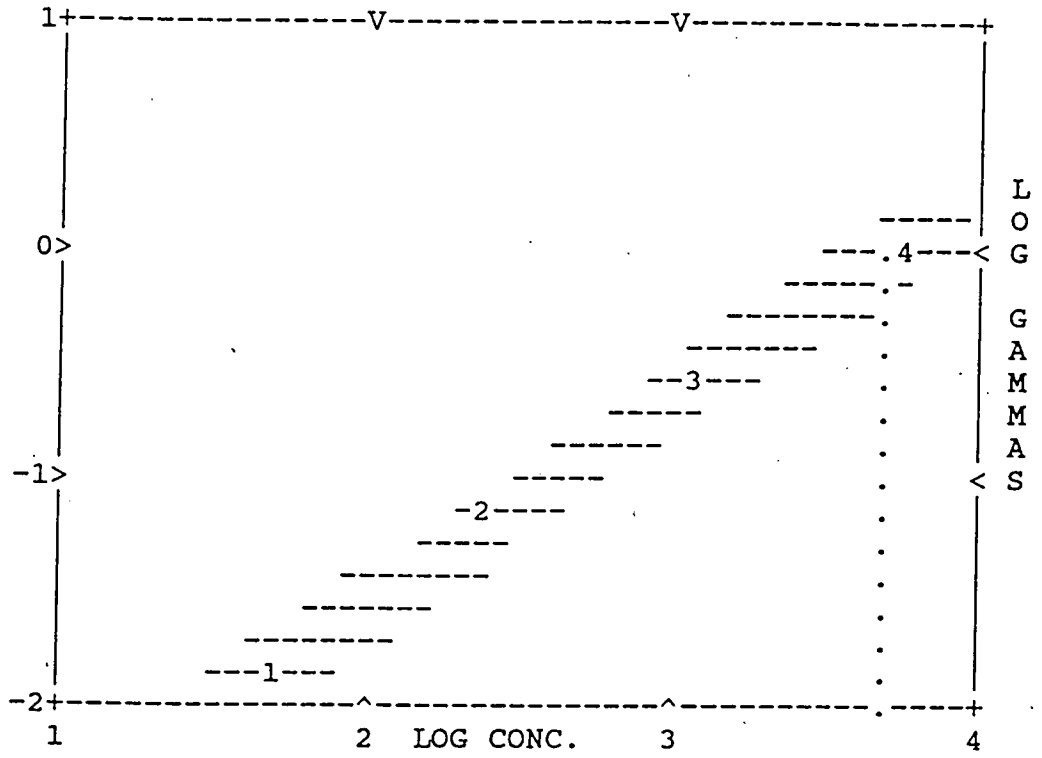
HOW TO PRINT A GRAPH

1. Wait for the graph to be displayed on the screen.
2. Use the Print Screen Key to print the graph.
3. Then, touch any key
   to return to the MICROTOX CALCULATIONS MENU.

TOUCH ANY KEY
And the graph will be displayed on the screen.

Display Ten

##	RES.	PROB.
1	0.181	100
2	0.116	41
3	0.120	44
4	0.174	92



Data Printout

SAMPLE RUN, DATA REDUCTION PROGRAM

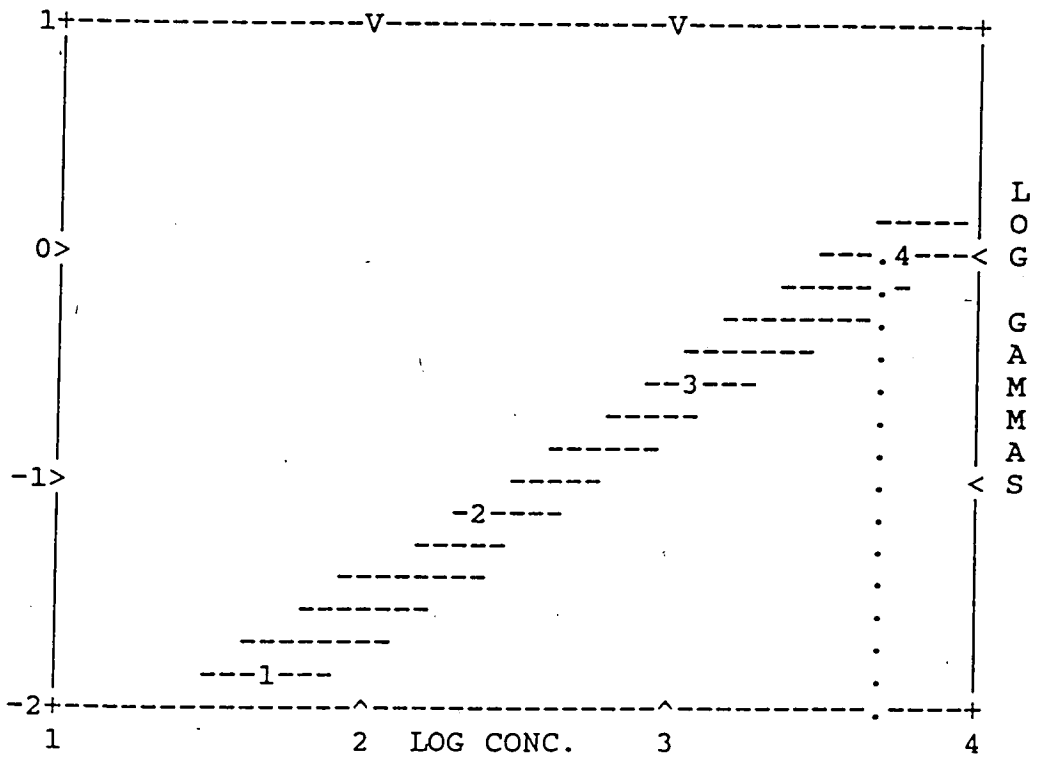
PAIR #	CONC.	G-OBS	G-EST
1	5.600	0.165	0.169
2	11.300	0.320	0.319
3	22.500	0.627	0.597
4	45.000	1.082	1.120

BLANK Bo/Bt= 100 / 88
BLANK RATIO= 0.8800

EC 50 = 39.652 (33.629 TO 46.755)

R =0.99898 SLOPE = 1.0981 INTERCEPT = +3.6802

##	RES.	PROB.
1	0.181	100
2	0.116	41
3	0.120	44
4	0.174	92



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Attachment 1

Microtox® Data Sheet

AQUA SURVEY, INC.
Microtox Data Sheet

ASI Number	Test Material	Test Start Date	Test Start Time

Nominal Concentration		Light Reading Interval - Minutes					
(%)	(mg/L)	Initial					
	A						
	B						
	C						
	Mean						
	A						
	B						
	C						
	Mean						
	A						
	B						
	C						
	Mean						
	A						
	B						
	C						
	Mean						
	A						
	B						
	C						
	Mean						
	A						
	B						
	C						
	Mean						
Date/Time and Initial							

Comments:

Signature	Date	Reviewed by	Date	Page ____ of

SOP NO.	TOX-1001
REVISION NO.	00
DATE	09/17/92

Prepared By: _____ 09/16/92
Date

Approved: _____ 9/16/92
York Terrell
GLP Testing Manager
Date

Approved: _____ 9/16/92
James Todd
Director of Operations
Date

Approved: _____ 09/16/92
Carl Bretton
Quality Assurance Unit
Date

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Specializing in Microbiology

PURE EARTH

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40 CFR 503 SLUDGE PATHOGEN TESTING SAMPLING SUGGESTIONS

SAMPLE VOLUME:

It is important that volume is based on the Total Solids percentage of the biosolid to be tested or analyzed.

Sample to achieve at least 4 grams dry weight equivalent for Salmonella and Helminth Ova testing.

At least 8 grams dry weight equivalent for Enteric Viruses and 1 gram dry weight equivalent for Fecal Coliform analysis.

TO NOTE - EXAMPLE:

> If Salmonella, Helminth Ova and Enteric Viruses are required and your Total Solids percentage is 50%, you should provide 8 grams wet weight for Salmonella, 8 grams wet weight for Helminth Ova and 16 grams wet weight for Enteric Viruses.

It is important the Total Minimum wet weight sample to be provided = 32 grams.

Optimal wet weight would be approximately 100 grams. This would allow for a suitable margin of error.

SAMPLE CONTAINER:

The Sample Container must be sterile and properly marked with sample data, location, time, Total Solids percentage (or performed on Pure Earth Environmental Laboratory) sample (with signature change of custody) and your ID# and PO#.

SAMPLE SHIPMENT:

1. Ship same day as sampling, on wet/blue ice, via overnight delivery.
2. Ship sample to arrive PREFERABLY ON TUESDAY OR WEDNESDAY.

Please make every effort to give 48 hours notice of sample arrival to Pure Earth Environmental Laboratory in order for us to put you on our busy schedule.

SHIP TO: Pure Earth Environmental Laboratory, Inc.
7184 North Park Drive
Pennsauken, NJ 08110

SAMPLING:

aseptic technique and sample according to general accepted methods from the 40 CFR 503 protocol.

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DETERMINATION OF TOTAL DRY SOLIDS
Method D4994-89,ASTM (1992)

1. Weigh a dry weigh pan at a constant weight. Place 50 ml or 50 grs. sludge portion for solids determination into the pan and weigh.

2. Place the pan and its contents into an oven maintained at 103-105 C for at least 1 hour.

3. Cool the sample to room temperature in a desiccator and weigh again.

4. Repeat the drying (1 hr each), cooling , and weighing steps until the loss in weight is no more than 4% of the previous weight.

5. Calulate the fraction of total dry solids (T) using the formula: T=A/B
   A=weight of the sample after drying (Pan dish is T off on the balance)
   B= weight of the sample before drying

   example: A=3.5
   B=5.0 0.7 x 100=70%

FECAL COLIFORMS, BIOSOLID, 503 METHOD

I. PURPOSE

1.0 This procedure outlines the steps involved in utilizing the Most Probable Number (MPN) method to analyze biosolid samples for the presence of fecal coliforms as per EPA regulations. The 503 method is described for liquid and solid samples of Class A, Alternative 1 or Class B, Alternative 1.

2.0 The 503 method describes guidelines for reducing pathogen levels in biosolids (sludge). Class A designation is given to biosolids in which the pathogens are below detectable levels. Biosolids are designated Class B if pathogens are detectable but have been reduced to levels which allow restricted use.

3.0 Class A, Alternative 1 is defined to signify that those biosolids have been thermally treated by being subjected to one of four time-temperature regimes. There are a total of seven alternatives for achieving Class A pathogen levels.

4.0 Class B, Alternative 1 is defined to signify that those biosolids may contain some pathogens, but the indicator organisms are being monitored. There are a total of three alternatives for meeting Class B pathogen requirements.

5.0 CLASS A, ALTNERATIVE 1, SOLID SAMPLES

5.1 This procedure is a modification of the 503 method. The procedure is modified because it has been noted by this laboratory that when testing solid samples the following is found.

5.1.1 If 10.0 g or 5.0 g of solid sample is added to 10.0 ml of broth, ALL the liquid culture medium is absorbed and the results cannot be determined.

5.1.2 It has been found by this laboratory that 1.0 g should replace all steps requesting 10.0 g and that 0.5 g should replace all steps requesting 1.0 g. The results obtained with this adjustment yield results consistent with the standards set forth in the method 503.

5.2 Four series (rows) of 5 tubes of EC broth are set up in a rack and allowed to warm to room temperature.

5.2.1 1st series: Double strength EC broth
5.2.2 2nd series: Double strength EC broth
5.2.3 3rd series: Double strength EC broth
5.2.4 4th series: Single strength EC broth

5.3 Blend 50 g of sample in 450 ml of PBS on high speed for 2 minutes. This blended sample is a 0.1 dilution of original sample.

5.4 Place 11 ml of sample into 99 ml of PBS. This is Dilution A, which is a 1×10^{-2} dilution of the original sample.

5.5 Inoculate the tubes with the following aliquots:

5.5.1 1st series: 10 ml blended sample (dilution factor = 1)
5.5.2 2nd series: 5 ml blended sample (dilution factor = 0.5)
5.5.3 3rd series: 10 ml Dilution A (dilution factor = 0.1)
5.5.4 4th series: 1 ml Dilution A (dilution factor = 0.01)

5.6 Incubate tubes in 44.5° C water bath for 24 hours.

5.7 After incubation view tubes for turbidity and gas production in fermentation vials. Positive reactions are indicative of fecal coliforms.

5.8 Only three of the four series will be used for estimating MPN. Choose the greatest dilution that gives a positive result in all five tubes and then the next two higher dilutions.

5.8.1 The greatest dilution equals the most diluted sample, which is that of Series 4.

5.8.2 If Series 2, 3 or 4 have all 5 tubes with positive growth, then the combination of positives will be Series 2,3,4.

5.8.3 If there are no positives, or if only Series 1 has all five tubes with positive growth, then the combination of positives will be 1,2,3.

5.9 Select MPN index/100 ml based on combination of positives. MPN index/100 ml is contained in the table on Form SAP 330B.

5.10 Calculation: Compute MPN/g using the following equation.

$$\text{MPN/g} = \frac{10 \times \text{MPN index/100 ml}}{\text{largest volume used} \times \%\ \text{total solids}}$$

10 = factor of equation
MPN index/100 ml = value obtained from Form SAP 330B
Largest volume used = least diluted sample from combination of positives
If used 2,3,4 combo, then largest volume is 0.5 g

If used 1,2,3 combo, then largest volume is 1 g
% Total solids = value obtained from determining % total solids (separate assay)

5.11 Specifications: Class A Alternatives are allowed 1,000 MPN/g of total solids (dry weight).

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FECAL COLIFORM TEST FOR SLUDGE
(All steps are performed aseptically)

Class B Alternative 1

The geometric mean of the Fecal Coliform density must not exceed 2 million CFU or MPN per gram of sewage sludge solids (dry weight basis).

LIQUID SAMPLES

1.) Transfer 1.0 ml of well mixed sample to 99.0 ml of PBS in a screw cap bottle. Shake a minimum of 25 times. (this is dilution "A", and is 0.010 ml of the original sample).

2.) Transfer 1.0 ml of dilution "A" to a second 99.0 ml blank of PBS, and mix as before. (this is dilution "B", and is 0.00010 ml of the original sample).

3.) Transfer 1.0 ml of dilution "B" to a third 99.0 ml blank of PBS, and mix as before. (this is dilution "C", and is 0.0000010 ml of the original sample). GO TO MPN OR MF ANALYSIS.

SOLID SAMPLES

1.) Weigh out 50.0 grams of well-mixed sample and place in a blender.

2.) Add 450.0 ml of PBS to the blender and blend on high for 2 minutes. (1.0 ml of this sample contains 0.10 grams of the original sample).

3.) Transfer 11.0 ml of the blender contents to a screw cap bottle containing 99.0 ml of PBS. Shake a minimum of 25 times. (this is dilution "A", and 1.0 ml of this sample contains 0.010 grams of the original sample).

4.) Follow the procedure for liquid samples starting at step 2.

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MPN FOR FECAL COLIFORM CLASS B, ALTERNATIVE 1.

FOUR SERIES OF FIVE TUBES ARE TO BE USED.

1.) Inoculate the first series of 5 tubes containing EC broth with 10.0 ml of dilution "B" (this is 0.0010 of the original sample).
2.) Inoculate the second series of 5 tubes containing EC broth with 1.0 ml of dilution "B" (0.00010).
3.) Inoculate the third series of 5 tubes containing EC broth with 10.0 ml of dilution "C" (0.000010).
4.) Inoculate the fourth series of 5 tubes containing EC broth with 1.0 ml of dilution "C" (0.0000010).
5.) Incubate the inoculated tubes in a water bath at 44.5° +/- 0.2° for 24 +/- hours. (place tubes in the water bath within 30 minutes of inoculation. The depth should be sufficent to cover all liquid in tubes.).
6.) A positive result for fecal coliforms is the production of gas within 24 +/- 2 hours.

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FECAL COLIFORM TEST FOR SLUDGE
(All steps are performed aseptically)

Class A Alternative 1

Fecal coliform density must be less then 1,000 MPN fecal coliform per gram of total solids (dry weight basis).

LIQUID SAMPLES

(four series of five tubes containing EC broth and inverted vial will be used for analysis).

1.) Transfer 10.0 ml of well mixed sample directly into the first series of five tubes.

2.) Transfer 1.0 ml of well mixed sample directly into the second series of five tubes.

3.) Transfer 1.0 ml of well mixed sample to a 99.0 ml PBS blank. Mix by vigorously shaking a minimum of 25 times. This is dilution "A".

4.) Transfer 10.0 ml of dilution "A" to the third series of five tubes.

5.) Transfer 1.0 ml of dilution "A" to a fourth series of five tubes. GO TO STEP 5 OF MPN FOR FECAL COLIFORM CLASS B, ALTERNATIVE 1.

SOLID SAMPLES

(four series of five tubes containing Lauryl Tryptose broth of various concentrations with inverted vials)

1.) Transfer 10.0 grams of well mixed sample directly into the first series of five tubes containing 10.0 ml of double strength Lauryl Tryptose broth.

2.) Transfer 1.0 gram of well mixed sample directly into the second series of five tubes containing 10.0 ml of single strength Lauryl Tryptose broth.

3.) Prepare dilution "A" as described for CLASS B, ALTERNATIVE 1: "SOLID SAMPLE".

4.) Transfer 10.0 ml of dilution "A" to the third series of five tubes containing single strength Lauryl Tryptose broth.

5.) Transfer 1.0 ml of dilution "A" to the fourth series of five tubes containing single strength Lauryl Tryptose broth.

6.) Incubate the inoculated tubes at 35.0° +/- 0.5 for 24 +/- 2 h.

7.) A presumptive identification of coliform organisms is the production of gas within 24 +/- 2 h. Any negative tubes are to be reincubated and reexamined at the end of 48 +/- 3 h.

8.) All positive tubes are to be inoculated into tubes of EC broth containing an inverted

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(step 8 continued from sludge, FC Class A, Alternative 1)
vial. Incubate at 44.5°+/- 0.2 for 24 +/- 2 h in a water bath which covers the liquid in the tubes completely.

9.) A positive result for fecal coliforms is the production of gas within 24 +/- 2 h.

10.) Proceed with identification of MPN (MPN chart included).

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SALMONELLA SP. TEST FOR SLUDGE
(all steps are performed aseptically)

CLASS A CRITERIA

Salmonella sp. density must be below 3 MPN per 4 grams (dry weight basis) in order to qualify for Class A.

LIQUID SAMPLES

1.) Follow the same procedure as for liquid samples of fecal coliform analysis CLASS A, ALTERNATIVE 1 (therefore obtain the "A" dilution). Only three series of five tubes will be used for MPN. The tubed medium should be as follows:

A.) Series 1: 10.0 ml of double strength DSE broth.
B.) Series 2: 10.0 ml of single strength DSE broth.
C.) Series 3: 10.0 ml of double strength DSE broth.

2.) Inoculate SERIES 1 with 10.0 ml of well mixed liquid sample.
3.) Inoculate SERIES 2 with 1.0 ml of well mixed liquid sample.
4.) Inoculate SERIES 3 with 10.0 ml of DILUTION "A".
5.) Incubate all tubes at 35.0° +/- 0.5 for 24 +/- 2 h.
6.) A positive result will show the presence of growth (turbidity).
7.) All positive results are to be inoculated on Hektoen enteric and XLD media. Incubate at plates at 35.0° +/- 0.5 for 24 +/- 2 h.
8.) A positive Hektoen enteric media result is a colony which is blue-green with a black center. A positive XLD media result is a colony which is red with a black center.
9.) All positives are then to be used for the standard determination of MPN.

SOLID SAMPLES

1.) Follow the same procedure as for solid samples of fecal coliform analysis CLASS "A", ALTERNATIVE 1 (therefore obtain dilution "A"). Only three series of five tubes will be used for MPN. The tubed media should be as follows:

A.) Series 1: 10.0 ml of double strength DSE broth.
B.) Series 2: 10.0 ml of single strength DSE broth.
C.) Series 3: 10.0 ml of double strength DSE broth.

2.) Inoculate SERIES 1 with 10.0 grams of well mixed solid sample.
3.) Inoculate SERIES 2 with 1.0 gram of well mixed sample.

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PURE EARTH

4.) Inoculate SERIES 3 with 10.0 ml of dilution "A".

5.) Go to step 5 for liquid samples, Class "A", ALTERNATIVE 1 for Salmonella sp. in sludge.

* It has been noted by this laboratory that when testing solid samples the following is found:

If 10.0 grams or 5.0 grams of solid sample is added to 10.0 ml of broth, ALL liquid is absorbed and the results can not be determined. It has been found by this laboratory that 1.0 gram should replace all steps requesting 10.0 grams, and that 0.5 grams should replace all steps requesting 1.0 grams. The results obtained with this adjustment yield results consistent with the standards set forth in the method 503.

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PREPARATION OF DULCITOL SELENITE CYSTINE BROTH (DSE).

(all steps should be performed aseptically)

SINGLE STRENGTH FORMULA

1.) Place 11.5 grams of the Selinite Cystine broth (SE) medium into a sterile 500 ml screw top bottle.
2.) Add 2.5 grams of Dulcitol to the SE broth.
3.) Add 500 ml of reagent grade water.
4.) Dissolve fully (with frequent agitation) but AVOID HEATING.
5.) Dispense in 10 ml amounts into 50 ml sterile test tubes.

DOUBLE STRENGTH FORMULA

1.) Simply double the amount of SE broth medium (23.0 grams) and Dulcitol (5.0 grams) placed in 500 ml of water.

SOUTHWEST LABORATORY OF OKLAHOMA, INC.

TM

Standard Operating Procedure

Zero Headspace Extraction for TCLP / SPLP

Document No.: SWL-IN-701

Rev No. / Date: Rev. 4.0 — 05/06/98

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RED STAMP INDICATES ORIGINAL

APPROVALS	DATES
Chuck Hoover Procedure Prepared By	5/6/98 Date
Program Manager or Section Supervisor	5/7/98 Date
Mike Butler Laboratory Safety Officer	5/6/98 Date
Chuck Hoover Laboratory QA/QC Officer	5/6/98 Date

(Effective Date is 5 calendar days after the last signature above - QA/QC Officer)

Document Status

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ZERO HEADSPACE EXTRACTION FOR TCLP / SPLP

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1.0 SCOPE AND APPLICATION

1.1 The Toxicity Characteristic Leaching Procedure (TCLP) and Synthetic Precipitation Leaching Procedure (SPLP) is to determine the mobility of both organic and inorganic analytes in liquid, solid, and multiphasic wastes.

1.2 If a total analysis of the waste demonstrates that individual analytes are not present in the waste, or that they are present but at such low concentrations that the appropriate regulatory levels could not be exceeded, a TCLP need not be run.

1.3 If an analysis of any one of the liquid fractions of a TCLP extract indicates that a regulated compound is present at such high concentrations that, even after accounting for dilution from the other fractions of the extract, the concentration would be above the regulatory level for that compound, then the waste is hazardous and it is not necessary to analyze the remaining fractions of the extract.

2.0 METHOD SUMMARY

2.1 For liquid waste (i.e., those containing less than 0.5% dry solid materials), after filtration through a 0.6 to 0.8um glass fiber filter, is defined as the TCLP / SPLP extract.

2.2 For wastes containing greater than or equal to 0.55% solids, the liquid, if any, is separated from the solid phase and stored for later analysis. The solid phase is extracted with an amount of extraction fluid equal to 20 times the weight of the solid phase. Following extraction, the liquid phase is separated from the solid phase by filtration through a 0.6 to 0.8 um glass fiber filter.

2.3 If compatible, the initial liquid of the waste is added to the liquid extract, and these are analyzed together. If incompatible, the liquids are analyzed separately and the results are mathematically combined to yield a volume-weighted average concentration.

3.0 HEALTH AND SAFETY.

Refer to the Lab Safety Plan (SWL-GA-111) for precautions.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING & STORAGE.

N/A

5.0 INTERFERENCES AND POTENTIAL PROBLEMS.

Potential interferences that may be encountered during analysis are discussed in the individual analytical SOP's.

6.0 EQUIPMENT / APPARATUS

6.1 Agitation apparatus: Associated Design and MFG. Co., Model No. 3740-24-BRE-TM. Capable of maintaining 30 ± 2rpm.

6.2 Zero-Headspace Extraction Vessel. This device is for use only when the waste is being tested for the mobility of volatile analytes. The ZHE allows for liquid/solid separation within the device, and effectively precludes headspace. This type of vessel allows for

initial liquid/solid separation, extraction, and final extract filtration without opening the vessel. The ZHE should be checked for leaks after every extraction. Pressurize the device to 50psi, allow it to stand unattended for one hour, and recheck the pressure. If the pressure is lost, check all fittings and inspect and replace O-rings, if necessary. Retest the device.

6.3 Filter. Glass fiber filters with a pore size of 0.6 to 0.8 um.

6.4 pH meter. Accumet Model 915.

6.5 Top Loading Balance. Mettler PM2000.

6.6 Magnetic stirrer.

6.7 ZHE Extract Collection Devices. PTFE gas-tight syringe

7.0 REAGENTS

7.1 Reagent Water. Millipore Super-Q (Carbon Filtration)

7.2 Sodium hydroxide (1N). Dissolve 40g of NaOh in 500 ml distilled water, dilute to 1L.

7.3 Glacial acetic acid, concentration. ACE reagent grade.

7.4 TCLP Extraction fluid #1. Add 5.7ml glacial acetic acid (see Section 7.3) to 500ml reagent water (see Section 7.1), add 64.3ml of 1N NaOH (see Section 7.2), and dilute to 1 liter. When correctly prepared, the pH of this solution will be 4.93 ± 0.05. If pH is outside these limits the fluid will be discarded and fresh extraction fluid prepared.

7.5 SPLP Extraction Fluid: This fluid is reagent water (Step 7.1) and is used to determine cyanide and volatiles leachability.

8.0 PROCEDURE

8.1 Preliminary determination of percent solids:

8.1.1 If the waste will obviously yield no liquid when subjected to pressure filtration proceed to Section 8.2.

8.1.2 Pre-weigh the filter and the container that will receive the filter. Record weight on TCLP Percent Moisture Worksheet (Attachment 1).

8.1.3 Assemble the filter holder and filter. Place the filter on the support screen and secure.

8.1.4 Weigh out a sub-sample of the waste (100gram minimum) and record the weight.

8.1.5 Allow slurries to stand to permit the solid phase to settle. Transfer the waste sample to the filter holder and gradually apply vacuum of 1-10psi, until air moves through the filter. Slowly increase the vacuum in 10psi increments to 50psi. When liquid flow ceases stop the filtration.

8.1.6 The material in the filter holder is defined as the solid phase and the filtrate is defined as the liquid phase.

8.1.7 Some waste such as oil wastes and paints will not filter and is defined as a solid. Use only one filter for this procedure and do not replace.

8.1.8 Determine the weight of the liquid phase by subtracting the weight of the filtrate container from the total weight of the filtrate-filled container. Determine the weight of the solid phase by subtracting the weight of the liquid phase from the weight of the total waste sample.

8.1.9 Determine the percent solids as follows:

$$\text{Percent solids} = \frac{\text{Weight of solid}}{\text{Total weight of waste}} \times 100$$

8.1.10 If the percent solid is equal to or greater than 0.5%, then proceed to Section 8.2 to determine if particle size reduction is necessary or to Section 8.1.11 if it's noticed that water is entrapped in the solid material. If the percent solid is less than 0.5%, proceed to 8.3 with a fresh portion of the waste.

8.1.11 Remove the solid phase and filter from the filtration apparatus.

8.1.12 Dry the filter and solid phase at 100 ± 20°C until the two successive weighings yield the same value within 1%. Record the final weight.

8.1.13 Calculate the percent solids as follows:

$$\text{Percent solids} = \frac{\text{(Wt. Of dry waste + filter)} - \text{tared wt. of filter}}{\text{Initial weight of waste}} \times 100$$

8.1.14 If the percent solids is less than 0.5% proceed to Section 8.3. If the percent dry solid is equal to or greater than 0.5%, proceed to Section 8.2 and determine if particle size reduction is necessary.

8.2 Determination if particle size reduction is required.

8.2.1 Particle size reduction is not required if the waste is smaller than 1cm in its narrowest dimensions. Proceed to Section 8.3.1.

8.2.2 If the waste is larger than 1cm in its narrowest dimension, prepare the waste for extraction by cutting, crushing or grinding to reduce the particle size. When complete proceed to Section 8.3.1.

8.2.3 Wastes and appropriate reduction equipment should be refrigerated to 4°C prior to particle size reduction. The means used to effect particle size reduction must not generate heat. If reduction is necessary, exposure of the waste to the atmosphere should be avoided to the extent possible.

8.3 Extraction procedure

8.3.1 The ZHE device has approximately a 500ml internal capacity. The ZHE can accommodate a maximum of 25 grams of solid, due to the need to add an amount of extraction fluid equal to 20 times the weight of the solid phase.

8.3.2 Do not allow the waste, the initial liquid phase, or the extract to be exposed to the atmosphere for any more time than is absolutely necessary. Any manipulation of these materials should be done when cold (4°C) to minimize loss of volatiles.

8.3.3 If the waste contains <0.5% dry solids (Section 8.1), the liquid portion of the waste, after filtration, is defined as the extract. Filter enough of the sample so that the amount of filtered liquid will support all of the volatile analysis required. The recommended sample size is as follows:

8.3.3.1 For waste containing <0.5% solids, weigh out a 500 gram sub-sample of waste and record the weight.

8.3.3.2 For wastes containing >0.5% solids, determine the amount of waste to charge into the ZHE as follows:

$$\text{weight of waste to charge ZHE} = \frac{25}{\text{percent solids (see Section 8.1)}} \times 100$$

8.3.3.3 Measure out waste to required weight ± 0.1g.

8.3.4 Waste slurries need not be allowed to stand to permit the solid phase to settle. Do not centrifuge wastes prior to filtration.

8.3.5 Transfer the entire sample (liquid and solid phases) quickly to the ZHE. Secure the filter and support screens onto the top flange of the device and secure the top flange to the ZHE body. Tighten all ZHE fitting sand place the device in the vertical position (gas inlet/outlet flange on the bottom).

8.3.6 Attach a gas line to the gas inlet/outlet valve and, with the liquid inlet/outlet valve open, apply gentle pressure of 1-10psi to force all headspace slowly out of the ZHE into a hood. At the first sign of liquid from the liquid inlet/outlet valve, quickly close the valve and discontinue pressure. If the waste is 100% solid, slowly increase pressure to a maximum of 50psi to force most of the headspace out of the device and proceed to Section 8.3.9.

8.3.7 Attach the evacuated pre-weighed gas-tight syringe to the liquid inlet/outlet valve and open the valve. Begin applying gentle pressure of 1-10psi to force the liquid phase of the sample into the syringe. If no additional liquid has passed through the filter in any 2 minute interval, slowly increase the pressure in 10psi increments to a maximum of 50psi. When liquid flow has ceased such that continued pressure filtration at 50psi does not result in any additional filtrate within a 2 minutes period, stop the filtration. Close the liquid inlet/outlet valve, discontinue pressure, and disconnect and weight the syringe.

8.3.8 The material in the ZHE is defined as the solid phase of the waste and the filtrate is defined as the liquid phase. If the original waste contained <0.5% dry solids, this filtrate is defined as the extract and is analyzed directly. Store the liquid phase at 4°C with zero-headspace till time of analysis.

8.3.9 Determine the amount of the extraction fluid to add to the extractor vessel as follows:

$$\text{Amount Of Extraction Fluid} = \frac{20 \times \text{percent solids (see Section 8.1)} \times \text{weight of waste filtered}}{100}$$

8.3.10 Determination of Extraction Fluid

8.3.10.1 For TCLP-ZHE use TCLP extraction fluid #1. (See Section 7.5)

8.3.10.2 SPLP Extraction Fluid is used for volatile leachability. (See Section 7.5)

8.3.11 With the ZHE in the vertical position, attach a line from the extraction fluid reservoir to the liquid inlet/outlet valve. The line must contain fresh extraction fluid and be pre-flushed with fluid to eliminate any air pockets in the line. Release gas pressure on the ZHE piston (from the gas inlet/outlet valve), open the liquid inlet outlet valve, and begin transferring extraction fluid into the ZHE.

8.3.12 After the extraction fluid has been added, close the liquid inlet/outlet valve and disconnect the extraction fluid line. Check the ZHE to ensure that all vaives are closed. Manually rotate the device end-over-end 2 or 3 times. Pressurize the ZHE to 5-10psi and slowly open the liquid inlet/outlet valve to bleed out any headspace. Re-pressurize the ZHE with 5-10psi and check all fittings to ensure that they are closed.

8.3.13 Place the ZHE in the rotary extractor and rotate at 30 ± 2 rpm for 18 ± 2 hours. Ambient temperature must be maintained at 23 ± 2°C during agitation.

8.3.14 Following the 18hour extraction, check the pressure of the ZHE piston by quickly opening and closing the gas inlet/outlet valve and noting the escape of gas. If the pressure has not been maintained, the device is leaking. Check the ZHE for leaking as specified in Section 6.2, and perform the extraction again with a new sample of waste. If the pressure within the device has been maintained, the material in the extractor vessel is ready to be separated into its liquid and solid phases. Filter through the glass fiber filter, using the ZHE device as discussed in Section 8.1.

8.3.15 If the waste contained an initial liquid phase, the liquid should be combined. A separate container must be used if combining would create multiple phases.

8.3.16 If the original waste contained no liquid the filtered extract obtained in Section 8.3 is defined as the TCLP / SPLP extract. If the waste contained an initial liquid phase, the filtered material obtained from Section 6.2 and the initial liquid phase (Section 8.3.6) are collectively defined as the TCLP / SPLP extract.

8.3.17 Following collection of the TCLP / SPLP extract, immediately prepare the extract for analysis and store with minimal headspace at 4°C until analysis.

9.0 CALCULATIONS. N/A

10.0 QUALITY ASSURANCE / QUALITY CONTROL

10.1 A minimum of one blank (using the same extraction fluid as used for samples) must be analyzed for every 20 extractions that have been conducted in an extraction vessel.

10.2 A matrix spike shall be performed for each waste type. A minimum of one matrix spike must be analyzed for each analytical batch. Matrix spikes are to be added after filtration of the TCLP extract and before preservation. Matrix spikes should be added at a concentration equivalent to the corresponding regulatory limit.

10.3 All quality control measures described in the appropriate analytical methods shall be followed.

11.0 WASTE DISPOSAL. N/A

12.0 METHOD PERFORMANCE. N/A

13.0 REFERENCES. USEPA, SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods. Method 1311 & 1312.

14.0 DEFINITIONS. N/A

15.0 ATTACHMENTS. Percent Moisture Worksheet

ATTACHMENT 1
TCLP / SPLP Percent Moisture Worksheet

TCLP/SPLP Extraction Log, Ref: SWL-IN-700 / IN-701

TCLP Extraction Log (700-ATT1.DOC) SWLO SOP rev. #____ Book # IN700-LOG-1 Page _____

Type of Extraction (circle): TCLP / Zero Head Space

Analyst Initials:_____ **Date:**_____ **Time:**_____ **Batch ID:**_____

#	SWL ID#	Particle Reduction Necessary?	INITIAL pH	ACID pH	FINAL pH	AMOUNT EXTRACTED	START TEMP & TIME	END TEMP & TIME
1								
2								
3								
4								
5								
6								
7								
8								
9								
10								
11								
12								
13								
14								
15								
16								
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22								
23								
24								
25								

COMMENTS:_____

Southwest Laboratory of Oklahoma, Inc.

Verified:_____

SOUTHWEST LABORATORY OF OKLAHOMA, INC.

Standard Operating Procedure

TCLP / SPLP Non-Volatile Extractions

Document No.: SWL-IN-700

Rev No. / Date: Rev. 4.0 — 05/06/98

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APPROVAL	DATES
Chuck Homer Procedure Prepared By	5/6/98 Date
Laboratory Manager or Director	5/8/98 Date
Laboratory Safety Officer	5/6/98 Date
Chuck Homer Laboratory QA/QC Officer	5/6/98 Date

(Effective Date for is 5 calendar days after the last signature above - QA/QC Officer)

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TCLP / SPLP NON-VOLATILE EXTRACTIONS

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1.0 SCOPE AND APPLICATION

1.1 The purpose of this SOP, Toxicity Characteristic Leaching Procedure / Synthetic Precipitation Leaching Procedures (TCLP / SPLP) is to determine the mobility of both organic and inorganic analytes in liquid, solid, and multiphasic wastes.

1.2 If a total analysis of the waste demonstrates that individual analytes are not present in the waste, or that they are present but at such low concentrations that the appropriate regulatory levels could not be exceeded, the TCLP / SPLP need not be run.

1.3 If an analysis of any one of the liquid fractions of the TCLP / SPLP extract indicates that a regulated compound is present at such high concentrations that, even after accounting for dilution from the other fractions of the extract, the concentration would be above the regulatory level for that compound, then the waste is hazardous and it is not necessary to analyze the remaining fractions of the extract.

2.0 METHOD SUMMARY

2.1 For liquid wastes (i.e., those containing less than 0.5% dry solid material), the waste, after filtration through a 0.6 to 0.8 um glass fiber filter, is defined as the TCLP / SPLP extract.

2.2 For wastes containing greater than or equal to 0.5% solids, the liquid, if any, is separated from the solid phase and stored for later analysis. The solid phase is extracted with an amount of extraction fluid equal to 20 times the weight of the solid phase. Following extraction, the liquid phase is separated from the solid phase by filtration through a 0.6 to 0.8 um glass fiber filter.

2.3 If compatible, the initial liquid of the waste is added to the liquid extract, and these are analyzed together. If incompatible, the liquids are analyzed separately and the results are mathematically combined to yield a volume-weighted average concentration.

3.0 HEALTH AND SAFETY. Refer to the Lab Safety Plan (SWL-GA-111) for precautions.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING & STORAGE. N/A

5.0 INTERFERENCES. Potential interferences that may be encountered during analysis are discussed in the individual analytical SOP's.

6.0 APPARATUS

6.1 Agitation apparatus: Associated Design and MFG. Co., Model No. 3740-24-BRE-TM. Capable of maintaining 30 +/-2 rpm.

6.2 Bottle Extraction Vessel. Two liter borosilicate glass jar.

6.3 Filtration Device.

6.4 Filters. Glass fiber filters with a pore size of 0.6 to 0.8 um. When analyzing for metals, the filters are rinsed with 1N nitric acid, followed by three consecutive rinses with deionized water.

6.5 pH meter. Accumet Model 915.

6.6 Top Loading Balance. Mettler PM2000, or equivalent — All measurements shall be ± 0.1g of the desired weight. All balances used shall be able to read to 0.01g.

6.7 Beakers. 250 ml glass.

6.8 Watchglass.

6.9 Magnetic stirrer.

7.0 REAGENTS

7.1 Reagent Water. Millipore – ASTM Type II (by conductivity).

7.2 Hydrochloric acid (1N). Add 83 ml conc. HCl to 500 ml distilled water, dilute to 1 L.

7.3 Nitric acid (1N). Add 64 ml conc. HNO_3 to 500 ml distilled water, dilute to 1 L.

7.4 Sodium hydroxide (1N). Dissolve 40 g of NaOH in 500 ml distilled water, dilute to 1 L.

7.5 Glacial acetic acid, conc., ACS reagent grade.

7.6 TCLP Extraction fluid #1. Add 5.7 ml glacial acetic acid(7.5) to 500 ml reagent water(7.1), add 64.3 ml of 1N NaOH(7.4), and dilute to 1 liter. When correctly prepared, the pH of this solution will be 4.93+/- 0.05. If pH is outside these limits the fluid will be discarded and fresh extraction fluid prepared.

7.7 TCLP Extraction fluid #2. Dilute 5.7 ml glacial acetic acid(7.5) with reagent water(7.1) to a volume of 1 L. When correctly prepared, the pH of this fluid will be 2.88 +/- 0.05. If pH is outside limits the fluid will be discarded and fresh fluid extraction fluid prepared.

7.8 SPLP Extraction Fluid #1: This fluid is made by adding the 60/40 weight percent mixture of sulfuric and nitric acids (or a suitable dilution) to reagent water (Step 7.1) until the pH is 4.20 ± 0.05. This fluid is used to determine the leachability of soil from a site that is east of the Mississippi River, and the leachability of wastes and wastewaters.

NOTE: Solutions are unbuffered and exact pH may not be attained.

7.9 SPLP Extraction Fluid #2: This fluid is made by adding the 60/40 weight percent mixture of sulfuric and nitric acids to reagent water (Step 7.1) until the pH is 5.00 ± 0.05. The fluid is used to determine the leachability of soil from a site that is west of the Mississippi River.

7.10 SPLP Extraction Fluid #3: This fluid is reagent water (Step 7.1) and is used to determine cyanide and volatiles leachability.

NOTE: These extraction fluids should be monitored frequently for impurities. The pH should be checked prior to use to ensure that these fluids are made up accurately. If impurities are found or the pH is not within the above specifications, the fluid shall be discarded and fresh extraction fluid prepared.

8.0 PROCEDURE

8.1 Preliminary determination of percent solids:

8.1.1 If the waste will obviously yield no liquid when subjected to pressure filtration proceed to Section 8.2.

8.1.2 Pre-weigh the filter and the container that will receive the filter. Record weight on TCLP / SPLP Percent Moisture Log (Attachment 1).

8.1.3 Assemble the filter holder and filter. Place the filter on the support screen and secure.

8.1.4 Weigh out a subsample of the waste (100 gram minimum) and record the weight.

8.1.5 Allow slurries to stand to permit the solid phase to settle. Transfer the waste sample to the filter holder and gradually apply vacuum of 1-10 psi, until air moves through the filter. Slowly increase the vacuum in 10 psi increments to 50 psi. When liquid flow ceases stop the filtration.

8.1.6 The material in the filter holder is defined as the solid phase and the filtrate is defined as the liquid phase.

8.1.7 Some wastes such as oily wastes and paints will not filter and is defined as a solid. Use only one filter for this procedure and do not replace.

8.1.8 Determine the weight of the liquid phase by subtracting the weight of the filtrate container from the total weight of the filtrate-filled container. Determine the weight of the solid phase by subtracting the weight of the liquid phase from the weight of the total waste sample.

8.1.9 Determine the percent solids as follows:

$$\text{Percent solids} = \frac{\text{weight of solid}}{\text{total weight of waste}} \times 100$$

8.1.10 If the percent solid is equal to or greater than 0.5%, then proceed to Section 8.2 to determine if particle size reduction is necessary or to Section 8.1.11 if it's noticed that water is entrapped in the solid material. If the percent solid is less than 0.5%, then the filtrate will be the TCLP / SPLP extract.

8.1.11 Remove the solid phase and filter from the filtration apparatus.

8.1.12 Dry the filter and solid phase at 100 +/- 20^0 C until the two successive weighings yield the same value within 1%. Record the final weight.

8.1.13 Calculate the percent solids as follows:

$$\text{Percent solids} = \frac{\text{(Wt. of dry waste + filter) - tared wt. of filter}}{\text{Initial weight of waste}} \times 100$$

8.1.14 If the percent solid is less than 0.5 % then the filtrate from Section 8.1.5 will be the TCLP / SPLP extract. If the percent dry solid is equal to or greater than 0.5%,return to Section 8.1.1 with a fresh portion of waste and determine if particle size reduction is necessary.

8.2 Determination if particle size reduction is required.

8.2.1 Particle size reduction is not required if the waste will pass through a 9.5 mm (0.375 in.) sieve. Proceed to Section 8.3.1.

8.2.2 If the waste will not pass through a 9.5 mm sieve then prepare the waste for extraction by cutting, crushing or grinding to reduce the particle size. When complete proceed to Section 8.3.1.

8.3 Determination of appropriate extraction fluid. If the solid content of the waste is greater than or equal to 0.5% determine the appropriate fluid for TCLP extraction as follows:

8.3.1 Weigh out a small subsample of the solid phase, reduce the solid, if necessary, to a particle size of 1mm in diameter or less. Transfer 5.0 g of the waste to a 250 ml beaker.

8.3.2 Add 96.5 ml of reagent water to the beaker, cover with a watchglass, and stir vigorously for 5 minutes on a magnetic stirrer. Measure and record the pH on the TCLP pH Run Log (Attachment 1). If the pH is <5.0 use extraction fluid #1 and proceed to section 8.4.1.

8.3.3 If the pH was greater than 5.0, add 3.5 ml 1N HCl, cover with a watch glass, heat to 50^0 C, and hold at 50^0 C for 10 minutes.

8.3.4 Let the solution cool to room temperature and record the pH. If the pH is <5.0, use extraction fluid #1. If the pH is >5.0, use extraction fluid #2.

8.3.5 For SPLP the following extraction fluid determination is made:

8.3.5.1 For soils, if the sample is from a site that is east of the Mississippi River, extraction fluid #1 should be used. If the sample is from a site that is west of the Mississippi River, extraction fluid #2 should be used.

8.3.5.2 For wastes and wastewater, extraction fluid #1 should be used.

8.3.5.3 For cyanide-containing wastes and/or soils, extraction fluid #3 (reagent water) must be used because leaching of cyanide-containing samples under acidic conditions may result in the formation of hydrogen cyanide gas.

8.4 Extraction procedure

8.4.1 Enough solids should be generated for extraction such that the volume of extract will be sufficient to support all of the analyses required. A good rule of thumb is 1-L per analyses.

8.4.2 Determine the amount of the raw sample needed to provide combined filtrate and extract totaling 2000mL. The following formula should be used to determine this amount:

$$(1-S/100)\,(RS) + 20(S/100)\,(RS) = 2000$$

where: S = % solids
RS = raw sample wieght

e.g. if the raw sample were 20% solids:

$$(1-20/100)\,(RS) + 20(20/100)\,(RS) = 2000$$
$$0.8RS + 4RS = 2000$$
$$RS = 417 \text{ grams } \pm .1 \text{ g}$$

8.4.3 Determine the amount of the extraction fluid to add to the extractor vessel a follows:

$$\text{Amount of extraction fluid} = \frac{20 \times \text{percent solids (8.1)} \times \text{weight of waste filtered}}{100}$$

8.4.4 Add the solids and appropriate amount of extraction fluid to the extractor vessel. Record information on TCLP / SPLP non-volatile extraction log (Attachment 1).

8.4.5 Close the extractor bottle tightly, secure in the rotary agitation device, and rotate at 30 +/- 2 rpm for 18 +/- 2 hours. Ambient temperature shall be maintained at 23 +/- 2°C during the extraction. Record both the time start and temperature at start on extraction log.

8.4.6 Following the 18 hour extraction, separate the material in the extractor vessel into its component liquid and solid phase by filtering through a new glass fiber filter as outlined in Section 8.1. For final filtration of the extract, the glass fiber filter may be changed, if necessary, to facilitate filtration. Filters shall be acid-washed (6.4) if evaluating the mobility of metals.

8.5 Prepare the extract as follows:

8.5.1 If the waste contained no initial liquid phase, the filtered liquid material is the extract.

8.5.2 If compatible, combine the filtered liquid obtained in Step 8.4 with the initial liquid phase of the waste obtained in Section 8.1. This combined liquid is defined as the extract.

8.5.3 If the initial liquid phase of the waste, obtained in Section 8.1, is not compatible with the filtered liquid resulting from Section 8.4, do not combine these liquids. Analyze these liquids, collectively defined as the extract, and combine the results mathematically. Determine the volume of the individual phases and combine the results by using the volume-weighted average:

$$\text{Final Analyte Concentration} = \frac{(V_1)(C_1) + (V_2)(C_2)}{V_1 + V_2}$$

where: V_1 = The volume of the first phase (liters).
C_1 = The concentration of the analyte of concern in the first phase (mg/l).
V_2 = The volume of the second phase (liters).
C_2 = The concentration of the analyte of concern in the second phase (mg/l).

8.5.4 Following collection of the extract, the pH of the extract should be recorded.

8.5.5 Immediately aliquot and preserve the extract for analysis. Metals aliquots must be acidified with nitric acid to pH <2. Metal matrix spike samples are to be spiked prior to the addition of HNO_3. All other aliquots must be stored at 4°C until analyzed.

9.0 CALCULATIONS. Not Applicable

10.0 QUALITY ASSURANCE

10.1 A minimum of one blank (using the same extraction fluid as used for samples) must be analyzed for every 20 extractions that have been conducted in an extraction vessel.

10.2 A matrix spike shall be performed for each waste type. A minimum of one matrix spike must be analyzed for each analytical batch. Matrix spikes are to be added after filtration of the extract and before preservation. Matrix spikes should be added at a concentration equivalent to the corresponding regulatory limit.

10.3 The method of standard additions (MSA) shall be used for metals if:

10.3.1 Recovery of the matrix spike for an analyte is < 50% and the concentration does not exceed the regulatory limit.

10.3.2 The concentration of the contaminant measured in the extract is within 20% of the regulatory level.

10.4 All quality control measures described in the appropriate analytical methods shall be followed.

11.0 WASTE DISPOSAL. Not Applicable

12.0 METHOD PERFORMANCE. Not Applicable

13.0 REFERENCES

- USEPA, SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods. Method 1311 & 1312.

14.0 DEFINITIONS — Not Applicable

15.0 ATTACHMENTS — TCLP / SPLP WORKSHEET

ATTACHMENT 1
TCLP / SPLP Worksheet

TCLP/SPLP Extraction Log, Ref: SWL-IN-700 / IN-701

TCLP Extraction Log (700-ATT1.DOC) SWLO SOP rev. #____ Book # IN700-LOG-1 Page _____

Type of Extraction (circle): TCLP / Zero Head Space

Analyst Initials:_____ **Date:**_____ **Time:**_____ **Batch ID:**_____

#	SWL ID#	Particle Reduction Necessary?	INITIAL pH	ACID pH	FINAL pH	AMOUNT EXTRACTED	START TEMP & TIME	END TEMP & TIME
1								
2								
3								
4								
5								
6								
7								
8								
9								
10								
11								
12								
13								
14								
15								
16								
17								
18								
19								
20								
21								
22								
23								
24								
25								

COMMENTS:_____

Southwest Laboratory of Oklahoma, Inc.

Verified:_____

ATTACHMENT B
RESUMES OF KEY LABORATORY PERSONNEL

SUMMARY OF TRAINING AND EXPERIENCE

Date: January 12, 1999

Name: York Terrell

Title: GLP Testing Manager

Address: Aqua Survey, Incorporated
499 Point Breeze Road
Flemington, New Jersey 08822

Education:

1973 Reading Area Community College
Reading, Pennsylvania
Major: Biology

1974 Penn State University
Berks Campus
Reading, Pennsylvania
Major: Anatomy

Employment History:

1990-present GLP Testing Manager
Chemical Receiving Officer for GLP compounds
Aqua Survey, Incorporated
Flemington, New Jersey
Duties: Supervise and conduct aquatic studies under GLP compliance for Pure Compound Department. Serve as Study Director for acute and chronic aquatic effect studies for product registration/production. Prepare and review protocols and reports for acute and chronic aquatic studies for product registration/production. Participate in the conduct and preparation of some acute and chronic bioassays. Participate in the conduct and preparation of some ocean disposal and sediment bioassays. Participate in the conduct and preparation of some Microtox® and *Daphnia magna* IQ Test™ bioassays.

1983-1990 Principal Investigator
American Standard Biosciences Corporation
Reading, Pennsylvania
Duties: Served as Principal Investigator and Study Director for acute aquatic, avian, and mammalian toxicology department. Prepared and reviewed protocols and reports of acute aquatic, avian, and mammalian toxicology department. Conducted acute aquatic effect studies for product registration/production using rainbow trout, bluegill sunfish, fathead minnow and the freshwater cladoceran, *Daphnia magna*.

Conducted eight (8)-day avian dietary studies for product registration using five-to-ten-day-old bobwhite quail and ten-to- fourteen-day-old mallard ducks. Conducted acute avian oral toxicity studies for product registration using sixteen-week-old bobwhite quail and mallard ducks. Conducted acute avian neurotoxicity studies using sixteen-week-old white leghorn hens. Conducted pyrogen and safety studies for the production of drug and medical devices using albino rabbits and albino mice. Conducted acute mammalian oral and inhalation toxicity studies for product registration/production using albino rats. Conducted acute mammalian dermal and eye irritation as well as dermal sensitization studies using albino rabbits and guinea pigs. Responsible for all gross necropsy.

1970-1983 Chief Biologist
Cannon Laboratory, Inc.
Reading, Pennsylvania
Duties: Supervised and conducted acute mammalian toxicity studies. Instituted animal care program for AAALAC accredition. Participated in bioavailability studies of pharmaceutical drugs. Participated in chronic feeding, inhalation and dermal studies for product registration using albino rats and mice. Conducted biological phase of metabolism studies for product registration using cows and goats as test models. Participated in the conduct of the biological phase of bioaccumulation studies for product registration using rainbow trout, bluegill sunfish, and channel catfish. Participated in the conduct of chronic aquatic effect studies for product registration using fathead minnows. Participated in reproduction studies for product registration using bobwhite quail, mallard ducks, and albino rats. Conducted special effect studies of pharmaceutical drugs using primates as a test model. Supervised the animal care program for the NCI bioassay program.

1969-1970 Biologist
K & G Laboratory
Parsippany, New Jersey
Duties: Conducted acute oral, skin and eye toxicity test using albino rats and rabbits. Participated in intramuscular implantation studies using albino rabbits. Participated in special effect studies of pharmaceutical drugs; participated in the breeding of albino rats and mice.

1967-1969 Laboratory Technician/Animal Caretaker
Food & Drug Administration
Maspeth (Long Island), New York
Duties: Responsible for the collection of specimens for clinical evaluation during subacute, subchronic, chronic and special effect studies. Responsible for the daily dosage of animals. Responsible for maintenance of all species of animals used in safety evaluation studies. Participated in Caesarean-sectioning of rat dams and fetal specimen preparation for reproduction studies.

Training Courses:

1967 AALAS Certification 240 hours

	Sloan Kettering Institute	
1968	Techniques for Collecting Specimens for Clinical Evaluation FDRL Training Program	4 hours
1969	Techniques for Caesarean-Sectioning FDRL Training Program	4 hours
1970	Techniques for Intramuscular Implantation K & G Lab Training Program	4 hours
1972	Symposium - The Use of Domestic Animals in Research Society of Toxicology (SOT)	
1976	Workshop - Use of the Nude Mice in Research American Association for Laboratory Animal Sciences (AALAS)	4 hours
1977	Seminar on Avian and Aquatic Toxicology Center for Professional Advancement	24 hours
1978	Symposium on Applied Toxicology and GLP's University of Mississippi	32 hours
1979	Inhalation Toxicology - Intensive Course Center for Professional Advancement	24 hours
1988	Environmental Toxicology - Intensive Course Center for Professional Advancement	24 hours
1990	Dioxin Hazard Safety Training Roy F. Weston	4 hours
1991	Use of the Bausch and Lomb Spectronic 21 Betz Laboratories	4 hours
1992	Good Laboratory Practice Training Seminar Aqua Survey, Inc.	2 hours
1992	Society of Quality Assurance, Mid-Atlantic Region (MARSQA) Workshop	6 hours
1992	Seminars on Pollution and Water Resources Columbia University	8 hours

1993	Mid - Atlantic Chapter Society of Toxicology	
1993	Good Laboratory Practice Training Seminar Golman Associates International, Inc.	16 hours
1997	Methods for Aquatic Toxicity Identification Evaluations. ASI in-house training	16 hours
1997	SETAC Endocrine Disrupters Workshop Philadelphia, PA	

Professional Committees:

1992-Present	Society of Quality Assurance, Mid-Atlantic Region (MARSQA)
1983-1990	Member of Review Committee for the Humane Use of Animals in Research
1971-1990	Animal Association for Laboratory Animal Science
1974-1978	New York Academy of Science

Awards:

1977	Cannon Laboratory Employee of the Year
1978	Cannon Laboratory Most Dedicated Person Award

Publications/Presentations:

Terrell, Y., *et al.* 1991. "The *Daphnia magna* IQ Toxicity Test™ and Microtox® are Compared for Rapid Testing With Seven Common Pesticides". Platform presentation, Society of Toxicology. Poster presentation, Society of Environmental Toxicology and Chemistry (SETAC).

Terrell, T., *et al.* 1993. "Predictive Ability of the *Daphnia magna* IQ Toxicity Test® for Ten Diverse Water Treatment Additives". Bull. Environ. Contam. Toxicol. (1993) 51:909-913©, 1993 Springer-Verlog New York Inc.

SUMMARY OF TRAINING AND EXPERIENCE

Date: January 12, 1999

Name:. Thomas J. Dolce

Title: Supervising Biologist

Address: Aqua Survey, Inc.
499 Point Breeze Road
Flemington, NJ 08822

908/788-8700

Education:

1987-1992 Cook College, Rutgers University
New Brunswick, NJ
Degree: Bachelor of Science
Major: Biology

1987-1992 Cook College, Rutgers University
New Brunswick, NJ
Degree: Bachelor of Science
Major: Natural Resource Management (Wildlife Science)

Employment History:

1998-Present Laboratory Manager
Aqua Survey, Inc.
Flemington, NJ 08822
Duties: Managing NPDES, ocean disposal and freshwater sediment studies. Scheduling, testing and assigning personnel to specific projects. Maintain contact with clients for all work in house, make sure personnel are properly trained for projects they are involved with.

1996- 1998 Supervising Biologist
Aqua Survey, Inc.
Flemington, NJ 08822
Duties: Responsible for maintaining NPDES program and keeping up to date on regulations. Trained for any necessary GLP testing. Conduct field sampling fro NPDES and ocean disposal testing. Assist in estuarine field surveys as risk assessment studies.

1989-1996 Bioassay Technician
Aqua Survey, Inc.
Flemington, NJ
Duties: Conduct acute and chronic effects testing. Conduct field sampling.

1993-1994 Aquatic Toxicologist
CFM Environmental Services
Somerville, NJ
Duties: Culturing of fish, invertebrates and algae. Conducted acute and chronic bioassays on these organisms through various effluents. Conducted quality control, field sampling and microbiological analysis.

1993-1993 Technician
Rutgers Marine Field Station
Tuckerton, New Jersey
Duties: Responsible for feeding and care of live fish. Conducted experiments on fish behavior. Conducted dissection, data entry and tag release program in an estuary. Conducted field sampling of larval fish in an estuary. Maintained wet and dry labs .

Professional Societies:

1992-Present American Fisheries Society (Member)

SUMMARY OF TRAINING AND EXPERIENCE

Date:	January 5, 1999
Name:	G. Stephen Hornberger
Title:	Bioassay Scientist
Address:	Aqua Survey, Incorporated 499 Point Breeze Road Flemington, New Jersey 08822

Education:

1975	The Pennsylvania State University University Park, PA 16802 Degree: Bachelor of Science Environmental Resource Management

Employment History:

1996-Present	Bioassay Scientist Aqua Survey, Inc. 499 Point Breeze Road Flemington, NJ 08822 Duties: Project management of dredging and disposal projects, Special projects, Toxicity Identification Evaluations (TIE), Also involved in GLP projects and whole effluent toxicity tests, (WET). Responsible for maintenance of laboratory systems and chemistry monitoring equipment and the co-ordination of testing services with contract laboratories, including the evaluation of contract laboratory data and reports.
1995-1996	Client Services Representative Accutest Laboratories Dayton, New Jersey Duties: Responsible for maintaining good client relationships through responsive liaison between the client and Accutest laboratory operations and providing timely response to technical questions and requests for quotations.
1978-1995	Inorganic Laboratory Manager, Senior Chemist, Environmental Specialist RECON Environmental Corp. Raritan, New Jersey

Duties: During the eighteen year tenure duties included: Field sampling, laboratory analyses, management of inorganic laboratory, project management, compliance with regulatory issues, analytical method development, laboratory personnel management, inventory management, report generation and client consultations.

CREDENTIALS

- B.S., Environmental Resource Management (1975) The Pennsylvania State University
- OSHA (CFR 1910.120) 40-hr. Hazardous Site Workers Training (1990), University of Medicine and Dentistry of New Jersey
- N-2 Industrial Wastewater System Operator License #5288 NJDEP
- Methods for Acute Toxicity Identification Evaluations
  ASI in-house training

PUBLICATIONS

ASTM Specialty Technical Publication 1282, Sampling Environmental Media, September 1996., Municipal Solid Waste Sampling and Characterization.

SUMMARY OF TRAINING AND EXPERIENCE

Date: January 13, 1999

Name: Michelle A. Thomas

Title: Laboratory Technician

Address: Aqua Survey, Incorporated
499 Point Breeze Road
Flemington, New Jersey 08822

908/788-8700

Education:

1992-1996 The Richard Stockton College of New Jersey
Pomona, New Jersey
Major: Environmental Science
Degree: Bachelor of Science

1992 Hunterdon Regional High School
Flemington, New Jersey, 08822

Employment History:

1997 – Present Laboratory Technician
Aqua Survey, Inc.
Flemington, New Jersey 08822
Duties: Responsible for acute and chronic NPDES testing; conducting field sampling for NPDES and ocean disposal testing. Assists with various aspects of ocean disposal testing (bioaccumulation testing, elutriate tests, % moisture, grain size); assist with TOC analyses. experienced in doing basic culture work; assists with report writing (NPDES and ocean disposal reports)

1996 Environmental Health Intern
Hunterdon County Health Department
Duties: Observed and assisted with the various aspects of the health department, i.e., sanitary inspections (septic and food), well inspections, water sampling, underground storage tank removal, plan reviews etc.

Training Courses:

1997 Toxicology and Risk Assessment course,
Delaware Valley College
Doylestown, PA

SUMMARY OF TRAINING AND EXPERIENCE

Date: January 12, 1999

Name: Robert M. Fristrom

Title: Laboratory/Field Technician

Address: Aqua Survey, Inc.
499 Point Breeze Road
Flemington, NJ 08822

908/788-8700

Education:

1981-1985 North Hunterdon Regional High School
Clinton, New Jersey

Employment History:

1985-Present Laboratory/Field Technician
Aqua Survey, Inc.
Flemington, NJ
Duties: Field duties include mobile lab preparation, set-up and daily maintenance of field tests, and sampling support for in-house acute and chronic testing. as well as sampling for ocean disposal testing. Laboratory duties include set-up and maintenance of flow-through dilutor systems, routine physical and chemical analyses, sample preparation and test organism maintenance for acute and chronic testing as well as ocean disposal testing. Assist in scheduling NPDES testing and maintaining client contact

Training Courses:

1986 Fathead Minnow Culture Course
Fisheries Academy, U.S. Fish and Wildlife Service.

APPENDIX H
COMPARISON OF RESULTS - FIELD TEST KIT VS. LABORATORY

FIELD AUDIT DEMONSTRATION SUMMARY
BIOREMEDIATION FACILITY
NSWC CRANE
CRANE, INDIANA

The following summarizes the results of the analytical field screening for nitroaromatics and nitrosamines (TNT and RDX) in soil samples at the Naval Surface Warfare Center (NSWC) Crane Division in Crane, Indiana. The field screening demonstration was performed at the request of SouthDiv and EPA to determine the comparability of field screening results to that of EPA SW846 Method 8330 and their suitability for use in meeting the data quality objectives in support full-scale operations at the Biofacility. The audit demonstration was based upon the outline provided by EPA and summarized in Attachment D.

Fifteen soil samples were collected in the area of Mine Fill A. These samples were homogenized and split in the field. One sample aliquot was analyzed in the field for TNT and RDX using the Ensys field test kits and the other aliquot was submitted to Southwest Laboratories for analysis referencing SW846 Method 8330. Field sample analysis was performed by extracting 20 grams of soil with acetone and analyzing the resultant extract by spectrophotometer. Quantitation is based on colorimetric comparison of the sample to that of known calibration standards within a working range of 1.0 mg/kg to 30.0 mg/kg. Sample analysis by Southwest Laboratories was performed following their standard operating procedures which are based on EPA SW846 Method 8330. The results of the comparison of field screening and laboratory analysis is summarized in Tables 1, Table 2, Attachment A, Attachment B and Attachment C.

The field test results were compared with the laboratory results for TNT and RDX to identify "false negatives" (i.e., detected by the laboratory method, but not by the field method) and "false positives" (i.e., detected by the field method, but not by the laboratory method). In cases where both methods showed detectable levels of RDX or TNT, a comparison was made by calculating the percent difference between the two results.

It is important to note that the reporting limits for the field screening kits are much higher than those of the off-site laboratory. The reporting limits for RDX and TNT using the Ensys kits are 1.0 mg/kg. The reporting limits for RDX and TNT analysis performed by Southwest Laboratories were 1.0 mg/kg and 0.25 mg/kg respectively. In cases where the laboratory detected either TNT or RDX below the level achievable by the appropriate field screening kit, results were deemed to be in agreement since both the field and off-site laboratory techniques achieved their specific objectives.

There were no false negative results for either TNT or RDX using the field screening kits. There were no false positive results for RDX and only one false positive result for TNT using the field test kits. The sample which exhibited the false positive result for TNT was found to be present right at the reporting limit.

TNT was detected in 12 of the 15 samples collected by both the field screening and off-site laboratory methods. TNT concentrations ranged from non-detected to 46200 mg/kg. Due to the high concentrations in a number of these samples, significant dilutions were required for both field screening methods and laboratory analysis. Eight of the 15 samples were within the calibration range of the field test kits and did not require any dilution. The percent difference between the laboratory and field concentrations were calculated for each of these samples and are presented in Table 1. The average TNT percent difference was 26%.

RDX was detected in 14 of the 15 samples collected by both the field screening and off-site laboratory methods. RDX concentrations ranged from non-detected to 38700 mg/kg. Due to the high concentrations in a number of these samples, significant dilutions were required for both field screening methods and laboratory analysis. The percent difference between the laboratory and field concentrations were calculated for each of these samples and are presented in Table 2. The average RDX percent difference was 41%.

Conclusions

During full-scale operations, field test kits will be used to assist in the delineation of the approximate excavation limits and the process monitoring of explosive degradation within the compost piles to determine when final confirmation samples should be collected. An off-site subcontract laboratory will be used for the final confirmation results in both the excavation areas and the final compost piles. Since the primary purpose of the test kits will be to help refine when analytical samples should be submitted to off-site laboratories, the accuracy of the measurement is not critical and only needs to approximate the concentration levels and provide a low percentage of false negative values. The laboratory and field screening results indicate that use of the Ensys kits resulted in low false positive and false negative rates. Percent differences between the field test kits and laboratory data were acceptable. Based upon these data quality objectives the field test kits are considered acceptable for use. It should be noted that no comparisons were made on the compost matrix and since field test kits can be impacted by sample matrix, the final recipe should be evaluated to ensure similar results are obtained.

Table 1

SUMMARY OF TNT FIELD SCREENING AND LABORATORY RESULTS

TNT

SAMPLE #	LABORATORY CONCENTRATION (MG/KG)	FIELD TEST KIT CONCENTRATION (MG/KG)	PERCENT DIFFERENCE
1	46200	44273	4.2
2	91	58	36.2
3	15	15	0
4	61	41	32.8
5	44400	43963	0.98
6	1	2	-100
7	12	7	41.7
8	Not Detected	1	Not Applicable
9	Not Detected	Not Detected	Not Applicable
10	19	19	0
12	547	406	25.8
15	123	104	15.4
16	367	198	46.0
17	19	17	10.5
BORROW	Not Detected	Not Detected	Not Applicable

Table 2

SUMMARY OF RDX FIELD SCREENING AND LABORATORY RESULTS

RDX

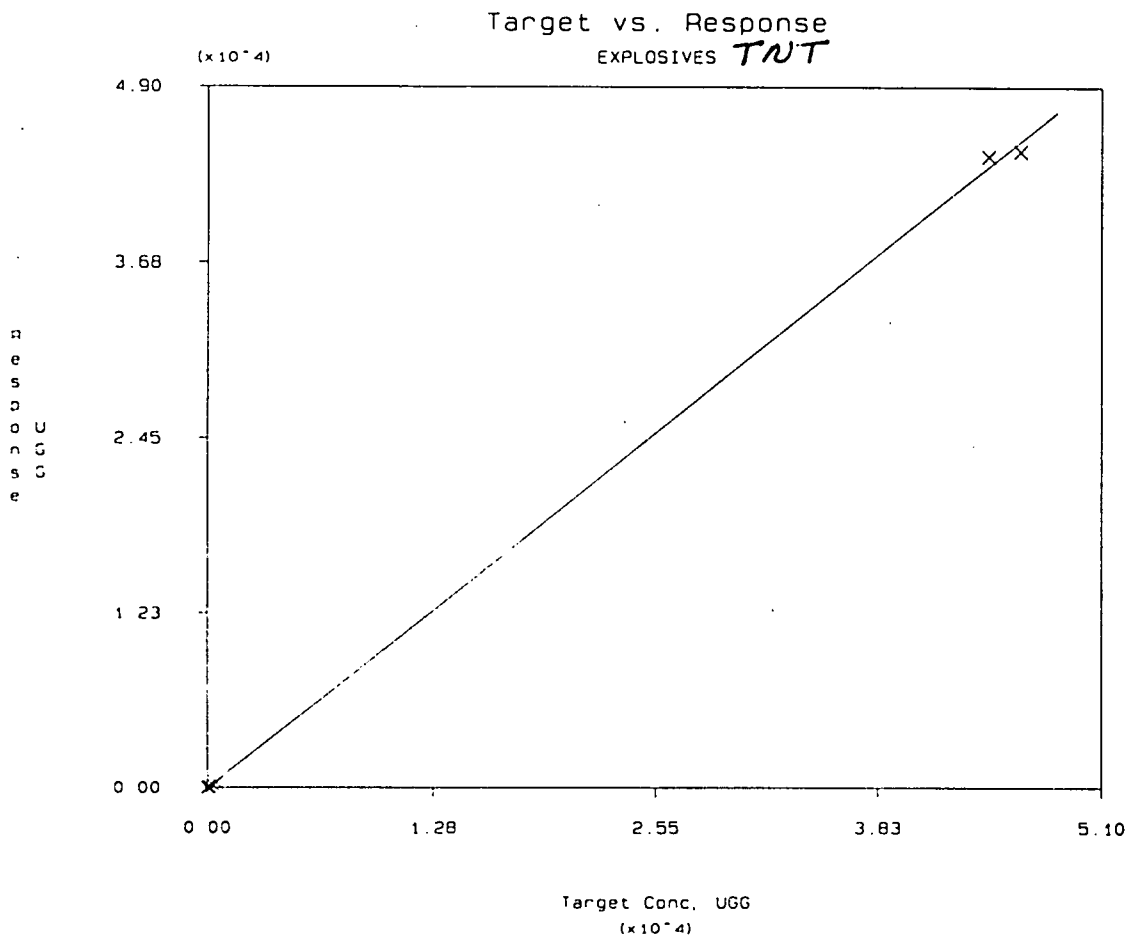
SAMPLE #	LABORATORY CONCENTRATION (MG/KG)	FIELD TEST KIT CONCENTRATION (MG/KG)	PERCENT DIFFERENCE
1	38700	78222	- 102
2	286	208	27.2
3	55	62	-12.7
4	237	218	8.02
5	37300	16222	56.5
6	77	103	-33.8
7	377	181	52.0
8	59	81	-37.3
9	61	74	-21.3
10	310	152	51.0
12	3480	2044	41.3
15	1140	587	48.5
16	3360	1157	65.6
17	740	561	24.2
BORROW	Not Detected	Not Detected	Not Applicable

Attachment A

RDX and TNT Summary Graphs and Statistics

TNT

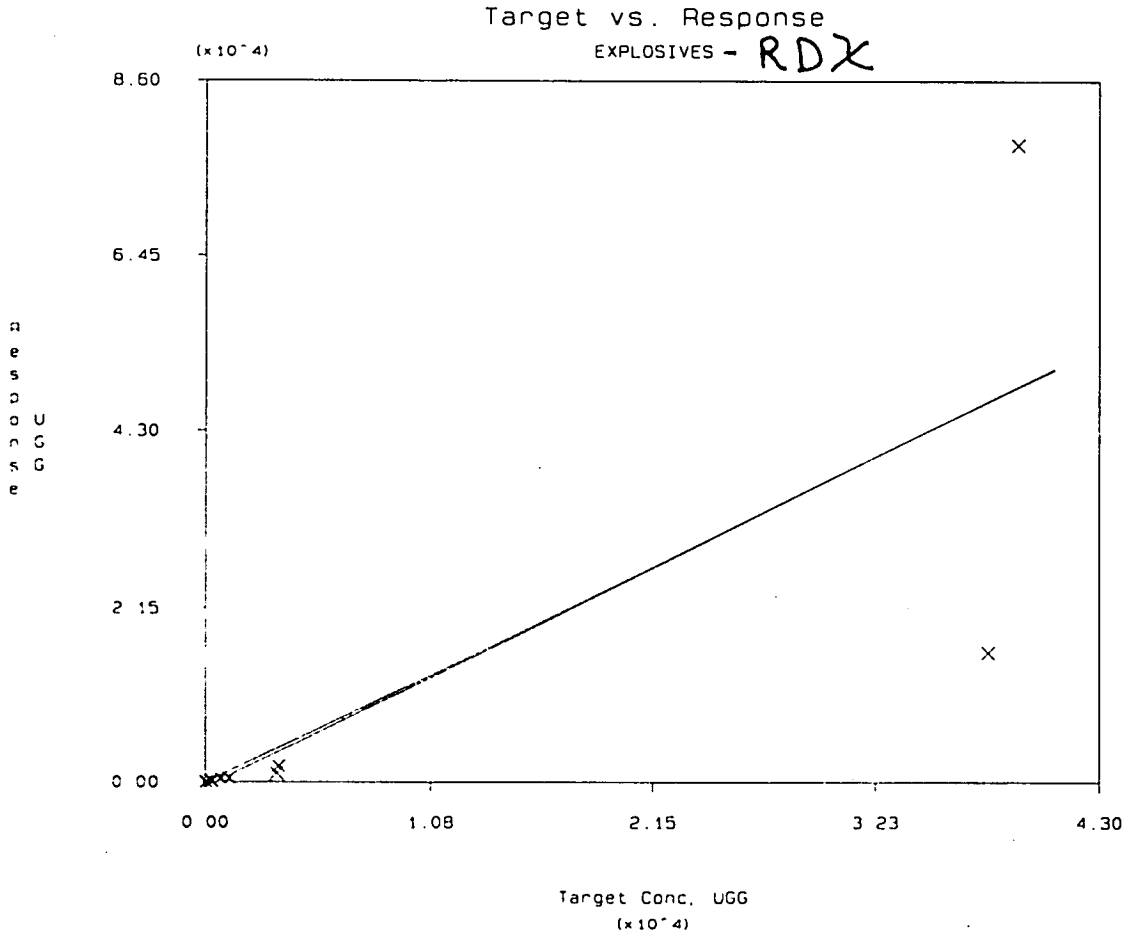
2/09/98 2: 47PM



RDX

2/09/98

2: 50PM



PRECERTIFICATION ANALYSIS TNT Report Date: 2/09/98

Method Name:	EXPLOSIVES	Laboratory:	SW
Test Name:	TNT	Analysis Date:	2/09/98
Units of Measure:	UGG	Matrix:	SO
		Run Number:	0

ANALYSIS OF RESIDUAL VARIATIONS

	Model with Intercept			Model through Origin		
	Slope: 0.97421900			Slope: 0.97355843		
	Y Intercept: -29.529687					
	(SS)	(df)	(MS)	(SS)	(df)	(MS)
Residual:	1074155.90	11	97650.5362	1083700.18	12	90308.3486
Total Error:	0.00000000	1	0.00000000	0.00000000	1	0.00000000
Lack Of Fit:	1074155.90	11	97650.5362	1083700.18	12	90308.3486
	LOF F-Ratio(F): 0.00000000			LOF F-Ratio(F): 0.00000000		
	Critical 95% F: 4.84			Critical 95% F:244.00		

ZERO INTERCEPT HYPOTHESIS

Calculated F: 0.09773920 Critical 95% F: 4.84

Zero Intercept Accepted

TABLE OF DATA POINTS

Targets: 13
Measurements per Target: 1

Target Values	Instrument Values
0.00000000	0.00000000
1.00000000	2.00000000
12.0000000	7.00000000
15.0000000	15.0000000
19.0000000	19.0000000
19.0000000	17.0000000
61.0000000	41.0000000
91.0000000	58.0000000
123.000000	104.000000
367.000000	198.000000
547.000000	406.000000
44400.0000	43963.0000
46200.0000	44273.0000

END OF LACK OF FIT TABLE

PRECERTIFICATION ANALYSIS RDX Report Date: 2/09/98

Method Name: EXPLOSIVES
Test Name: RDX
Units of Measure: UGG

Laboratory: SW
Analysis Date: 2/09/98
Matrix: SO
Run Number: 0

ANALYSIS OF RESIDUAL VARIATIONS

Model with Intercept

Slope: 1.26920392
Y Intercept: -634.03547

Model through Origin

Slope: 1.25045621

	(SS)	(df)	(MS)	(SS)	(df)	(MS)
Residual:	.183E+010	13	.140E+009	.183E+010	14	.131E+009
Total Error:	0.00000000	1	0.00000000	0.00000000	1	0.00000000
Lack Of Fit:	.183E+010	13	.140E+009	.183E+010	14	.131E+009

LOF F-Ratio(F): 0.00000000
Critical 95% F: 4.67

LOF F-Ratio(F): 0.00000000
Critical 95% F: 4.67

ZERO INTERCEPT HYPOTHESIS

Calculated F: 0.03564144 Critical 95% F: 4.67

Zero Intercept Accepted

TABLE OF DATA POINTS

Targets: 15
Measurements per Target: 1

Target Values	Instrument Values
0.00000000	0.00000000
55.0000000	62.0000000
59.0000000	81.0000000
61.0000000	74.0000000
77.0000000	103.000000
237.000000	218.000000
286.000000	208.000000
310.000000	152.000000
377.000000	181.000000
740.000000	561.000000
1140.00000	587.000000
3360.00000	1157.00000
3480.00000	2044.00000
37300.0000	16222.0000

PRECERTIFICATION ANALYSIS RDX, Cont. Report Date: 2/09/98

Method Name: EXPLOSIVES
Test Name: RDX
Units of Measure: UGG

Laboratory: SW
Analysis Date: 2/09/98
Matrix: SO
Run Number: 0

ANALYSIS OF RESIDUAL VARIATIONS

	Model with Intercept			Model through Origin		
	Slope: 1.26920392			Slope: 1.25045621		
	Y Intercept: -634.03547					
	(SS)	(df)	(MS)	(SS)	(df)	(MS)
Residual:	.183E+010	13	.140E+009	.183E+010	14	.131E+009
Total Error:	0.00000000	1	0.00000000	0.00000000	1	0.00000000
Lack Of Fit:	.183E+010	13	.140E+009	.183E+010	14	.131E+009
	LOF F-Ratio(F): 0.00000000			LOF F-Ratio(F): 0.00000000		
	Critical 95% F: 4.67			Critical 95% F: 4.67		

ZERO INTERCEPT HYPOTHESIS

Calculated F: 0.03564144 Critical 95% F: 4.67

Zero Intercept Accepted

TABLE OF DATA POINTS

Targets: 15
Measurements per Target: 1

Target Values	Instrument Values
38700.0000	78222.0000

END OF LACK OF FIT TABLE

Attachment B

Subcontract Analytical Data Package

MORRISON KNUDSEN CORPORATION

CHAIN OF CUSTODY RECORD

COC #68

720 Park Blvd., P.O. Box 73
Boise, Idaho 83729
(208) 386-5000

Project No.: 4324-0009-007-34

Project Name: NSWC Crane Biofacility

Samplers: (Signature)

Sampler: (Print) Peter Chevalier

Split Samples

Analysis Required: 8330 EXPL.

Sample Type	Sampling Point Description	Sample Date	Time	Sample I.D. Number	Split Samples Yes	Split Samples No	8330 EXPL.	Remarks
Soil	MFA 1	10/3/97	0945	CRA/BPS AD1		✓	✓	
	MFA 2		0950	CRA/BPS AD2				
	MFA 3		~~0955~~ 1055	CRA/BPS AD3				
	MFA 4		1100	CRA/BPS AD4				
	MFA 5		1105	CRA/BPS AD5				
	MFA 6		1110	CRA/BPS AD6				
	MFA 7		1115	CRA/BPS AD7				
	MFA 8		1120	CRA/BPS AD8				
	MFA 9		1125	CRA/BPS AD9				
	Bldg 1 10		1130	CRA/BPS AD10				
	Bldg 1 12		1230	CRA/BPS AD12				
	Bldg 1 15		1235	CRA/BPS AD15				
	Bldg 1 16		1240	CRA/BPS AD16				
	Bldg 1 17		1245	CRA/BPS AD17				
	Borrow		1300	CRA/BPS AD18				

Relinquished By: (Signature) / Company:	Date/Time	Received By: (Signature) / Company:	Relinquished By: (Signature) / Company:	Date/Time	Received By: (Signature) / Company:
	10/3/97 1530				
Relinquished By: (Signature) / Company:	Date/Time	Received By: (Signature) / Company:	Relinquished By: (Signature) / Company:	Date/Time	Received By: (Signature) / Company:
Relinquished By: (Signature) / Company:	Date/Time	Received By: (Signature) / Company:	Received for Laboratory By: (Signature) / Company:	Date/Time 10/4/97 0930	Total No. Samples This Shipping Container: / Company:
Relinquished By: (Signature) / Company:	Date/Time	Received By: (Signature) / Company:	Relinquished By: (Signature) / Company:	Date/Time	Received By: (Signature) / Company:

Seal #s 5277 5278

3

SAMPLE LOG-IN SHEET

Lab Name: SOUTHWEST LABORATORY OF OKLAHOMA

Page 1 of 1

Received By (Print Name): KIM WILLISON/SARAH HODSON

Log-in Date: 10-14-97

Received By: (Signature): K Willison

Case Number:

Sample Delivery Group No:

SAS Number:

REMARKS:

1. Custody Seal(s) Present/Absent* Intact/Broken
2. Custody Seal Nos.: NA
3. Chain-of-Custody Records Present/Absent*
4. Traffic Reports or Packing List Present/Absent*
5. Airbill Airbill/Sticker Present/Absent*
5. Airbill No: 7900198816 OC
7. Sample Tags Present/Absent
   Sample Tag Numbers Listed/Not Listed on Chain-of-Custody
8. Sample Condition: Intact/Broken*/Leaking
9. Does information on custody records, traffic reports, and sample tags agree? Yes/No*
10. Date Received at Lab: 10-14-97
11. Time Received 0930

Sample Transfer

Fraction:

Area #:

By: KW KW

On:

EPA SAMPLE #	CORRESPONDING SAMPLE TAG #	CORRESPONDING ASSIGNED LAB #	REMARKS: CONDITION OF SAMPLE SHIPMENT, ETC.
CRAIBPSA01		31436-01	pH check 3:
CRAIBPSA01MS		-02	
CRAIBPSA01MSD		-03	
CRAIBPSA02		-04	
CRAIBPSA03		-05	
CRAIBPSA04		-06	
CRAIBPSA05		-07	
CRAIBPSA06		-08	
CRAIBPSA07		-09	
CRAIBPSA08		-10	
CRAIBPSA09		-11	
CRAIBPSA10		-12	
CRAIBPSA12		-13	
CRAIBPSA15		-14	
CRAIBPSA16 (A016)	KW 10-14	-15	
CRAIBPSA17 (A017)	KW 10-14	-16	
CRAIBPS ~~BOPRO~~ (AD18)	KW 10-14	-17	

* Contact SMO and attach record of resolution

Received By:

Date:

Logbook No:

Logbook Page No:

FORM DC-1

3/90

2E

EXPLOSIVE SURROGATE RECOVERY FORM

Lab Name: SWL-TULSA Contract:

Code: SWOK Case No: MKF-OH SDG No:31436

	SAMPLE NUMBER	S1 #	OTHER
1	CRA/BPSAD1 20000X		0
2	CRA/BPSAD1 MS 20000X		0
3	CRA/BPSAD1 MSD 20000		0
4	CRA/BPSAD2 100X	84.5	0
5	CRA/BPSAD3 10X	98.6	0
6	CRA/BPSAD4 10X	92.1	0
7	CRA/BPSAD5 20000X		0
8	CRA/BPSAD6 10X	95.9	0
9	CRA/BPSAD7 100X	88.1	0
10	CRA/BPSAD8 10X	96.7	0
11	CRA/BPSAD9 10X	97.6	0
12	CRA/BPSAD10 50X	93.2	0
13	CRA/BPSAD12 250X	100	0
14	CRA/BPSAD15 100X	96.7	0
15	CRA/BPSAD16 200X	140	0
16	CRA/BPSAD17 50X	95.9	0
17	CRA/BPSAD18 10X	97.7	0
18	CRA/BPSAD18	102	0
19	BL1014SC	97.1	0
20	LC1014SC	95.5	0
21	LD1014SC	96.6	0
22			
23			
24			
25			
26			
27			
28			
29			
30			

S1 = 3,4-DNT (60-140)

Column to be used to flag recovery values
* Values outside of QC limits
D Surrogates diluted out

FORM II

4

3F

SOIL EXPLOSIVE MATRIX SPIKE/MATRIX SPIKE DUPLICATE RECOVERY FORM

Name: SWL-TULSA Contract:

Lab Code: SWOK Case No: MKF-OH SDG No:31436

Client Sample No: CRA/BPSAD1 MSD 20000

COMPOUND	SPIKE ADDED (ug/Kg)	SAMPLE CONCENTRATION (ug/Kg)	MS CONCENTRATION (ug/Kg)	MS % REC #	QC LIMITS REC.
HMX	125000	11600000	15100000	2800*	54-128
RDX	125000	38700000	40700000	1600*	66-109
TNB	125000			0*	57-130
DNB	125000			0*	74-122
TETRYL	125000			0*	32-119
NB	125000			0*	70-135
TNT	125000	46200000	65400000	15400*	44-124
4ADNT	125000			0*	60-140
2ADNT	125000			0*	60-140
26DNT	125000			0*	58-125
24DNT	125000			0*	59-120
2NT	125000			0*	79-124
4NT	125000			0*	74-128
3NT	125000			0*	79-121

No of Analyte recoveries out of QC Advisory Limits = 14

COMPOUND	SPIKE ADDED (ug/Kg)	MSD CONCENTRATION (ug/Kg)	MSD % REC #	% RPD#	QC LIMITS RPD	QC LIMITS REC.
HMX	125000	18800000	5760*	69*	30	54-128
RDX	125000	50600000	9520*	142*	30	66-109
TNB	125000		0*	100*	30	57-130
DNB	125000		0*	100*	45	74-122
TETRYL	125000		0*	100*	30	32-119
NB	125000		0*	100*	35	70-135
TNT	125000	77000000	24600*	46*	30	44-124
4ADNT	125000		0*	100*	40	60-140
2ADNT	125000		0*	100*	40	60-140
26DNT	125000		0*	100*	35	58-125
24DNT	125000		0*	100*	35	59-120
2NT	125000		0*	100*	35	79-124
4NT	125000		0*	100*	35	74-128
3NT	125000		0*	100*	35	79-121

No of Analyte recoveries out of QC Advisory Limits = 14
No of RPD's out of QC Limits 14

3G

SOIL EXPLOSIVE CONTROL SPIKE RECOVERY FORM

Name: SWL-TULSA Contract:

Lab Code: SWOK Case No: MKF-OH SDG No:31436

Client Sample No: LC1014SC

COMPOUND	SPIKE ADDED (ug/Kg)	SAMPLE CONCENTRATION (ug/Kg)	LCS CONCENTRATION (ug/Kg)	LCS % REC #	QC LIMITS REC.
HMX	32000		32500	102	57-137
RDX	12500		13600	109	77-113
TNB	11500		11200	97	64-125
DNB	9750		8420	86	82-118
TETRYL	7650		1410	18*	23-124
NB	7000		7410	106	69-143
TNT	10000		8560	86	75-118
4ADNT	9000		10400	116	60-140
2ADNT	8000		7570	95	60-140
26DNT	7750		7640	99	78-117
24DNT	4800		5000	104	74-126
2NT	14500		15900	110	78-123
4NT	9500		10400	109	79-125
3NT	10000		10600	106	78-121

No of Analyte recoveries out of QC Advisory Limits = 1

3G

SOIL EXPLOSIVE CONTROL SPIKE RECOVERY FORM

I Name: SWL-TULSA Contract:

Lab Code: SWOK Case No: MKF-OH SDG No:31436

Client Sample No: LD1014SC

COMPOUND	SPIKE ADDED (ug/Kg)	SAMPLE CONCENTRATION (ug/Kg)	LCSD CONCENTRATION (ug/Kg)	LCSD % REC #	QC LIMITS REC.
HMX	32000		32600	102	57-137
RDX	12500		13900	111	77-113
TNB	11500		10900	95	64-125
DNB	9750		8590	88	82-118
TETRYL	7650		968	13*	23-124
NB	7000		7480	107	69-143
TNT	10000		8840	88	75-118
4ADNT	9000		11300	126	60-140
2ADNT	8000		7830	98	60-140
26DNT	7750		7920	102	78-117
24DNT	4800		5120	107	74-126
2NT	14500		16000	110	78-123
4NT	9500		10400	109	79-125
3NT	10000		10800	108	78-121

N. of Analyte recoveries out of QC Advisory Limits = 1

4C

Lab Name: SWL-TULSA Contract:

Sample ID: BL1014SC

Lab Code: SWOK Case No: MKF-OH SDG No:31436

Matrix: (soil/water) Soil

Date Extracted: 10/14/97 Extn. Method: SONC

Date Analyzed (1): 10/17/97 Date Analyzed(2):

Time Analyzed (1): 22:25:00 Time Analyzed(2):

Instrument ID (1): IN13 Instrument ID(2):

LC Column ID (1): CARB-05 LC Column ID (2):

THIS METHOD BLANK APPLIES TO THE FOLLOWING SAMPLES AND QC'S.

	CUSTOMER SAMPLE NO.	LAB SAMPLE ID	DATE ANALYZED 1	DATE ANALYZED 2
1	CRA/BPSAD2 100X	31436.04	10/18/97	11/11/97
2	CRA/BPSAD7 100X	31436.09	10/18/97	10/25/97
3	LC1014SC	LC1014SC	10/18/97	
4	LD1014SC	LD1014SC	10/18/97	
5	CRA/BPSAD1 20000X	31436.01	10/18/97	10/25/97
6	CRA/BPSAD1 MS 20000X	31436.02MS	10/18/97	
7	CRA/BPSAD1 MSD 20000	31436.03MSD	10/18/97	
8	CRA/BPSAD4 10X	31436.06	10/19/97	10/25/97
9	CRA/BPSAD5 20000X	31436.07	10/19/97	10/25/97
10	CRA/BPSAD6 10X	31436.08	10/19/97	10/25/97
11	CRA/BPSAD8 10X	31436.10	10/19/97	10/28/97
12	CRA/BPSAD3 10X	31436.05	10/24/97	10/25/97
13	CRA/BPSAD9 10X	31436.11	10/24/97	10/28/97
14	CRA/BPSAD10 50X	31436.12	10/24/97	10/28/97
15	CRA/BPSAD12 250X	31436.13	10/24/97	10/28/97
16	CRA/BPSAD15 100X	31436.14	10/24/97	10/28/97
17	CRA/BPSAD16 200X	31436.15	10/24/97	10/28/97
18	CRA/BPSAD17 50X	31436.16	10/25/97	10/28/97
19	CRA/BPSAD18	31436.17	11/06/97	10/28/97
20				
21				
22				
23				
24				
25				

FORM IV

1D

EXPLOSIVE ANALYSIS DATA SHEET

EPA SAMPLE NO:

CRA/BPSAD1 ~~2000~~ ~~0X~~ 20000X 11/30/97

Lab Name: SWL-TULSA Contract:

Lab Code: SWOK Case No:MKF-OH SDG No: 31436

Matrix: (soil/water) SOIL Lab Sample ID: 31436.01

Sample Amt: 2gm % Moisture 11.31 Date Received: 10/14/97

Extraction Volume: 10ml Date Extracted: 10/14/97

Extraction Method: SONC Date Analyzed: 10/18/97

GPC Cleanup: (Y/N) N Dilution Factor: 20000.00

CAS NO.	COMPOUND	CONCENTRATION UNITS: (ug/L or ug/kg) ug/kg	Q
2691-41-0	HMX	11600000	J
121-82-4	RDX	38700000	
99-35-4	TNB	2500000	U
99-65-0	DNB	2500000	U
479-45-8	TETRYL	6500000	U
98-95-3	NB	2600000	U
118-96-7	TNT	46200000	
1946-51-0	4ADNT	2500000	U
35572-78-2	2ADNT	2500000	U
606-20-2	26DNT	2600000	U
121-14-2	24DNT	2500000	U
88-72-2	2NT	2500000	U
99-99-0	4NT	2500000	U
99-08-1	3NT	2500000	U

SURROGATE AMOUNT SPIKED (ug/kg dry wt.) 20000
3,4-DNT

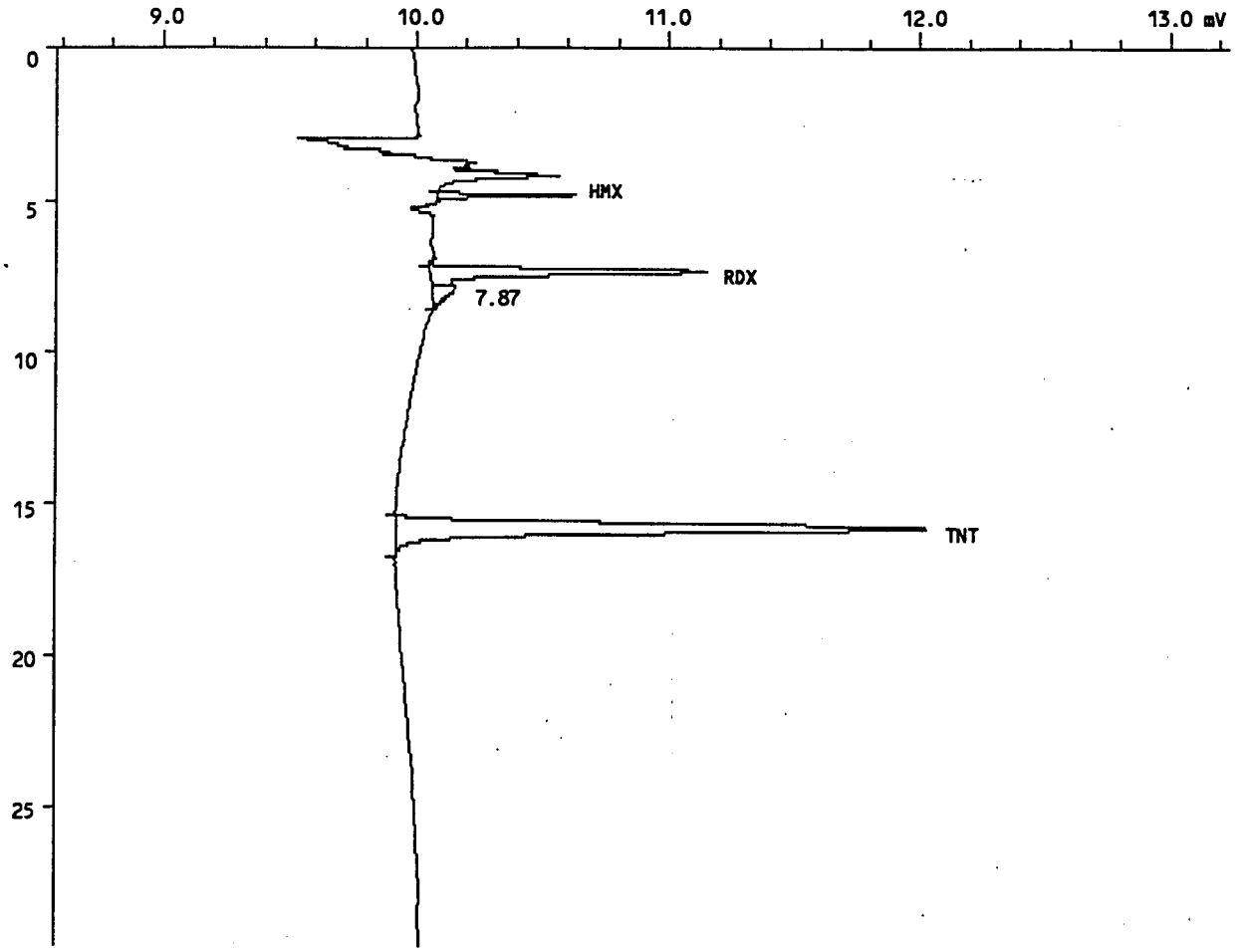
FORM I

LONG PLOT

Injection F: <MC3> 5 5EX1015D,5,1

Sample name.....: CRA/BPSAD1 20000X
Sample ID.....: 31436.01
INST ID: IN13, COL. TYPE: ODS-II, ID: CARB-05, INJ:200,COL#1

Acquired on 18-Oct-97 at 19:30:37
Reported on 05-Nov-97 at 12:29:11



INJECTION REPORT

Injection F: <MC3> 5 5EX1015D,5,1

Acquired on 18-Oct-97 at 19:30:37
Modified on 05-Nov-81 at 12:27:44
Reported on 05-Nov-97 at 12:28:54

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN13, COL. TYPE: ODS-II, ID: CARB-05, INJ:200,COL#1
Number of samples.: 26
Calibration file..: 5EX1015 Last modified on 05-Nov-81 at 11:22:30
Method file.....: EXPLOS Last modified on 05-Nov-81 at 08:54:26
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: CRA/BPSAD1 20000X
Sample ID.....: 31436.01
Sample type.....: Sample
Sample amount.....: 2.0000
Number of injections.....: 1
Bottle Number.....: 1

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 20.000
Dilution.....: 20000.000
Amount (g or mL).....: 2.000

PEAK INFORMATION

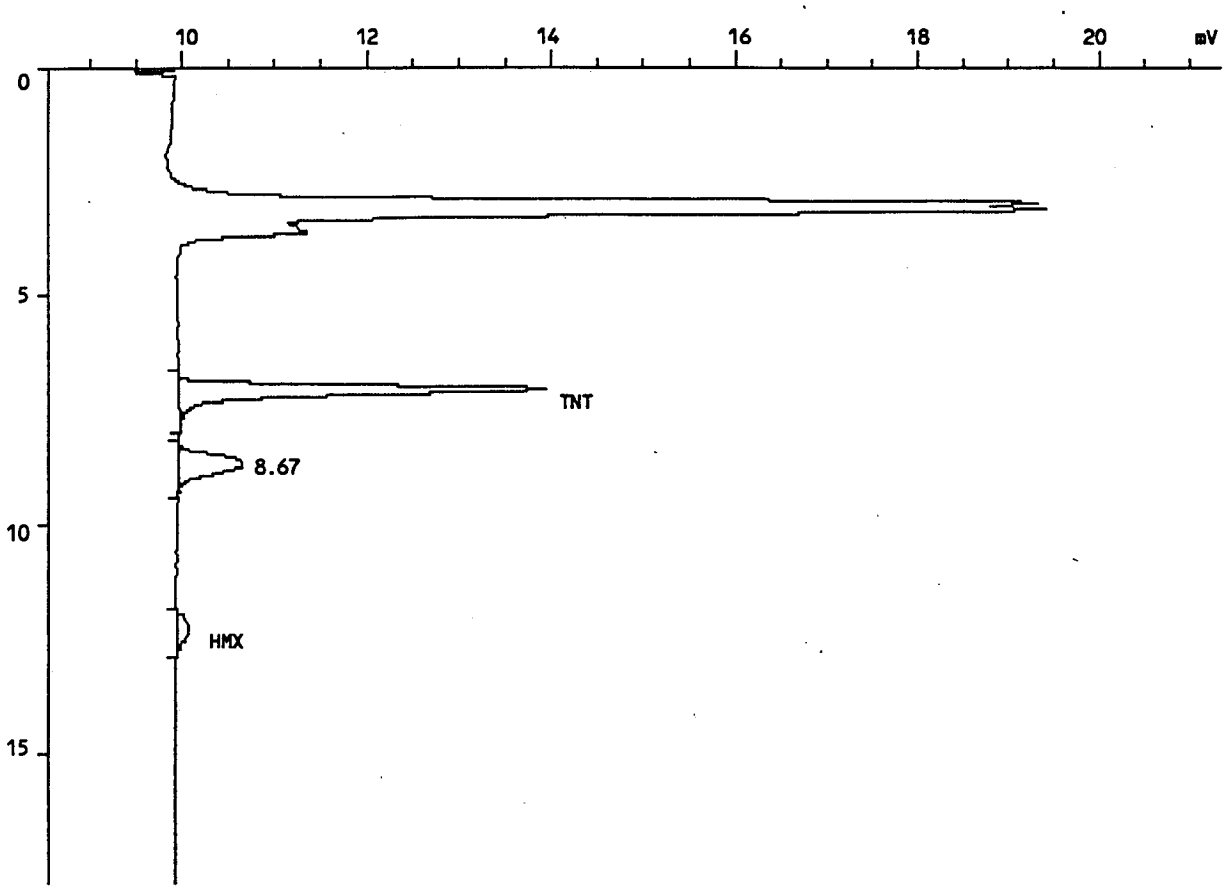
Peak	RT mins	Hght uV	Area uVs	ug/Kg	Peak name
1	4.837	544	4049	11633887.000	HMX*
2	7.355	1103	16613	38724740.000	RDX*
4	15.787	2110	45371	46249972.000	TNT*
Total		3757	66033	96608600.000	
Residual		91	2494	5813978.500	

LONG PLOT

Injection F: <MC3> 2 2CN1024,16,1

Sample name.....: CRA/BPSAD1 20000X
Sample ID.....: 31436.01
INST ID: IN12, COL. TYPE: LC-CN, ID: LC-CN-05, INJ:100, UV

Acquired on 25-Oct-97 at 03:38:11
Reported on 05-Nov-97 at 12:03:54



INJECTION REPORT

Injection F: <MC3> 2 2CN1024,16,1

Acquired on 25-Oct-97 at 03:38:11
Modified on 05-Nov-81 at 11:59:18
Reported on 05-Nov-97 at 11:59:18

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN12, COL. TYPE: LC-CN, ID: LC-CN-05, INJ:100, UV
Number of samples.: 67
Calibration file..: 2CN1024A Last modified on 05-Nov-81 at 11:54:16
Method file.....: LCCN Last modified on 05-Nov-81 at 11:58:32
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: CRA/BPSAD1 20000X
Sample ID.....: 31436.01
Sample type.....: Sample
Sample amount.....: 2.0000
Number of injections.....: 1
Bottle Number.....: 1

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 20.000
Dilution.....: 20000.000
Amount (g or mL).....: 2.000

PEAK INFORMATION

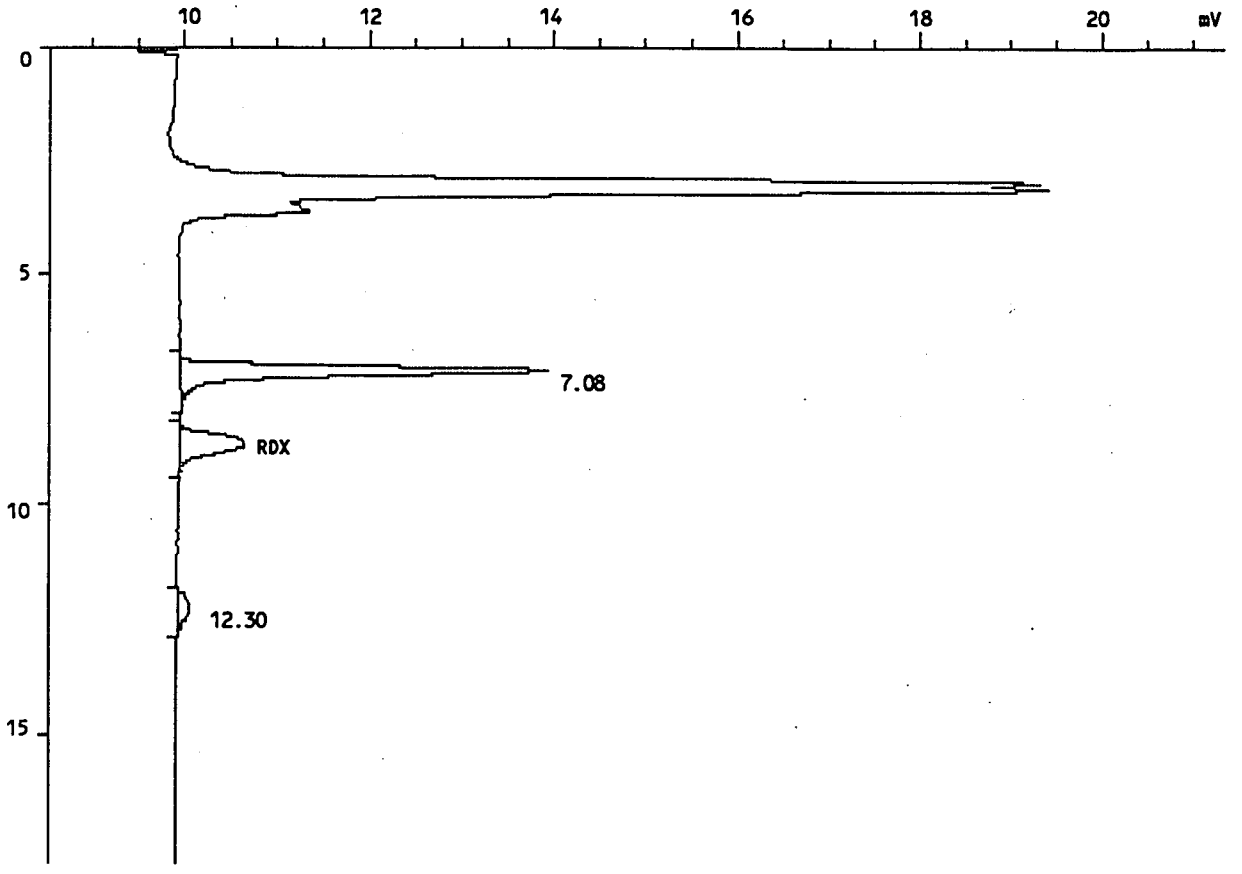
Peak	RT mins	Hght uV	Area uVs	ug/Kg	Peak name
1	7.083	3975	60753	47004160.000	TNT
3	12.299	125	4053	10737363.000	HMX
Total		4100	64806	57741524.000	
Residual		699	20191	0.000	

LONG PLOT

Injection F: <MC3> 2 2CN1024,16,1

Sample name.....: CRA/BPSAD1 20000X
Sample ID.....: 31436.01
INST ID: IN12, COL. TYPE: LC-CN, ID: LC-CN-05, INJ:100, UV

Acquired on 25-Oct-97 at 03:38:11
Reported on 05-Nov-97 at 12:20:03



INJECTION REPORT

Injection F: <MC3> 2 2CN1024,16,1

Acquired on 25-Oct-97 at 03:38:11
Modified on 05-Nov-81 at 12:05:38
Reported on 05-Nov-97 at 12:05:38

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN12, COL. TYPE: LC-CN, ID: LC-CN-05, INJ:100, UV
Number of samples.: 67
Calibration file..: 2CN1024B Last modified on 05-Nov-81 at 11:56:18
Method file.....: LCCN Last modified on 05-Nov-81 at 12:04:50
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: CRA/BPSAD1 20000X
Sample ID.....: 31436.01
Sample type.....: Sample
Sample amount.....: 2.0000
Number of injections.....: 1
Bottle Number.....: 1

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 20.000
Dilution.....: 20000.000
Amount (g or mL).....: 2.000

PEAK INFORMATION

Peak	RT mins	Hght uV	Area uVs	ug/Kg	Peak name
2	8.667	699	20191	34251232.000	RDX
Total		699	20191	34251232.000	
Residual		4100	64806	0.000	

1D

EXPLOSIVE ANALYSIS DATA SHEET

EPA SAMPLE NO:

CRA/BPSAD2 100X

b Name: SWL-TULSA Contract:

Lab Code: SWOK Case No:MKF-OH SDG No: 31436

Matrix: (soil/water) SOIL Lab Sample ID: 31436.04

Sample Amt: 2gm % Moisture 12.81 Date Received: 10/14/97

Extraction Volume: 10ml Date Extracted: 10/14/97

Extraction Method: SONC Date Analyzed: 10/18/97

GPC Cleanup: (Y/N) N Dilution Factor: 100.00

CAS NO.	COMPOUND	CONCENTRATION UNITS: (ug/L or ug/kg) ug/kg	Q
2691-41-0	HMX	51500	J
121-82-4	RDX	286000	
99-35-4	TNB	12500	U
99-65-0	DNB	12500	U
479-45-8	TETRYL	32500	U
98-95-3	NB	13000	U
118-96-7	TNT	91400	
1946-51-0	4ADNT	6930	J
35572-78-2	2ADNT	12500	U
606-20-2	26DNT	13000	U
121-14-2	24DNT	12500	U
88-72-2	2NT	12500	U
99-99-0	4NT	12500	U
99-08-1	3NT	12500	U

SURROGATE AMOUNT SPIKED (ug/kg dry wt.) 20000
3,4-DNT

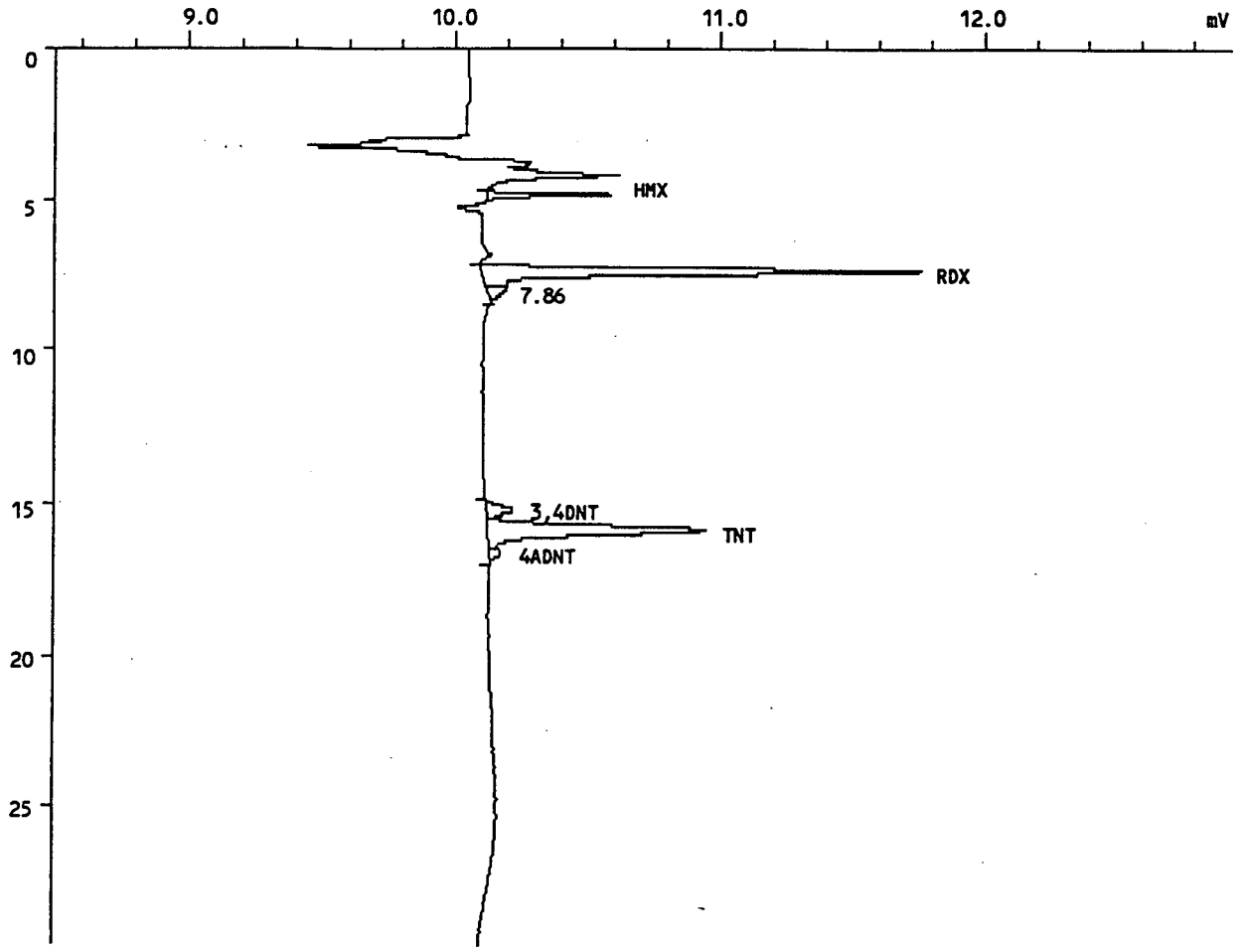
FORM I

LONG PLOT

Injection F: <MC3> 5 5EX1015C,8,1

Sample name.....: CRA/BPSAD2 100X
Sample ID.....: 31436.04
INST ID: IN13, COL. TYPE: ODS-II, ID: CARB-05, INJ:200,COL#1

Acquired on 18-Oct-97 at 02:08:29
Reported on 05-Nov-97 at 15:09:34



INJECTION REPORT

Injection F: <MC3> 5 5EX1015C,8,1

Acquired on 18-Oct-97 at 02:08:29
Modified on 05-Nov-81 at 15:09:10
Reported on 05-Nov-97 at 15:09:18

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN13, COL. TYPE: ODS-II, ID: CARB-05, INJ:200,COL#1
Number of samples.: 27
Calibration file..: 5EX1015 Last modified on 05-Nov-81 at 11:22:30
Method file.....: EXPLOS Last modified on 05-Nov-81 at 08:54:26
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: CRA/BPSAD2 100X
Sample ID.....: 31436.04
Sample type.....: Sample
Sample amount.....: 2.0000
Number of injections.....: 1
Bottle Number.....: 1

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 20.000
Dilution.....: 100.000
Amount (g or mL).....: 2.000

PEAK INFORMATION

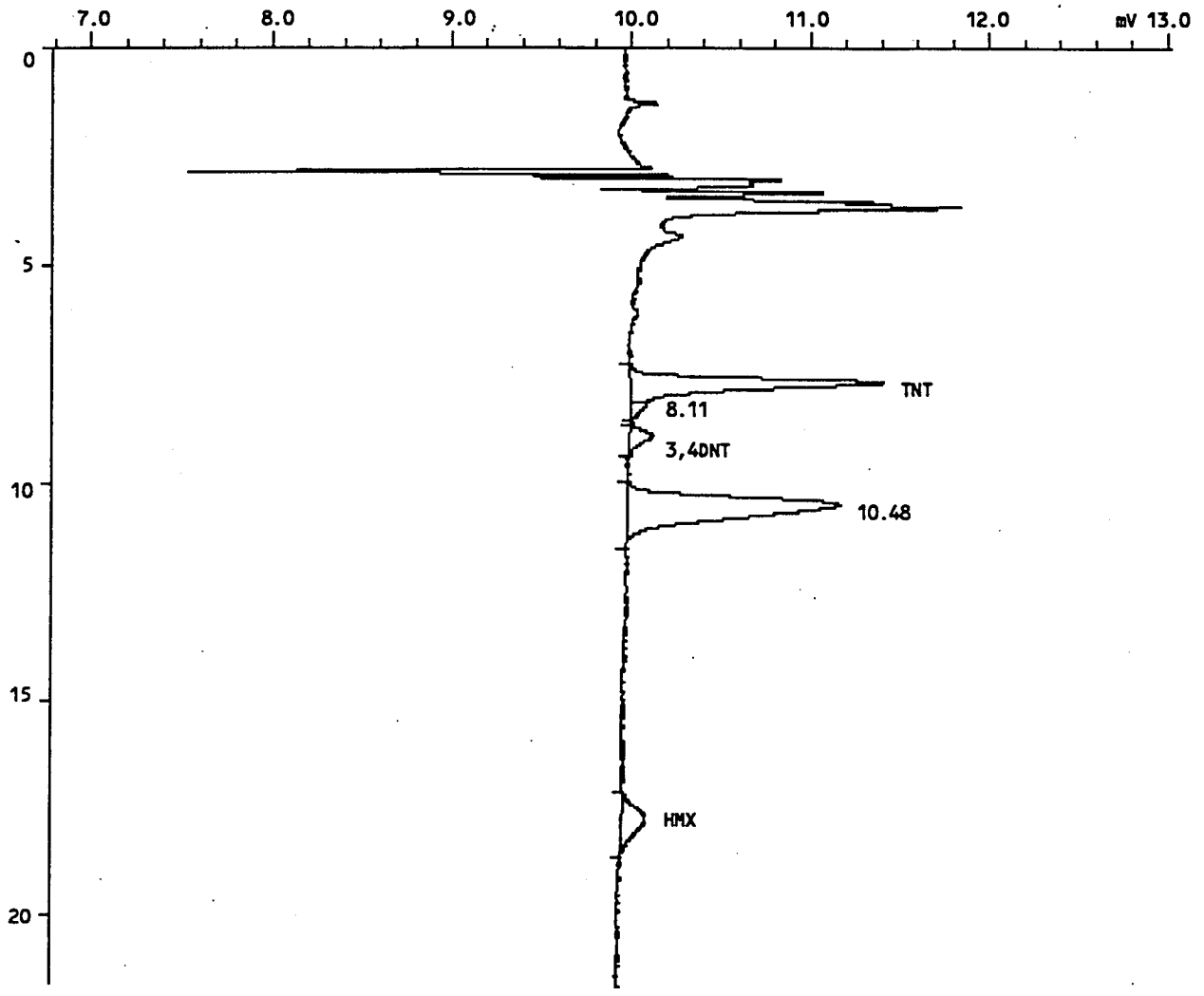
Peak	RT mins	Hght uV	Area uVs	ug/Kg	Peak name
1	4.859	474	3586	51524.742	HMX*
2	7.397	1657	24549	286120.094	RDX*
4	15.216	105	1954	16906.143	3,4DNT 84
5	15.840	830	17935	91411.500	TNT*
6	16.608	47	983	6930.034	4ADNT
Total		3113	49007	452892.531	
Residual		78	1683	19613.213	

LONG PLOT

Injection F: <MC3> 3 3CN1110B,4,1

Sample name.....: CRA/BPSAD2 100X
Sample ID.....: 31436.04
INST ID: IN10, COL. TYPE: LC-CN, ID: LC-CN-07, INJ:100, UV

Acquired on 11-Nov-97 at 19:54:01
Reported on 25-Nov-97 at 11:54:38



INJECTION REPORT

Injection F: <MC3> 3 3CN1110B,4,1

Acquired on 11-Nov-97 at 19:54:01
Modified on 25-Nov-81 at 11:53:40
Reported on 25-Nov-97 at 11:53:51

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN10, COL. TYPE: LC-CN, ID: LC-CN-07, INJ:100, UV
Number of samples.: 34
Calibration file..: 3CN1110A Last modified on 21-Nov-81 at 09:39:12
Method file.....: LCCN Last modified on 25-Nov-81 at 10:33:36
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: CRA/BPSAD2 100X
Sample ID.....: 31436.04
Sample type.....: Sample
Sample amount.....: 2.0000
Number of injections.....: 1
Bottle Number.....: 1

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 20.000
Dilution.....: 100.000
Amount (g or mL).....: 2.000

PEAK INFORMATION

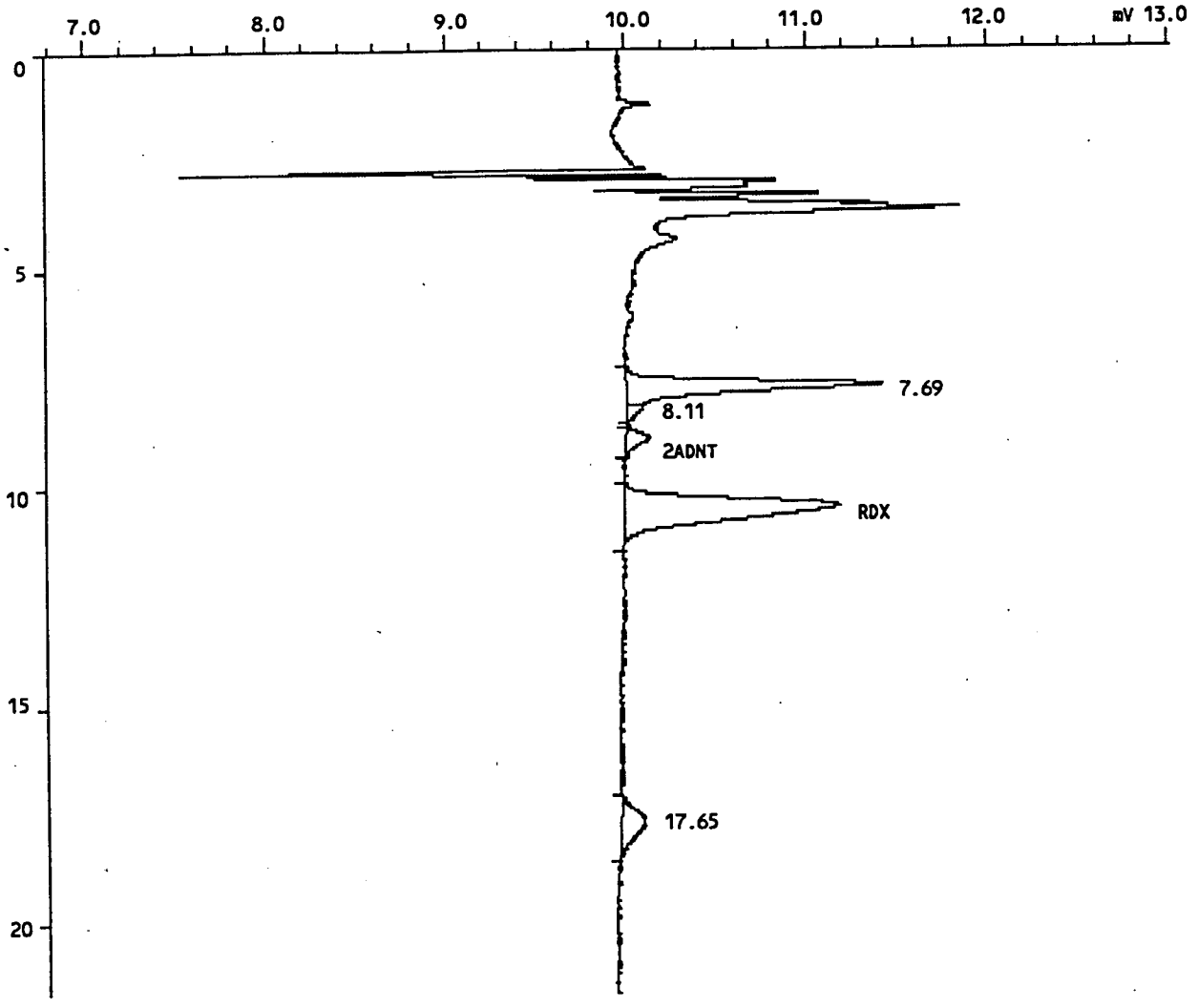
Peak	RT mins	Hght uV	Area uVs	ug/Kg	Peak name
1	7.685	1425	24447	98300.898	TNT
3	8.923	125	2500	16059.464	3,4DNT
5	17.653	133	5535	52316.688	HMX
Total		1683	32483	166677.047	
Residual		1288	38439	0.000	

LONG PLOT

Injection F: <MC3> 3 3CN1110B,4,1

Sample name.....: CRA/BPSAD2 100X
Sample ID.....: 31436.04
INST ID: IN10, COL. TYPE: LC-CN, ID: LC-CN-07, INJ:100, UV

Acquired on 11-Nov-97 at 19:54:01
Reported on 25-Nov-97 at 11:57:13



INJECTION REPORT

Injection F: <MC3> 3 3CN1110B,4,1

Acquired on 11-Nov-97 at 19:54:01
Modified on 25-Nov-81 at 11:56:52
Reported on 25-Nov-97 at 11:56:59

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN10, COL. TYPE: LC-CN, ID: LC-CN-07, INJ:100, UV
Number of samples.: 34
Calibration file..: 3CN1110B Last modified on 21-Nov-81 at 09:38:30
Method file.....: LCCN Last modified on 25-Nov-81 at 10:33:36
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: CRA/BPSAD2 100X
Sample ID.....: 31436.04
Sample type.....: Sample
Sample amount.....: 2.0000
Number of injections.....: 1
Bottle Number.....: 1

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 20.000
Dilution.....: 100.000
Amount (g or mL).....: 2.000

PEAK INFORMATION

Peak	RT mins	Hght uV	Area uVs	ug/Kg	Peak name
3	8.923	125	2500	9938.229	2ADNT
4	10.480	1191	37018	289653.188	RDX
Total		1316	39518	299591.406	
Residual		1656	31404	0.000	

1D

EXPLOSIVE ANALYSIS DATA SHEET

EPA SAMPLE NO:

CRA/BPSAD3 10X

b Name: SWL-TULSA Contract:

Lab Code: SWOK Case No:MKF-OH SDG No: 31436

Matrix: (soil/water) SOIL Lab Sample ID: 31436.05

Sample Amt: 2gm % Moisture 14.51 Date Received: 10/14/97

Extraction Volume: 10ml Date Extracted: 10/14/97

Extraction Method: SONC Date Analyzed: 10/24/97

GPC Cleanup: (Y/N) N Dilution Factor: 10.00

CAS NO.	COMPOUND	CONCENTRATION UNITS: (ug/L or ug/kg) ug/kg	Q
2691-41-0	HMX	36200	
121-82-4	RDX	55400	
99-35-4	TNB	989	J
99-65-0	DNB	1250	U
479-45-8	TETRYL	3250	U
98-95-3	NB	1300	U
118-96-7	TNT	15400	
1946-51-0	4ADNT	5470	
35572-78-2	2ADNT	1250	U
606-20-2	26DNT	1300	U
121-14-2	24DNT	1250	U
88-72-2	2NT	1250	U
99-99-0	4NT	1250	U
99-08-1	3NT	1250	U

SURROGATE AMOUNT SPIKED (ug/kg dry wt.) 20000
3,4-DNT

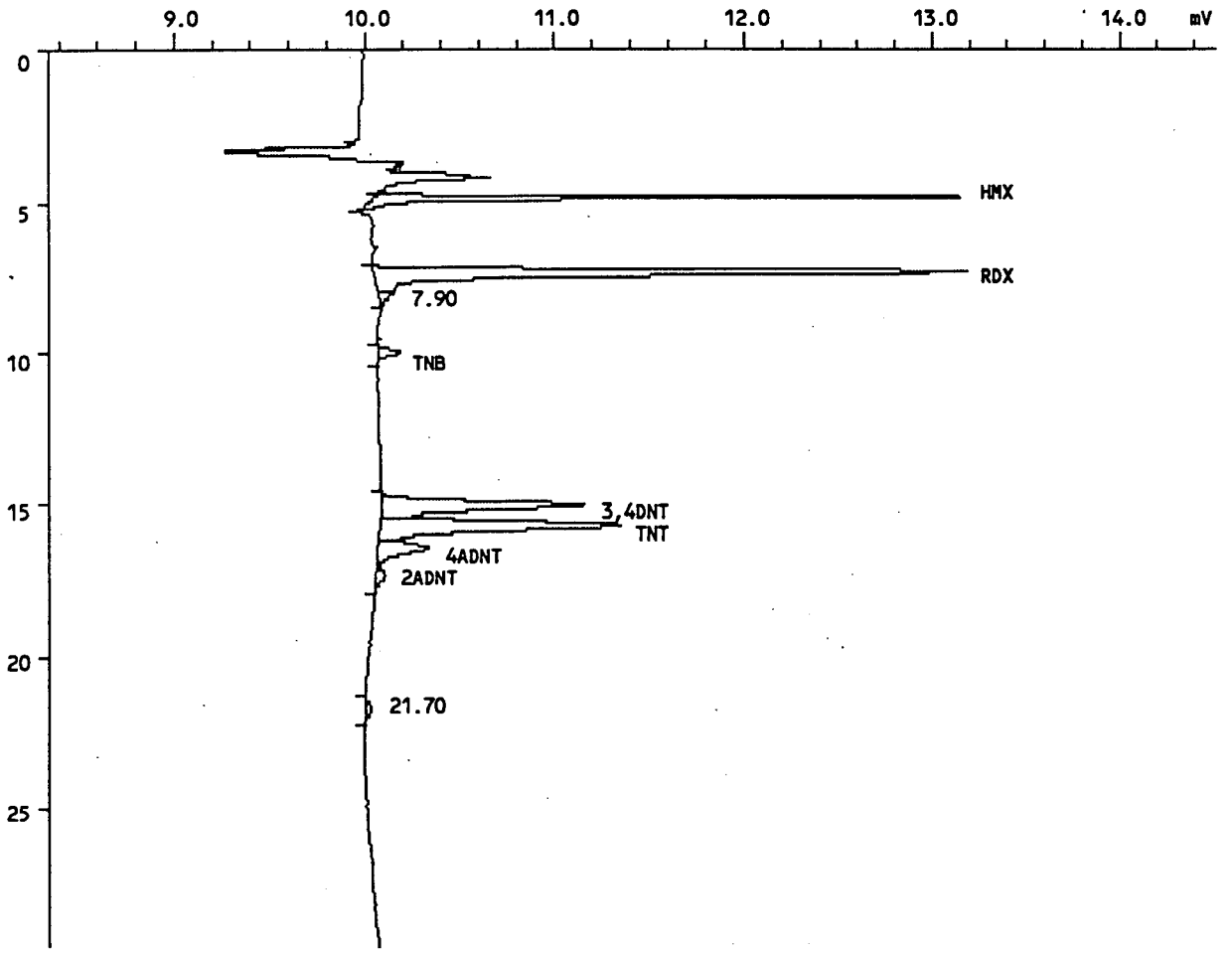
FORM I

LONG PLOT

Injection F: <MC3> 5 5EX1021E,3,1

Sample name.....: CRA/BPSAD3 10X
Sample ID.....: 31436.05
INST ID: IN13, COL. TYPE: ODS-II, ID: CARB-05, INJ:200,COL#1

Acquired on 24-Oct-97 at 19:37:09
Reported on 05-Nov-97 at 08:48:59



INJECTION REPORT

Injection F: <MC3> 5 5EX1021E,3,1

Acquired on 24-Oct-97 at 19:37:09
Modified on 05-Nov-81 at 08:44:12
Reported on 05-Nov-97 at 08:44:13

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN13, COL. TYPE: ODS-II, ID: CARB-05, INJ:200,COL#1
Number of samples.: 34
Calibration file..: 5EX1021 Last modified on 30-Oct-81 at 18:08:22
Method file.....: EXPLOS Last modified on 05-Nov-81 at 08:43:52
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: CRA/BPSAD3 10X
Sample ID.....: 31436.05
Sample type.....: Sample
Sample amount.....: 2.0000
Number of injections.....: 1
Bottle Number.....: 1

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 20.000
Dilution.....: 10.000
Amount (g or mL).....: 2.000

PEAK INFORMATION

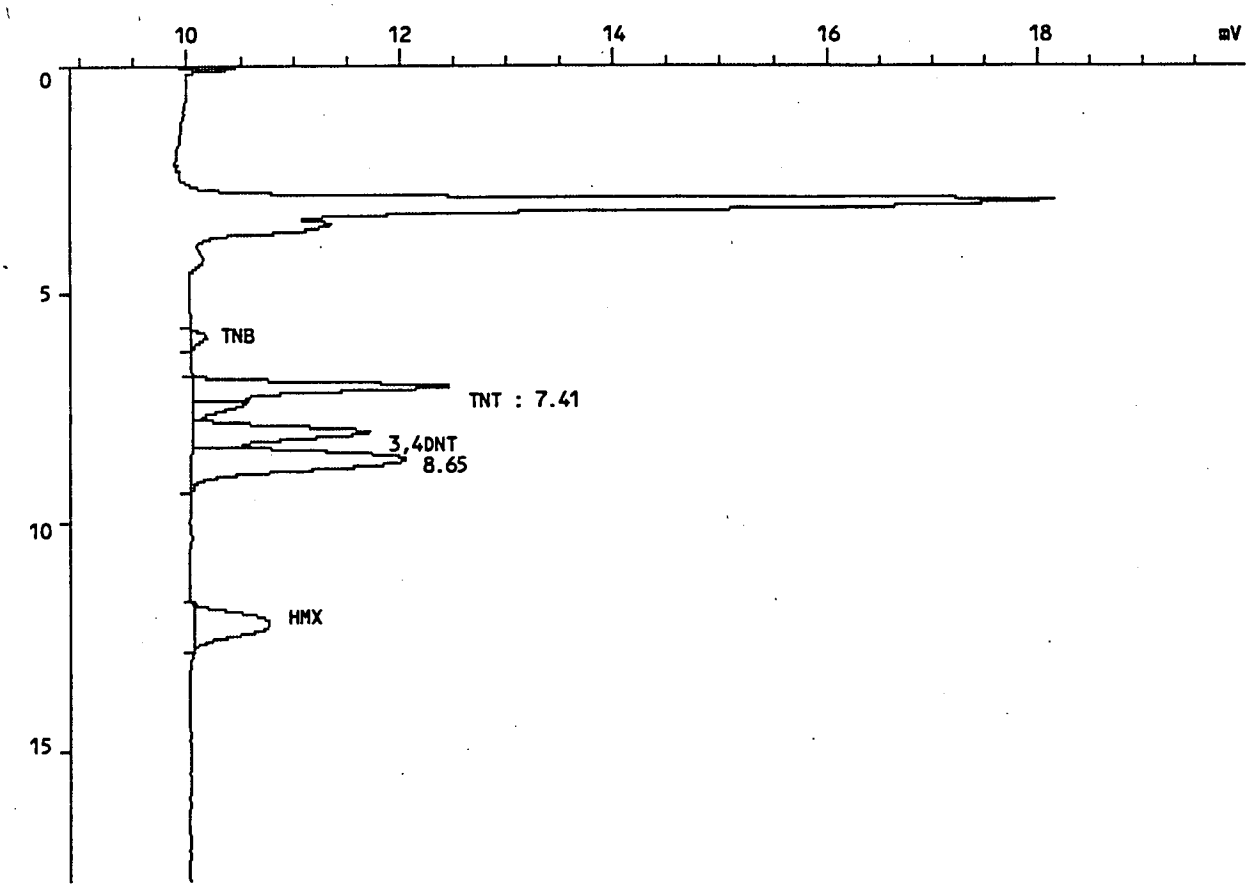
Peak	RT mins	Hght uV	Area uVs	ug/Kg	Peak name
1	4.853	3106	24716	36240.262	HMX*
2	7.365	3146	46977	55397.117	RDX*
4	9.979	123	1851	988.630	TNB*
5	15.067	1083	21962	19714.717	3,4DNT*
6	15.728	1287	28065	15403.521	TNT*
7	16.421	266	6816	5470.366	4ADNT*
8	17.280	47	1204	672.137	2ADNT
Total		9058	131591	133886.750	
Residual		122	2309	2140.049	

LONG PLOT

Injection F: <MC3> 2 2CN1024,18,1

Sample name.....: CRA/BPSAD3 10X
Sample ID.....: 31436.05
INST ID: IN12, COL. TYPE: LC-CN, ID: LC-CN-05, INJ:100, UV

Acquired on 25-Oct-97 at 04:42:42
Reported on 05-Nov-97 at 12:03:13



INJECTION REPORT

Injection F: <MC3> 2 2CN1024,18,1

Acquired on 25-Oct-97 at 04:42:42
Modified on 05-Nov-81 at 11:59:46
Reported on 05-Nov-97 at 11:59:45

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN12, COL. TYPE: LC-CN, ID: LC-CN-05, INJ:100, UV
Number of samples.: 67
Calibration file..: 2CN1024A Last modified on 05-Nov-81 at 11:54:16
Method file.....: LCCN Last modified on 05-Nov-81 at 11:58:32
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: CRA/BPSAD3 10X
Sample ID.....: 31436.05
Sample type.....: Sample
Sample amount.....: 2.0000
Number of injections.....: 1
Bottle Number.....: 1

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 20.000
Dilution.....: 10.000
Amount (g or mL).....: 2.000

PEAK INFORMATION

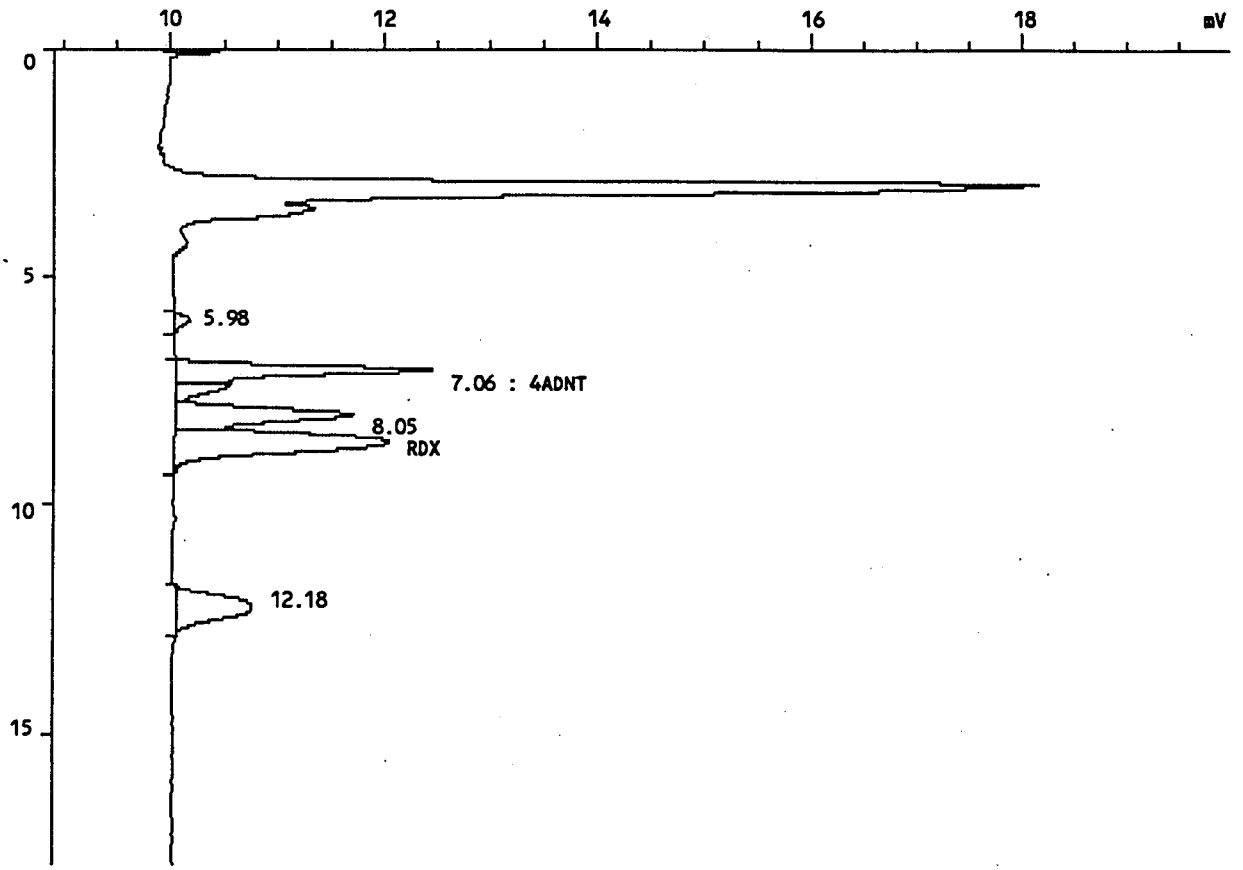
Peak	RT mins	Hght uV	Area uVs	ug/Kg	Peak name
1	5.979	147	2139	934.029	TNB
2	7.061	2416	35434	13707.490	TNT
4	8.048	1664	32738	18559.006	3,4DNT
6	12.181	714	23330	30901.182	HMX
Total		4940	93641	64101.703	
Residual		2521	65038	0.000	

LONG PLOT

Injection F: <MC3> 2 2CN1024,18,1

Sample name.....: CRA/BPSAD3 10X
Sample ID.....: 31436.05
INST ID: IN12, COL. TYPE: LC-CN, ID: LC-CN-05, INJ:100, UV

Acquired on 25-Oct-97 at 04:42:42
Reported on 05-Nov-97 at 12:19:08



INJECTION REPORT

Injection F: <MC3> 2 2CN1024,18,1

Acquired on 25-Oct-97 at 04:42:42
Modified on 05-Nov-81 at 12:06:04
Reported on 05-Nov-97 at 12:06:05

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN12, COL. TYPE: LC-CN, ID: LC-CN-05, INJ:100, UV
Number of samples.: 67
Calibration file..: 2CN1024B Last modified on 05-Nov-81 at 11:56:18
Method file.....: LCCN Last modified on 05-Nov-81 at 12:04:50
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: CRA/BPSAD3 10X
Sample ID.....: 31436.05
Sample type.....: Sample
Sample amount.....: 2.0000
Number of injections.....: 1
Bottle Number.....: 1

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 20.000
Dilution.....: 10.000
Amount (g or mL).....: 2.000

PEAK INFORMATION

Peak	RT mins	Hght uV	Area uVs	ug/Kg	Peak name
3	7.408	511	8596	5535.136	4ADNT
5	8.645	2010	56442	47872.523	RDX
Total		2521	65038	53407.660	
Residual		4940	93641	0.000	

1D

EXPLOSIVE ANALYSIS DATA SHEET

EPA SAMPLE NO:

CRA/BPSAD4 10X

Lab Name: SWL-TULSA Contract:

Lab Code: SWOK Case No:MKF-OH SDG No: 31436

Matrix: (soil/water) SOIL Lab Sample ID: 31436.06

Sample Amt: 2gm % Moisture 15.58 Date Received: 10/14/97

Extraction Volume: 10ml Date Extracted: 10/14/97

Extraction Method: SONC Date Analyzed: 10/19/97

GPC Cleanup: (Y/N) N Dilution Factor: 10.00

CAS NO.	COMPOUND	CONCENTRATION UNITS: (ug/L or ug/kg) ug/kg	Q
2691-41-0	HMX	49400	
121-82-4	RDX	237000	
99-35-4	TNB	254	PJ
99-65-0	DNB	1250	U
479-45-8	TETRYL	3250	U
98-95-3	NB	1300	U
118-96-7	TNT	60800	
1946-51-0	4ADNT	5500	
35572-78-2	2ADNT	1250	U
606-20-2	26DNT	1300	U
121-14-2	24DNT	1250	U
88-72-2	2NT	1250	U
99-99-0	4NT	1250	U
99-08-1	3NT	1250	U

SURROGATE AMOUNT SPIKED (ug/kg dry wt.) 20000
3,4-DNT

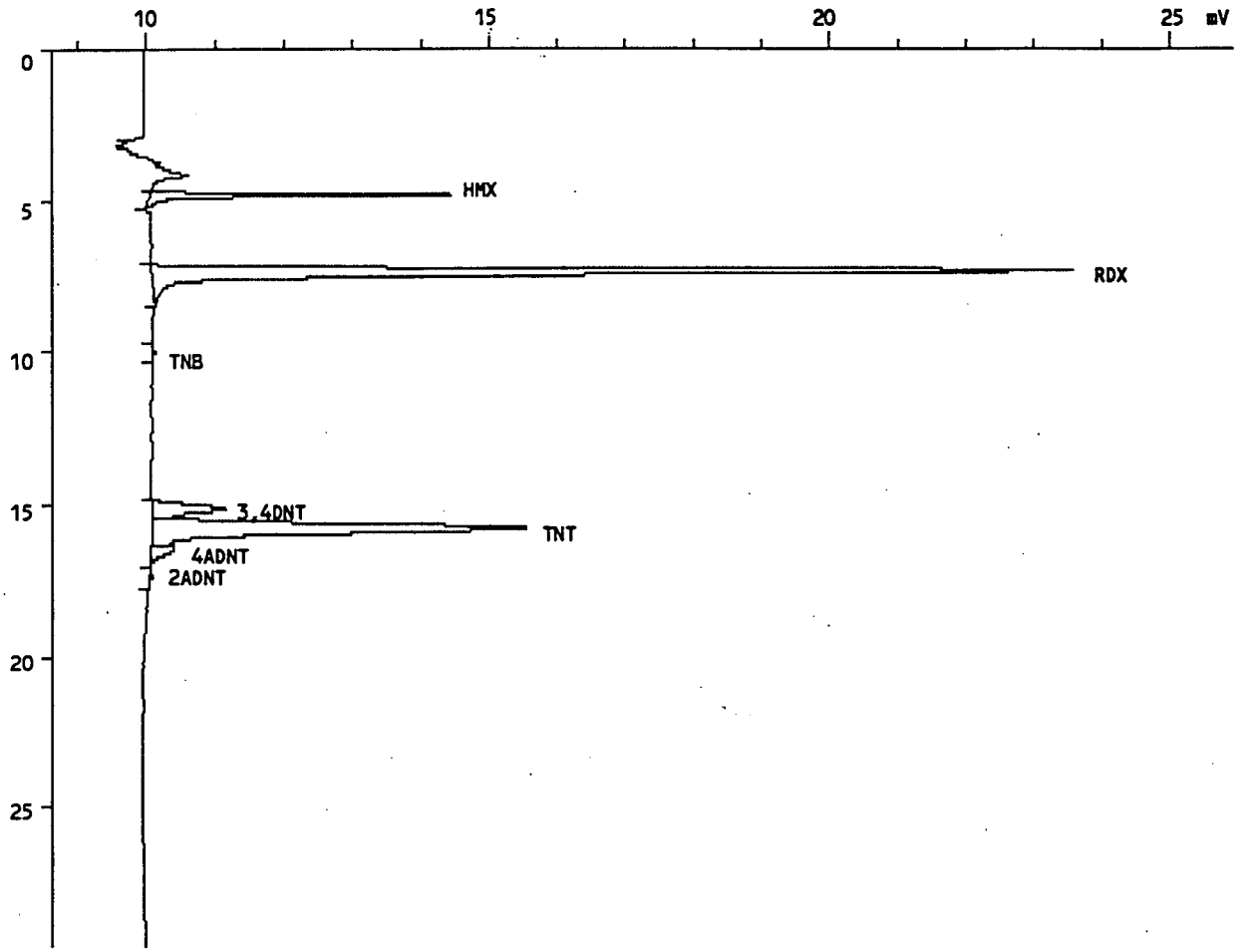
FORM I

LONG PLOT

Injection F: <MC3> 5 5EX1015D,12,1

Sample name.....: CRA/BPSAD4 10X
Sample ID.....: 31436.06
INST ID: IN13, COL. TYPE: ODS-II, ID: CARB-05, INJ:200,COL#1

Acquired on 19-Oct-97 at 00:43:28
Reported on 05-Nov-97 at 10:05:19



INJECTION REPORT

Injection F: <MC3> 5 5EX1015D,12,1

Acquired on 19-Oct-97 at 00:43:28
Modified on 05-Nov-81 at 10:04:48
Reported on 05-Nov-97 at 10:04:53

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN13, COL. TYPE: ODS-II, ID: CARB-05, INJ:200,COL#1
Number of samples.: 26
Calibration file..: 5EX1015 Last modified on 05-Nov-81 at 09:33:30
Method file.....: EXPLOS Last modified on 05-Nov-81 at 08:54:26
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: CRA/BPSAD4 10X
Sample ID.....: 31436.06
Sample type.....: Sample
Sample amount.....: 2.0000
Number of injections.....: 1
Bottle Number.....: 1

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 20.000
Dilution.....: 10.000
Amount (g or mL).....: 2.000

PEAK INFORMATION

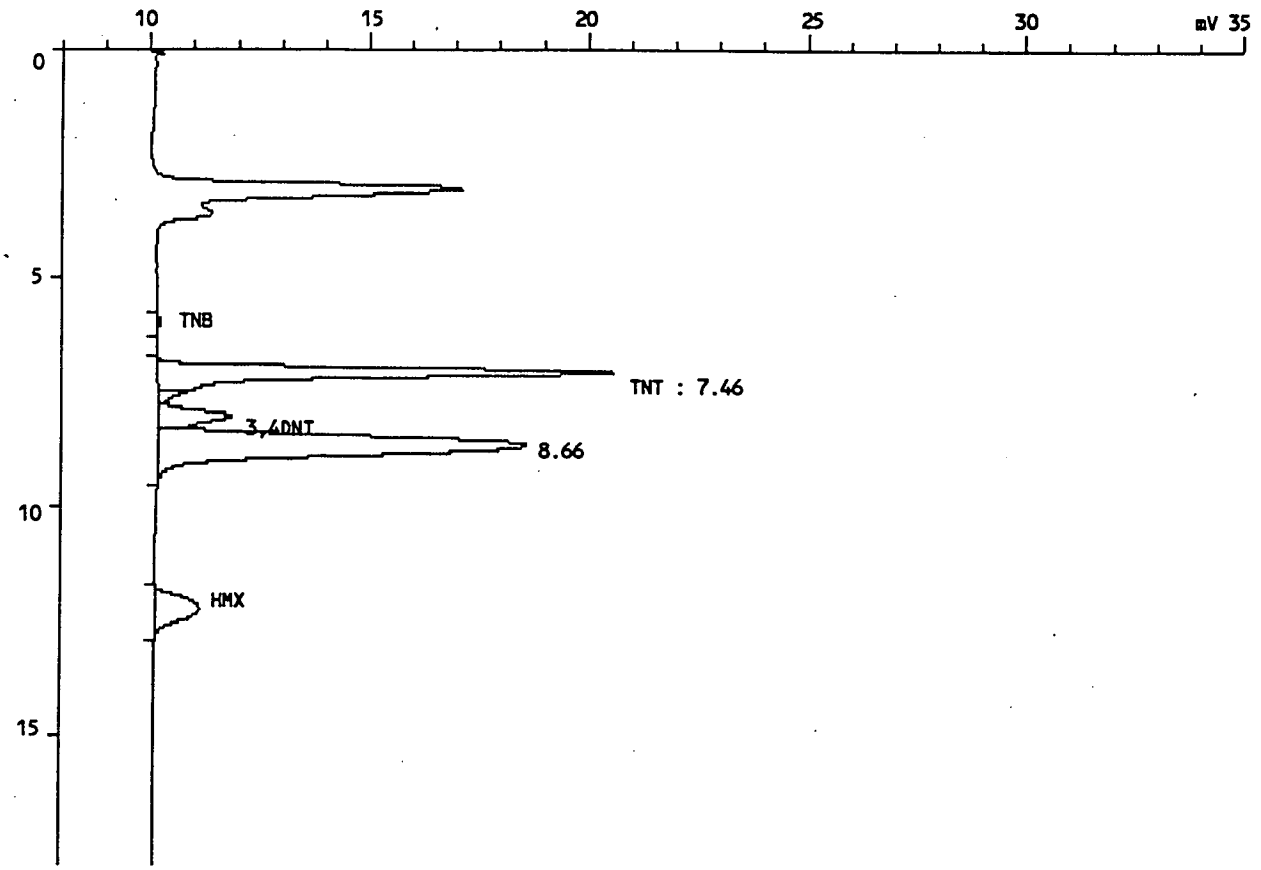
Peak	RT mins	Hght uV	Area uVs	ug/Kg	Peak name
1	4.848	4369	34351	49355.348	HMX*
2	7.371	13522	203406	237069.766	RDX*
3	10.005	38	484	254.251	TNB* PJ
4	15.168	1084	21290	18417.176	3,4DNT 92
5	15.797	5510	119285	60797.648	TNT*
6	16.485	335	7807	5505.865	4ADNT*
7	17.339	41	894	466.908	2ADNT
Total		24899	387517	371867.000	
Residual		0	0	0.000	

LONG PLOT

Injection F: <MC3> 2 2CN1024,19,1

Sample name.....: CRA/BPSAD4 10X
Sample ID.....: 31436.06
INST ID: IN12, COL. TYPE: LC-CN, ID: LC-CN-05, INJ:100, UV

Acquired on 25-Oct-97 at 05:14:58
Reported on 05-Nov-97 at 15:13:28



INJECTION REPORT

Injection F: <MC3> 2 2CN1024,19,1

Acquired on 25-Oct-97 at 05:14:58
Modified on 05-Nov-81 at 15:13:02
Reported on 05-Nov-97 at 15:13:09

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN12, COL. TYPE: LC-CN, ID: LC-CN-05, INJ:100, UV
Number of samples.: 67
Calibration file..: 2CN1024A Last modified on 05-Nov-81 at 11:54:16
Method file.....: LCCN Last modified on 05-Nov-81 at 14:48:18
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: CRA/BPSAD4 10X
Sample ID.....: 31436.06
Sample type.....: Sample
Sample amount.....: 2.0000
Number of injections.....: 1
Bottle Number.....: 1

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 20.000
Dilution.....: 10.000
Amount (g or mL).....: 2.000

PEAK INFORMATION

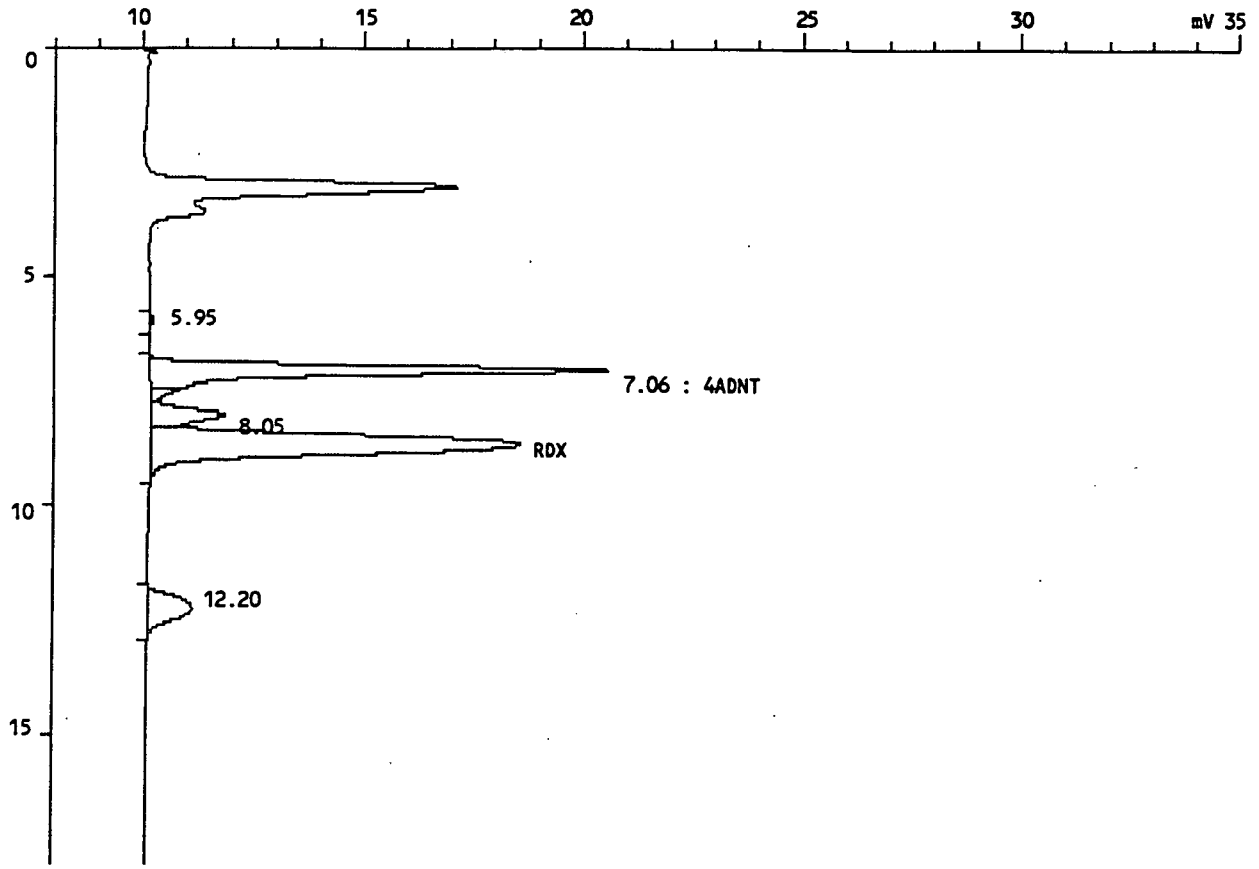
Peak	RT mins	Hght uV	Area uVs	ug/Kg	Peak name
1	5.947	62	889	388.388	TNB
2	7.061	10433	157764	61030.695	TNT
4	8.053	1679	32467	18405.371	3,4DNT
6	12.197	1010	34908	46235.270	HMX
Total		13185	226028	126059.719	
Residual		9280	247515	0.000	

LONG PLOT

Injection F: <MC3> 2 2CN1024,19,1

Sample name.....: CRA/BPSAD4 10X
Sample ID.....: 31436.06
INST ID: IN12, COL. TYPE: LC-CN, ID: LC-CN-05, INJ:100, UV

Acquired on 25-Oct-97 at 05:14:58
Reported on 05-Nov-97 at 15:14:26



INJECTION REPORT

Injection F: <MC3> 2 2CN1024,19,1

Acquired on 25-Oct-97 at 05:14:58
Modified on 05-Nov-81 at 15:13:50
Reported on 05-Nov-97 at 15:13:53

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN12, COL. TYPE: LC-CN, ID: LC-CN-05, INJ:100, UV
Number of samples.: 67
Calibration file..: 2CN1024B Last modified on 05-Nov-81 at 11:56:18
Method file.....: LCCN Last modified on 05-Nov-81 at 14:48:18
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: CRA/BPSAD4 10X
Sample ID.....: 31436.06
Sample type.....: Sample
Sample amount.....: 2.0000
Number of injections.....: 1
Bottle Number.....: 1

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 20.000
Dilution.....: 10.000
Amount (g or mL).....: 2.000

PEAK INFORMATION

Peak	RT mins	Hght uV	Area uVs	ug/Kg	Peak name
3	7.456	839	8362	5384.551	4ADNT
5	8.661	8441	239153	202843.719	RDX
Total		9280	247515	208228.266	
Residual		13185	226028	0.000	

1D

EXPLOSIVE ANALYSIS DATA SHEET

EPA SAMPLE NO:

CRA/BPSAD5 2000
0X 2000X
11/29/97

Lab Name: SWL-TULSA Contract:

Lab Code: SWOK Case No:MKF-OH SDG No: 31436

Matrix: (soil/water) SOIL Lab Sample ID: 31436.07

Sample Amt: 2gm % Moisture 3.02 Date Received: 10/14/97

Extraction Volume: 10ml Date Extracted: 10/14/97

Extraction Method: SONC Date Analyzed: 10/19/97

GPC Cleanup: (Y/N) N Dilution Factor: 20000.00

CAS NO.	COMPOUND	CONCENTRATION UNITS: (ug/L or ug/kg) ug/kg	Q
2691-41-0	HMX	13700000	J
121-82-4	RDX	37300000	
99-35-4	TNB	2500000	U
99-65-0	DNB	2500000	U
479-45-8	TETRYL	6500000	U
98-95-3	NB	2600000	U
118-96-7	TNT	44400000	
1946-51-0	4ADNT	2500000	U
35572-78-2	2ADNT	2500000	U
606-20-2	26DNT	2600000	U
121-14-2	24DNT	2500000	U
88-72-2	2NT	2500000	U
99-99-0	4NT	2500000	U
99-08-1	3NT	2500000	U

SURROGATE AMOUNT SPIKED (ug/kg dry wt.) 20000
3,4-DNT

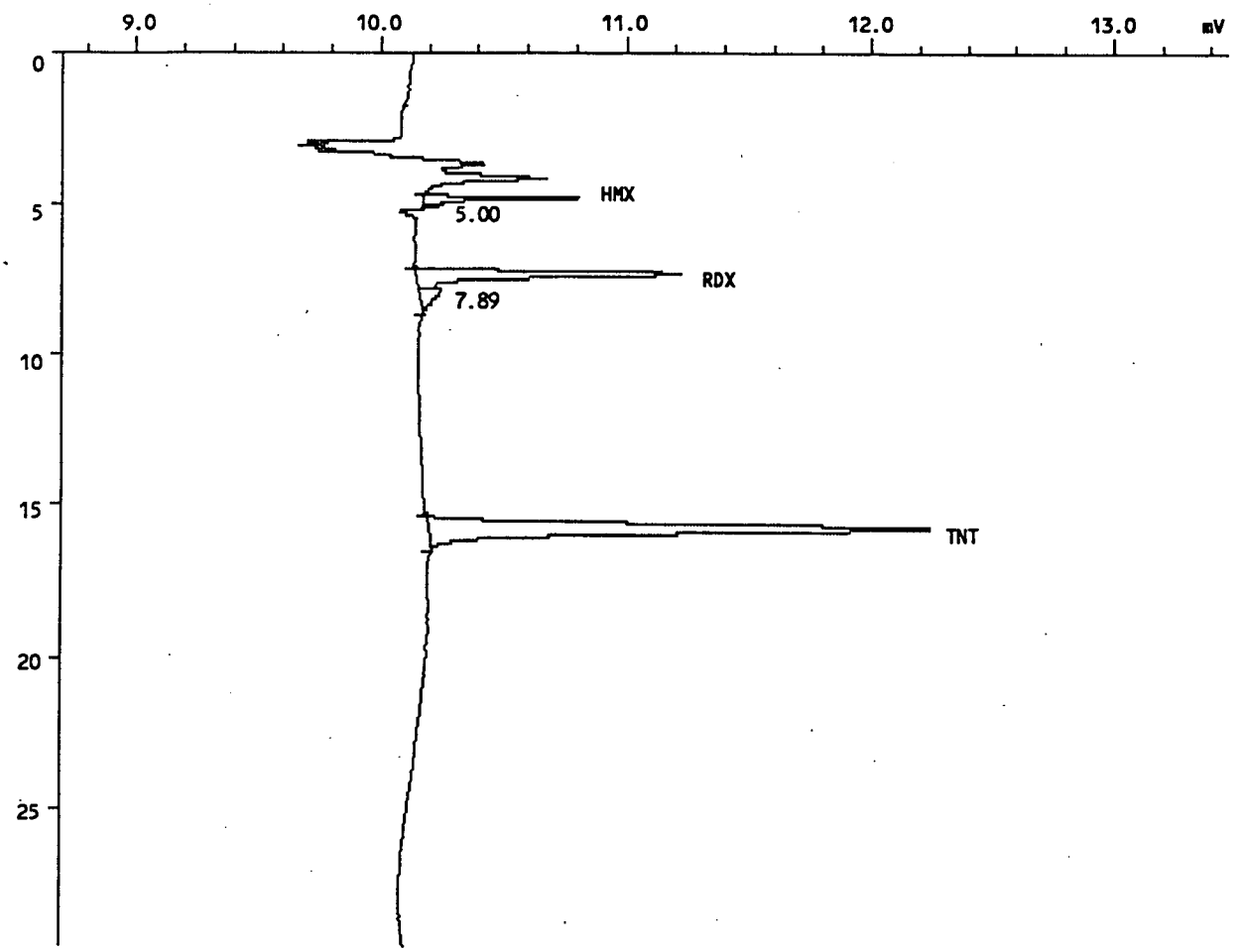
FORM I

LONG PLOT

Injection F: <MC3> 5 5EX1015D,13,1

Sample name.....: CRA/BPSAD5 20000X
Sample ID.....: 31436.07
INST ID: IN13, COL. TYPE: ODS-II, ID: CARB-05, INJ:200,COL#1

Acquired on 19-Oct-97 at 01:28:11
Reported on 05-Nov-97 at 10:06:13



INJECTION REPORT

Injection F: <MC3> 5 5EX1015D,13,1

Acquired on 19-Oct-97 at 01:28:11
Modified on 05-Nov-81 at 10:05:36
Reported on 05-Nov-97 at 10:05:58

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN13, COL. TYPE: ODS-II, ID: CARB-05, INJ:200,COL#1
Number of samples.: 26
Calibration file..: 5EX1015 Last modified on 05-Nov-81 at 09:33:30
Method file.....: EXPLOS Last modified on 05-Nov-81 at 08:54:26
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: CRA/BPSAD5 20000X
Sample ID.....: 31436.07
Sample type.....: Sample
Sample amount.....: 2.0000
Number of injections.....: 1
Bottle Number.....: 1

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 20.000
Dilution.....: 20000.000
Amount (g or mL).....: 2.000

PEAK INFORMATION

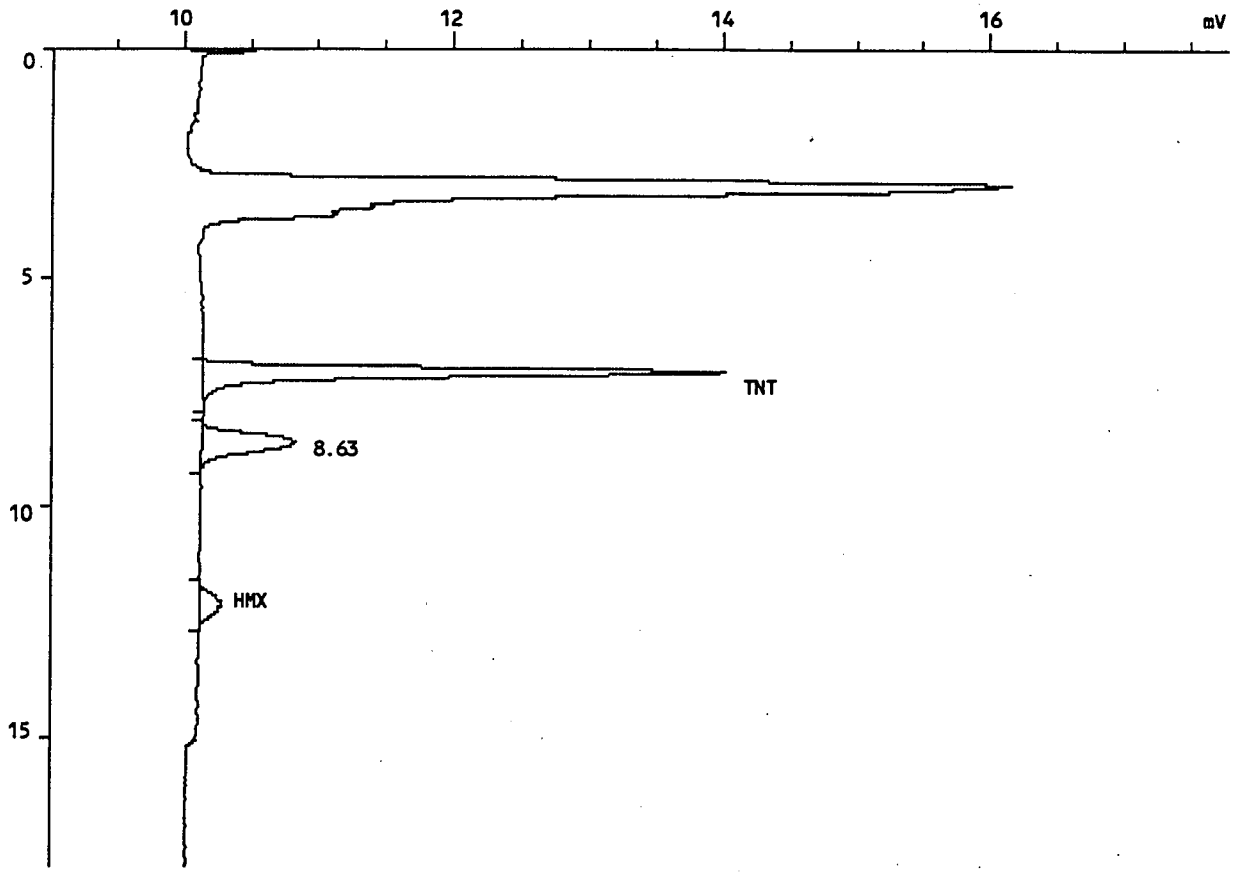
Peak	RT mins	Hght uV	Area uVs	ug/Kg	Peak name
1	4.843	635	4775	13720258.000	HMX* J
3	7.355	1079	15998	37290584.000	RDX*
5	15.781	2042	43540	44382984.000	TNT*
Total		3755	64312	95393824.000	
Residual		163	3222	7848206.500	

LONG PLOT

Injection F: <MC3> 2 2CN1024,20,1

Sample name.....: CRA/BPSAD5 20000X
Sample ID.....: 31436.07
INST ID: IN12, COL. TYPE: LC-CN, ID: LC-CN-05, INJ:100, UV

Acquired on 25-Oct-97 at 05:47:13
Reported on 05-Nov-97 at 12:02:13



INJECTION REPORT

Injection F: <MC3> 2 2CN1024,20,1

Acquired on 25-Oct-97 at 05:47:13
Modified on 05-Nov-81 at 12:00:14
Reported on 05-Nov-97 at 12:00:14

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN12, COL. TYPE: LC-CN, ID: LC-CN-05, INJ:100, UV
Number of samples.: 67
Calibration file..: 2CN1024A Last modified on 05-Nov-81 at 11:54:16
Method file.....: LCCN Last modified on 05-Nov-81 at 11:58:32
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: CRA/BPSAD5 20000X
Sample ID.....: 31436.07
Sample type.....: Sample
Sample amount.....: 2.0000
Number of injections.....: 1
Bottle Number.....: 1

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 20.000
Dilution.....: 20000.000
Amount (g or mL).....: 2.000

PEAK INFORMATION

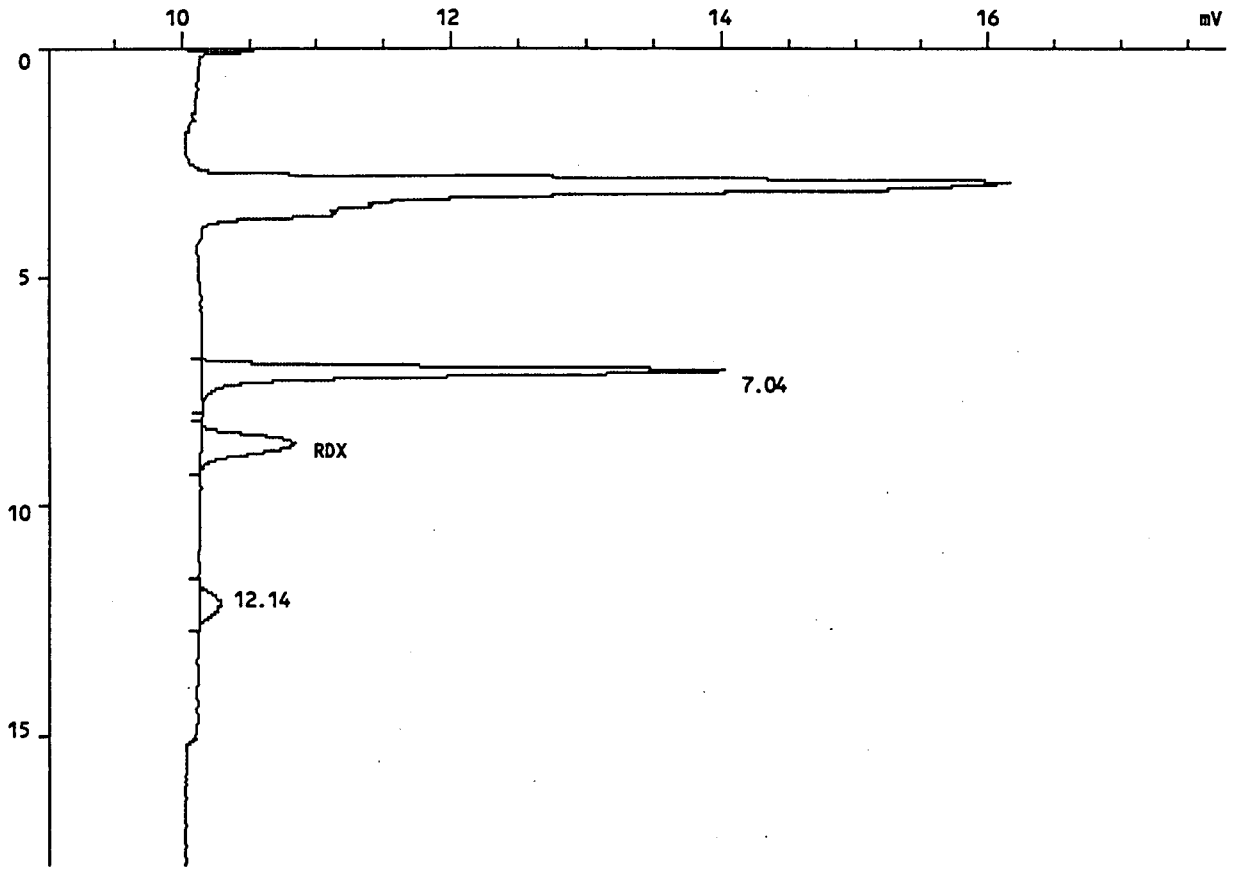
Peak	RT mins	Hght uV	Area uVs	ug/Kg	Peak name
1	7.040	3869	57231	44279396.000	TNT
3	12.139	159	4882	12933124.000	HMX
Total		4028	62113	57212520.000	
Residual		693	18877	0.000	

LONG PLOT

Injection F: <MC3> 2 2CN1024,20,1

Sample name.....: CRA/BPSAD5 20000X
Sample ID.....: 31436.07
INST ID: IN12, COL. TYPE: LC-CN, ID: LC-CN-05, INJ:100, UV

Acquired on 25-Oct-97 at 05:47:13
Reported on 05-Nov-97 at 12:18:08



INJECTION REPORT

Injection F: <MC3> 2 2CN1024,20,1

Acquired on 25-Oct-97 at 05:47:13
Modified on 05-Nov-81 at 12:06:30
Reported on 05-Nov-97 at 12:06:31

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN12, COL. TYPE: LC-CN, ID: LC-CN-05, INJ:100, UV
Number of samples.: 67
Calibration file..: 2CN1024B Last modified on 05-Nov-81 at 11:56:18
Method file.....: LCCN Last modified on 05-Nov-81 at 12:04:50
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: CRA/BPSAD5 20000X
Sample ID.....: 31436.07
Sample type.....: Sample
Sample amount.....: 2.0000
Number of injections.....: 1
Bottle Number.....: 1

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 20.000
Dilution.....: 20000.000
Amount (g or mL).....: 2.000

PEAK INFORMATION

Peak	RT mins	Hght uV	Area uVs	ug/Kg	Peak name
2	8.629	693	18877	32021586.000	RDX
Total		693	18877	32021586.000	
Residual		4028	62113	0.000	

1D
EXPLOSIVE ANALYSIS DATA SHEET

EPA SAMPLE NO:

CRA/BPSAD6 10X

'b Name: SWL-TULSA Contract:

Lab Code: SWOK Case No:MKF-OH SDG No: 31436

Matrix: (soil/water) SOIL Lab Sample ID: 31436.08

Sample Amt: 2gm % Moisture 12.83 Date Received: 10/14/97

Extraction Volume: 10ml Date Extracted: 10/14/97

Extraction Method: SONC Date Analyzed: 10/19/97

GPC Cleanup: (Y/N) N Dilution Factor: 10.00

CAS NO.	COMPOUND	CONCENTRATION UNITS: (ug/L or ug/kg) ug/kg	Q
2691-41-0	HMX	8220	J
121-82-4	RDX	77100	
99-35-4	TNB	1250	U
99-65-0	DNB	1250	U
479-45-8	TETRYL	3250	U
98-95-3	NB	1300	U
118-96-7	TNT	1430	
1946-51-0	4ADNT	1480	
35572-78-2	2ADNT	1250	U
606-20-2	26DNT	1300	U
121-14-2	24DNT	1250	U
88-72-2	2NT	1250	U
99-99-0	4NT	1250	U
99-08-1	3NT	1250	U

SURROGATE AMOUNT SPIKED (ug/kg dry wt.) 20000
3,4-DNT

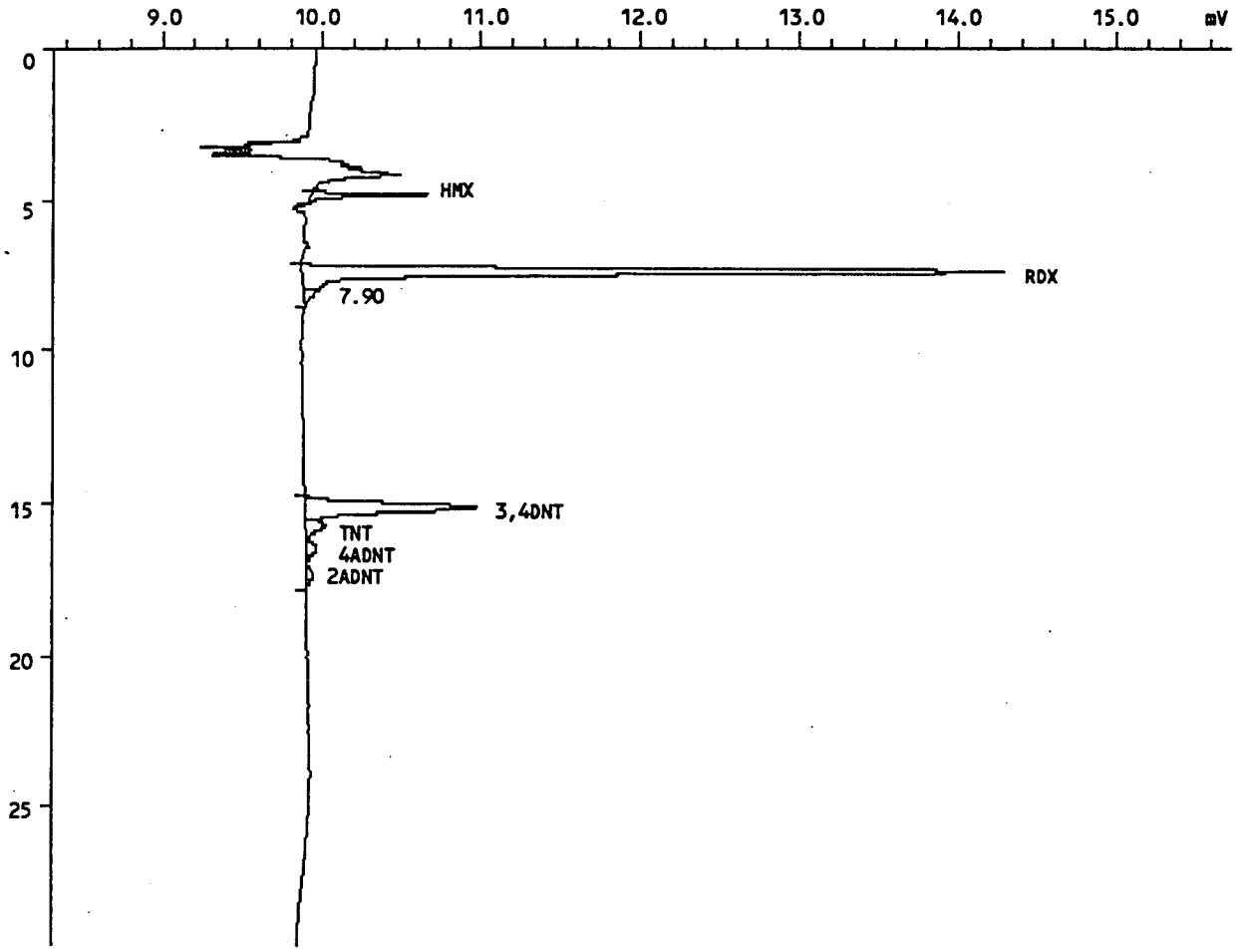
FORM I

LONG PLOT

Injection F: <MC3> 5 5EX1015D,14,1

Sample name.....: CRA/BPSAD6 10X
Sample ID.....: 31436.08
INST ID: IN13, COL. TYPE: ODS-II, ID: CARB-05, INJ:200,COL#1

Acquired on 19-Oct-97 at 02:12:54
Reported on 05-Nov-97 at 15:26:33



INJECTION REPORT

Injection F: <MC3> 5 5EX1015D,14,1

Acquired on 19-Oct-97 at 02:12:54
Modified on 05-Nov-81 at 15:24:00
Reported on 05-Nov-97 at 15:25:43

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN13, COL. TYPE: ODS-II, ID: CARB-05, INJ:200,COL#1
Number of samples.: 26
Calibration file..: 5EX1015 Last modified on 05-Nov-81 at 15:23:56
Method file.....: EXPLOS Last modified on 05-Nov-81 at 08:54:26
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: CRA/BPSAD6 10X
Sample ID.....: 31436.08
Sample type.....: Sample
Sample amount.....: 2.0000
Number of injections.....: 1
Bottle Number.....: 1

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 20.000
Dilution.....: 10.000
Amount (g or mL).....: 2.000

PEAK INFORMATION

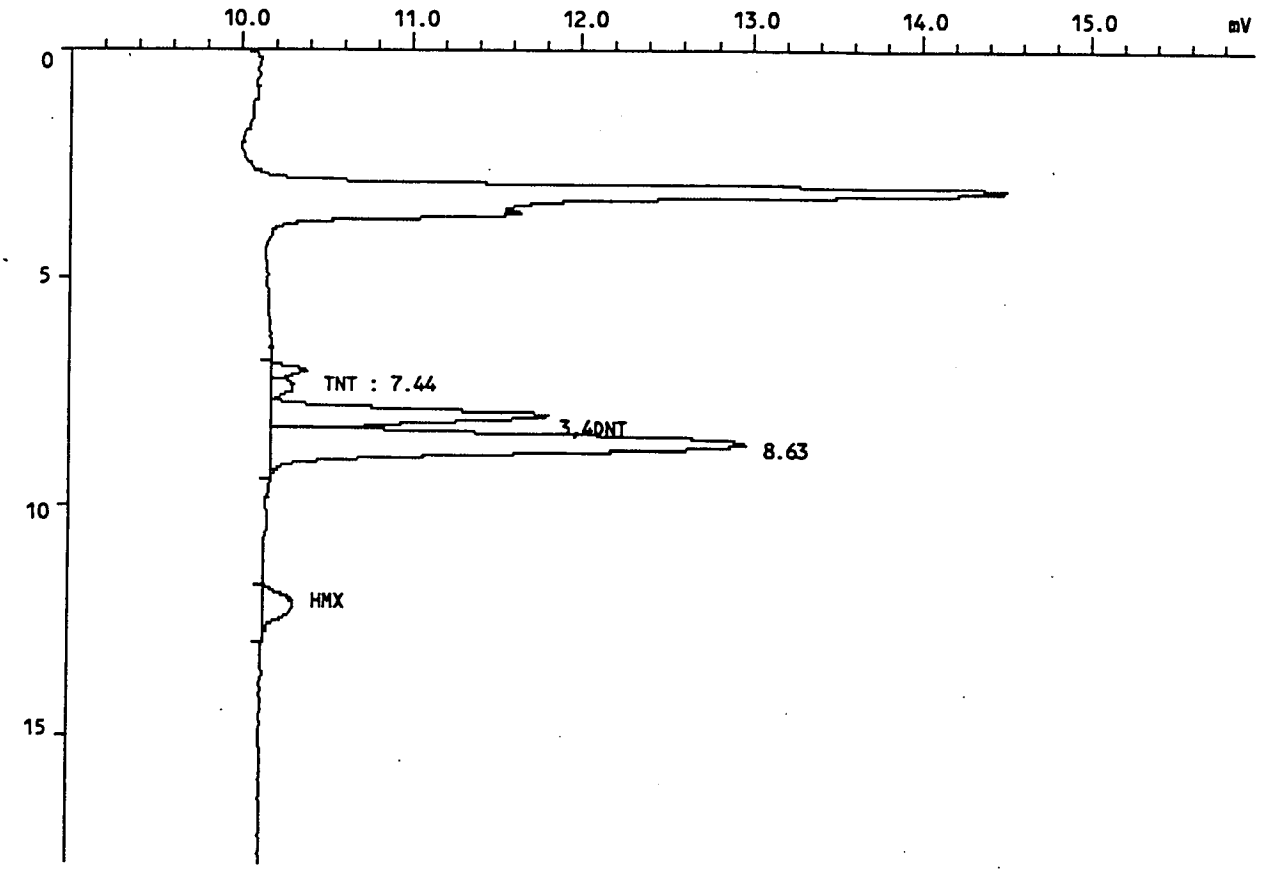
Peak	RT mins	Hght uV	Area uVs	ug/Kg	Peak name
1	4.843	739	5722	8220.932	HMX *
2	7.360	4425	66155	77103.688	RDX *
4	15.157	1083	22172	19179.885	3,4DNT *
5	15.755	125	2806	1430.231	TNT *
6	16.475	76	2094	1476.663	4ADNT *
7	17.381	47	1304	681.408	2ADNT
Total		6495	100253	108092.805	
Residual		115	2169	2528.429	

LONG PLOT

Injection F: <MC3> 2 2CN1024,21,1

Sample name.....: CRA/BPSAD6 10X
Sample ID.....: 31436.08
INST ID: IN12, COL. TYPE: LC-CN, ID: LC-CN-05, INJ:100, UV

Acquired on 25-Oct-97 at 06:19:29
Reported on 05-Nov-97 at 12:01:54



INJECTION REPORT

Injection F: <MC3> 2 2CN1024,21,1

Acquired on 25-Oct-97 at 06:19:29
Modified on 05-Nov-81 at 12:00:26
Reported on 05-Nov-97 at 12:00:27

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN12, COL. TYPE: LC-CN, ID: LC-CN-05, INJ:100, UV
Number of samples.: 67
Calibration file..: 2CN1024A Last modified on 05-Nov-81 at 11:54:16
Method file.....: LCCN Last modified on 05-Nov-81 at 11:58:32
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: CRA/BPSAD6 10X
Sample ID.....: 31436.08
Sample type.....: Sample
Sample amount.....: 2.0000
Number of injections.....: 1
Bottle Number.....: 1

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 20.000
Dilution.....: 10.000
Amount (g or mL).....: 2.000

PEAK INFORMATION

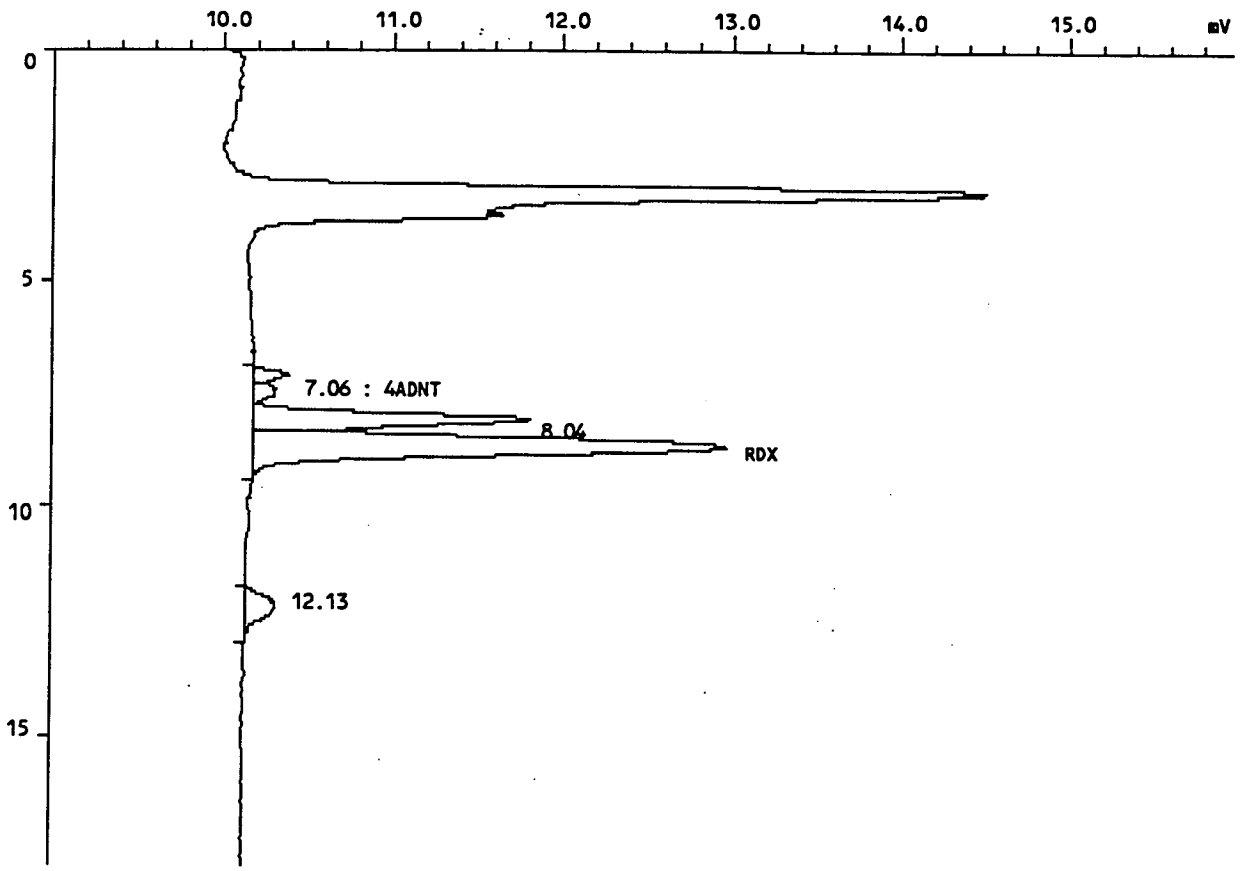
Peak	RT mins	Hght uV	Area uVs	ug/Kg	Peak name
1	7.056	209	2931	1133.934	TNT
3	8.037	1638	31585	17905.525	3,4DNT
5	12.133	171	5747	7611.323	HMX
Total		2017	40263	26650.781	
Residual		2927	81618	0.000	

LONG PLOT

Injection F: <MC3> 2 2CN1024,21,1

Sample name.....: CRA/BPSAD6 10X
Sample ID.....: 31436.08
INST ID: IN12, COL. TYPE: LC-CN, ID: LC-CN-05, INJ:100, UV

Acquired on 25-Oct-97 at 06:19:29
Reported on 05-Nov-97 at 12:15:31



INJECTION REPORT

Injection F: <MC3> 2 2CN1024,21,1

Acquired on 25-Oct-97 at 06:19:29
Modified on 05-Nov-81 at 12:06:44
Reported on 05-Nov-97 at 12:06:43

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN12, COL. TYPE: LC-CN, ID: LC-CN-05, INJ:100, UV
Number of samples.: 67
Calibration file..: 2CN1024B Last modified on 05-Nov-81 at 11:56:18
Method file.....: LCCN Last modified on 05-Nov-81 at 12:04:50
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: CRA/BPSAD6 10X
Sample ID.....: 31436.08
Sample type.....: Sample
Sample amount.....: 2.0000
Number of injections.....: 1
Bottle Number.....: 1

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 20.000
Dilution.....: 10.000
Amount (g or mL).....: 2.000

PEAK INFORMATION

Peak	RT mins	Hght uV	Area uVs	ug/Kg	Peak name
2	7.440	136	2599	1673.816	4ADNT
4	8.629	2792	79018	67021.586	RDX
Total		2927	81618	68695.398	
Residual		2017	40263	0.000	

1D

EXPLOSIVE ANALYSIS DATA SHEET

EPA SAMPLE NO:

CRA/BPSAD7 100X

b Name: SWL-TULSA Contract:

Lab Code: SWOK Case No:MKF-OH SDG No: 31436

Matrix: (soil/water) SOIL Lab Sample ID: 31436.09

Sample Amt: 2gm % Moisture 14.57 Date Received: 10/14/97

Extraction Volume: 10ml Date Extracted: 10/14/97

Extraction Method: SONC Date Analyzed: 10/18/97

GPC Cleanup: (Y/N) N Dilution Factor: 100.00

CAS NO.	COMPOUND	CONCENTRATION UNITS: (ug/L or ug/kg) ug/kg	Q
2691-41-0	HMX	45200	J
121-82-4	RDX	377000	
99-35-4	TNB	12500	U
99-65-0	DNB	12500	U
479-45-8	TETRYL	32500	U
98-95-3	NB	13000	U
118-96-7	TNT	12000	J
1946-51-0	4ADNT	12500	U
35572-78-2	2ADNT	12500	U
606-20-2	26DNT	13000	U
121-14-2	24DNT	12500	U
88-72-2	2NT	12500	U
99-99-0	4NT	12500	U
99-08-1	3NT	12500	U

SURROGATE AMOUNT SPIKED (ug/kg dry wt.) 20000
3,4-DNT

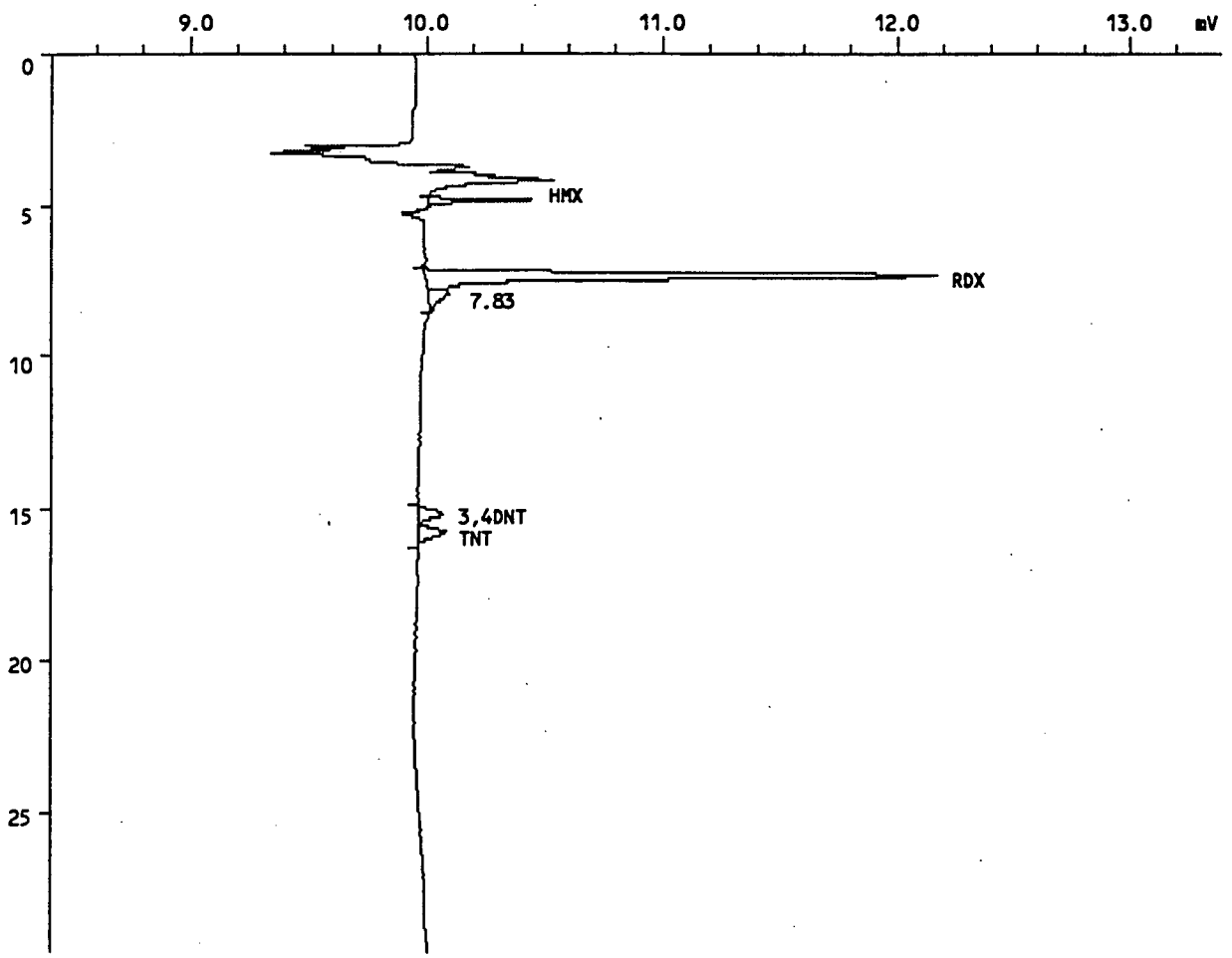
FORM I

LONG PLOT

Injection F: <MC3> 5 5EX1015C,15,1

Sample name.....: CRA/BPSAD7 100X
Sample ID.....: 31436.09
INST ID: IN13, COL. TYPE: ODS-II, ID: CARB-05, INJ:200,COL#1

Acquired on 18-Oct-97 at 07:21:36
Reported on 05-Nov-97 at 11:27:21



INJECTION REPORT

Injection F: <MC3> 5 5EX1015C,15,1

Acquired on 18-Oct-97 at 07:21:36
Modified on 05-Nov-81 at 11:22:34
Reported on 05-Nov-97 at 11:22:41

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN13, COL. TYPE: ODS-II, ID: CARB-05, INJ:200,COL#1
Number of samples.: 27
Calibration file..: 5EX1015 Last modified on 05-Nov-81 at 11:22:30
Method file.....: EXPLOS Last modified on 05-Nov-81 at 08:54:26
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: CRA/BPSAD7 100X
Sample ID.....: 31436.09
Sample type.....: Sample
Sample amount.....: 2.0000
Number of injections.....: 1
Bottle Number.....: 1

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 20.000
Dilution.....: 100.000
Amount (g or mL).....: 2.000

PEAK INFORMATION

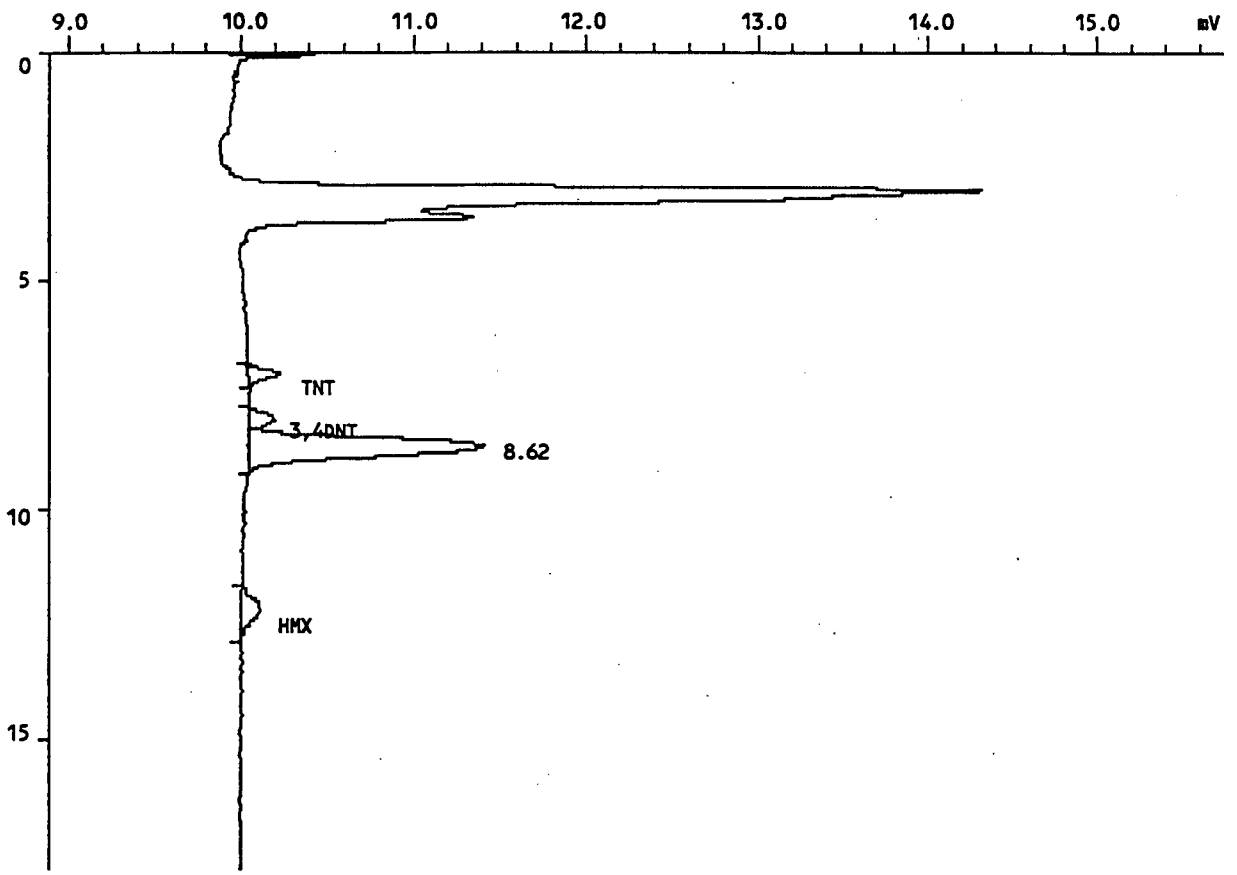
Peak	RT mins	Hght uV	Area uVs	ug/Kg	Peak name
1	4.837	430	3150	45258.512	HMX *
2	7.365	2180	32384	377436.625	RDX *
4	15.152	104	2037	17623.447	3,4DNT
5	15.792	113	2352	11986.732	TNT *
Total		2826	39923	452305.281	
Residual		91	2052	23917.504	

LONG PLOT

Injection F: <MC3> 2 2CN1024,22,1

Sample name.....: CRA/BPSAD7 100X
Sample ID.....: 31436.09
INST ID: IN12, COL. TYPE: LC-CN, ID: LC-CN-05, INJ:100, UV

Acquired on 25-Oct-97 at 06:51:44
Reported on 05-Nov-97 at 12:01:37



INJECTION REPORT

Injection F: <MC3> 2 2CN1024,22,1

Acquired on 25-Oct-97 at 06:51:44
Modified on 05-Nov-81 at 12:00:40
Reported on 05-Nov-97 at 12:00:41

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN12, COL. TYPE: LC-CN, ID: LC-CN-05, INJ:100, UV
Number of samples.: 67
Calibration file..: 2CN1024A Last modified on 05-Nov-81 at 11:54:16
Method file.....: LCCN Last modified on 05-Nov-81 at 11:58:32
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: CRA/BPSAD7 100X
Sample ID.....: 31436.09
Sample type.....: Sample
Sample amount.....: 2.0000
Number of injections.....: 1
Bottle Number.....: 1

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 20.000
Dilution.....: 100.000
Amount (g or mL).....: 2.000

PEAK INFORMATION

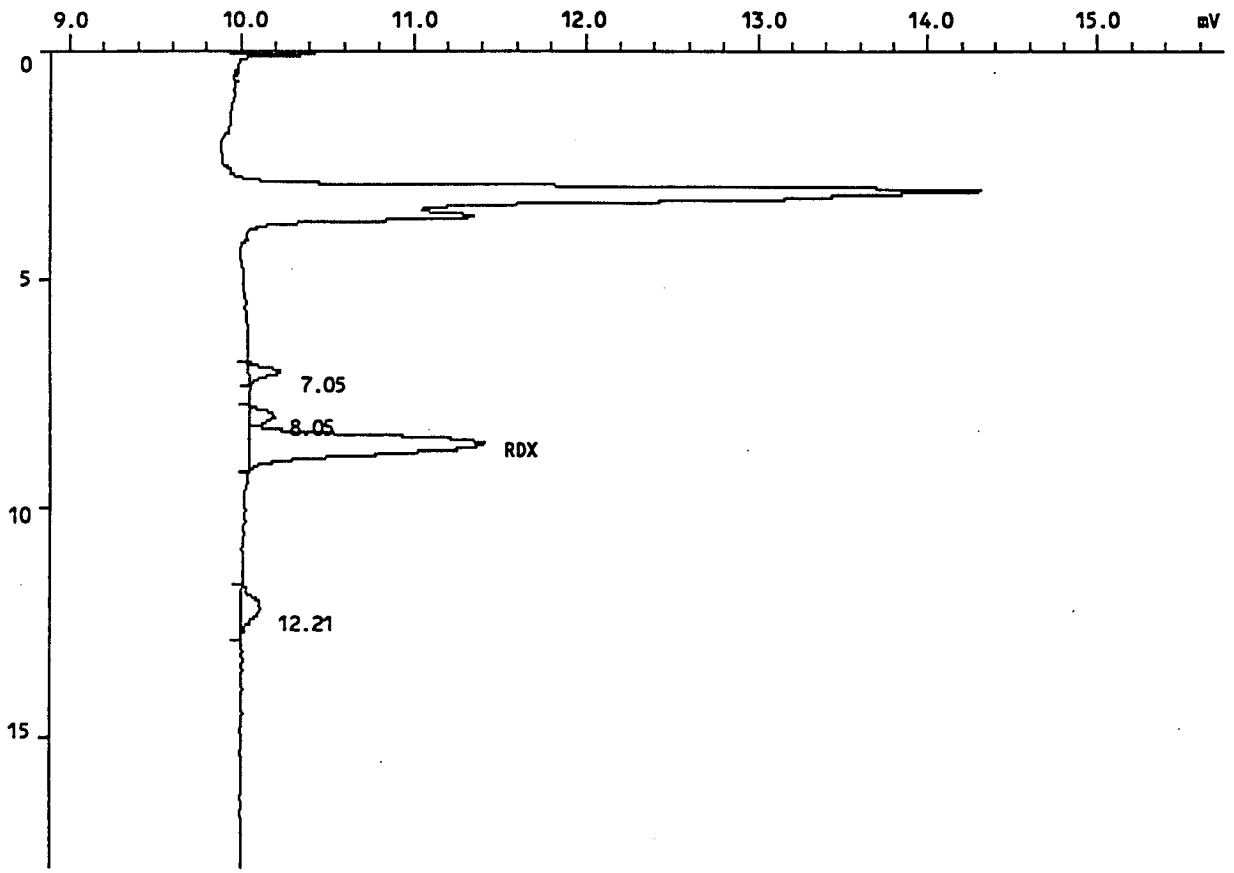
Peak	RT mins	Hght uV	Area uVs	ug/Kg	Peak name
1	7.051	186	2689	10401.404	TNT
2	8.053	151	2692	15259.720	3,4DNT
4	12.208	113	3647	48307.145	HMX
Total		449	9028	73968.266	
Residual		1371	37590	0.000	

LONG PLOT

Injection F: <MC3> 2 2CN1024,22,1

Sample name.....: CRA/BPSAD7 100X
Sample ID.....: 31436.09
INST ID: IN12, COL. TYPE: LC-CN, ID: LC-CN-05, INJ:100, UV

Acquired on 25-Oct-97 at 06:51:44
Reported on 05-Nov-97 at 12:15:05



INJECTION REPORT

Injection F: <MC3> 2 2CN1024,22,1

Acquired on 25-Oct-97 at 06:51:44
Modified on 05-Nov-81 at 12:06:56
Reported on 05-Nov-97 at 12:06:57

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN12, COL. TYPE: LC-CN, ID: LC-CN-05, INJ:100, UV
Number of samples.: 67
Calibration file..: 2CN1024B Last modified on 05-Nov-81 at 11:56:18
Method file.....: LCCN Last modified on 05-Nov-81 at 12:04:50
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: CRA/BPSAD7 100X
Sample ID.....: 31436.09
Sample type.....: Sample
Sample amount.....: 2.0000
Number of injections.....: 1
Bottle Number.....: 1

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 20.000
Dilution.....: 100.000
Amount (g or mL).....: 2.000

PEAK INFORMATION

Peak	RT mins	Hght uV	Area uVs	ug/Kg	Peak name
3	8.624	1371	37590	318833.625	RDX
Total		1371	37590	318833.625	
Residual		449	9028	0.000	

1D

EXPLOSIVE ANALYSIS DATA SHEET

EPA SAMPLE NO:

CRA/BPSAD8 10X

Lab Name: SWL-TULSA Contract:

Lab Code: SWOK Case No:MKF-OH SDG No: 31436

Matrix: (soil/water) SOIL Lab Sample ID: 31436.10

Sample Amt: 2gm % Moisture 12.93 Date Received: 10/14/97

Extraction Volume: 10ml Date Extracted: 10/14/97

Extraction Method: SONC Date Analyzed: 10/19/97

GPC Cleanup: (Y/N) N Dilution Factor: 10.00

CAS NO.	COMPOUND	CONCENTRATION UNITS: (ug/L or ug/kg) ug/kg	Q
2691-41-0	HMX	20700	
121-82-4	RDX	58500	
99-35-4	TNB	1250	U
99-65-0	DNB	1250	U
479-45-8	TETRYL	3250	U
98-95-3	NB	1300	U
118-96-7	TNT	312	J
1946-51-0	4ADNT	1250	U
35572-78-2	2ADNT	1250	U
606-20-2	26DNT	1300	U
121-14-2	24DNT	1250	U
88-72-2	2NT	1250	U
99-99-0	4NT	1250	U
99-08-1	3NT	1250	U

SURROGATE AMOUNT SPIKED (ug/kg dry wt.) 20000
3,4-DNT

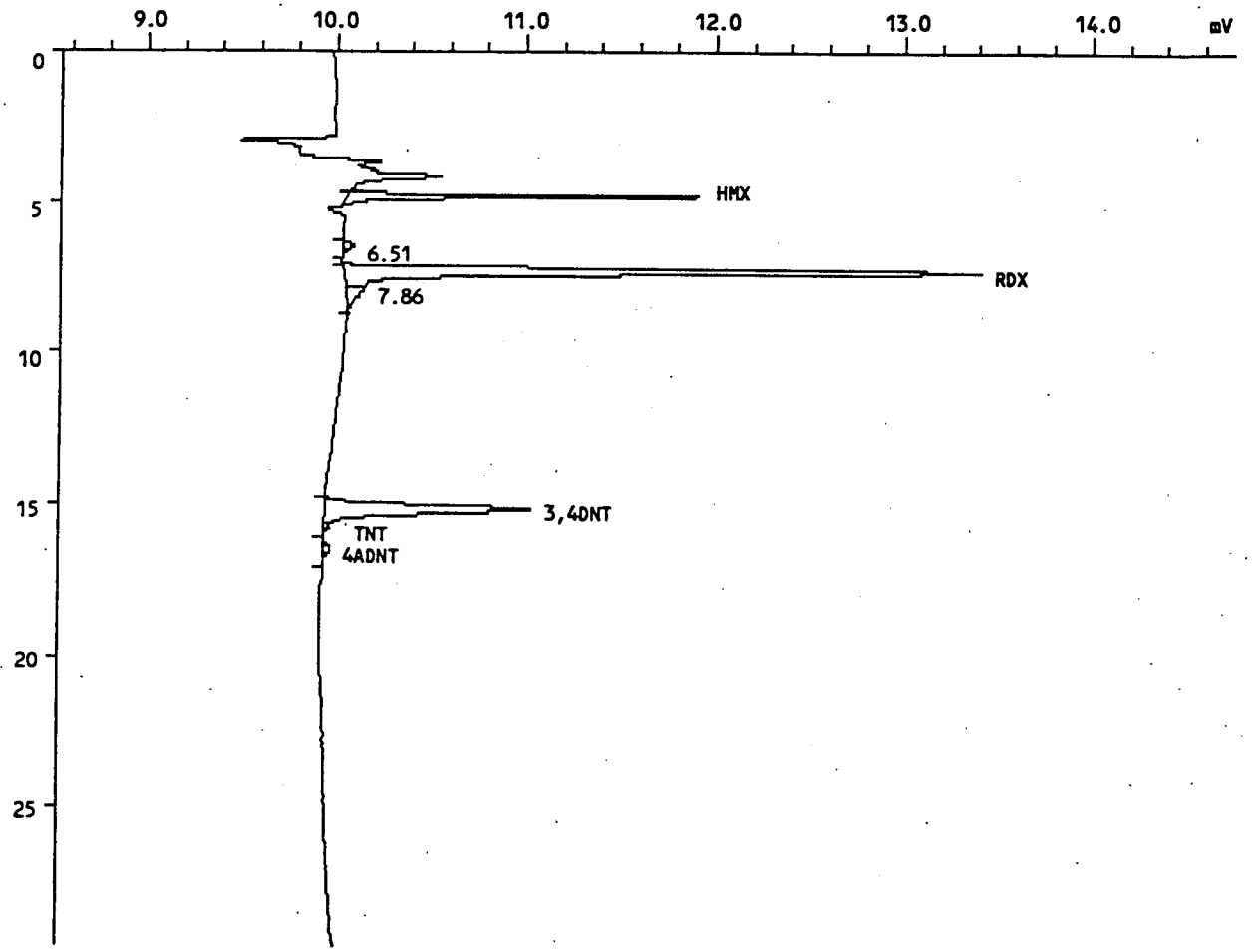
FORM I

LONG PLOT

Injection F: <MC3> 5 5EX1015D,16,1

Sample name.....: CRA/BPSAD8 10X
Sample ID.....: 31436.10
INST ID: IN13, COL. TYPE: ODS-II, ID: CARB-05, INJ:200,COL#1

Acquired on 19-Oct-97 at 03:42:22
Reported on 05-Nov-97 at 15:30:20



INJECTION REPORT

Injection F: <MC3> 5 5EX1015D,16,1

Acquired on 19-Oct-97 at 03:42:22
Modified on 05-Nov-81 at 15:29:44
Reported on 05-Nov-97 at 15:29:52

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN13, COL. TYPE: ODS-II, ID: CARB-05, INJ:200,COL#1
Number of samples.: 26
Calibration file..: 5EX1015 Last modified on 05-Nov-81 at 15:29:40
Method file.....: EXPLOS Last modified on 05-Nov-81 at 08:54:26
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: CRA/BPSAD8 10X
Sample ID.....: 31436.10
Sample type.....: Sample
Sample amount.....: 2.0000
Number of injections.....: 1
Bottle Number.....: 1

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 20.000
Dilution.....: 10.000
Amount (g or mL).....: 2.000

PEAK INFORMATION

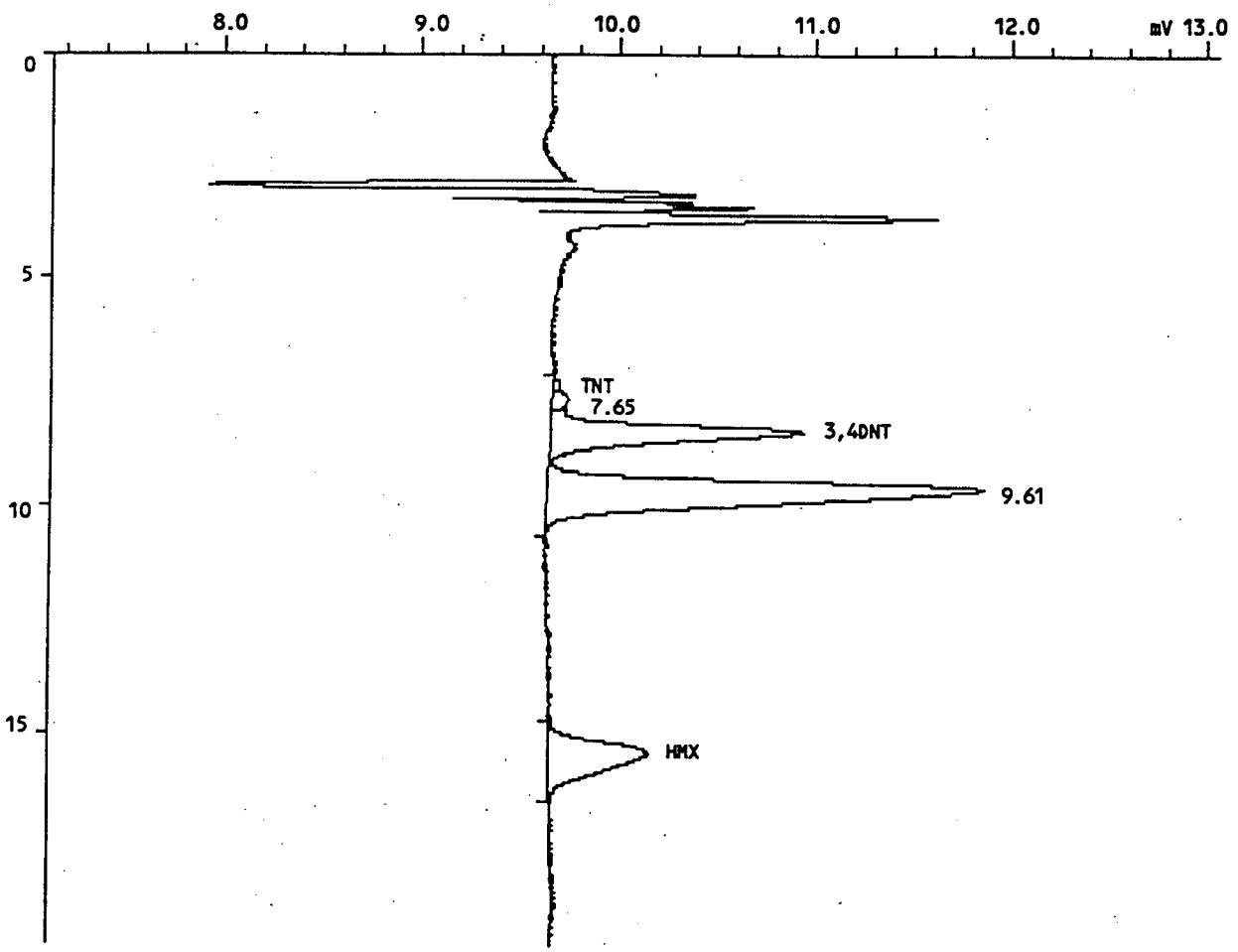
Peak	RT mins	Hght uV	Area uVs	ug/Kg	Peak name
1	4.843	1849	14421	20719.484	HMX*
3	7.355	3375	50214	58524.875	RDX*
5	15.163	1093	22350	19333.570	3,4DNT 98
6	15.589	49	612	311.865	TNT*
7	16.517	44	961	678.039	4ADNT
Total		6411	88558	99567.836	
Residual		175	3468	4278.950	

LONG PLOT

Injection F: <MC3> 3 3CN1027,14,1

Sample name.....: CRA/BPSAD8 10X
Sample ID.....: 31436.10
INST ID: IN10, COL. TYPE: LC-CN, ID: LC-CN-07, INJ:100, UV

Acquired on 28-Oct-97 at 05:44:14
Reported on 05-Nov-97 at 11:43:11



INJECTION REPORT

Injection F: <MC3> 3 3CN1027,14,1

Acquired on 28-Oct-97 at 05:44:14
Modified on 05-Nov-81 at 10:53:00
Reported on 05-Nov-97 at 10:53:01

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN10, COL. TYPE: LC-CN, ID: LC-CN-07, INJ:100, UV
Number of samples.: 56
Calibration file..: 3CN1027A Last modified on 05-Nov-81 at 10:52:08
Method file.....: LCCN Last modified on 05-Nov-81 at 10:52:26
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: CRA/BPSAD8 10X
Sample ID.....: 31436.10
Sample type.....: Sample
Sample amount.....: 2.0000
Number of injections.....: 1
Bottle Number.....: 1

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 20.000
Dilution.....: 10.000
Amount (g or mL).....: 2.000

PEAK INFORMATION

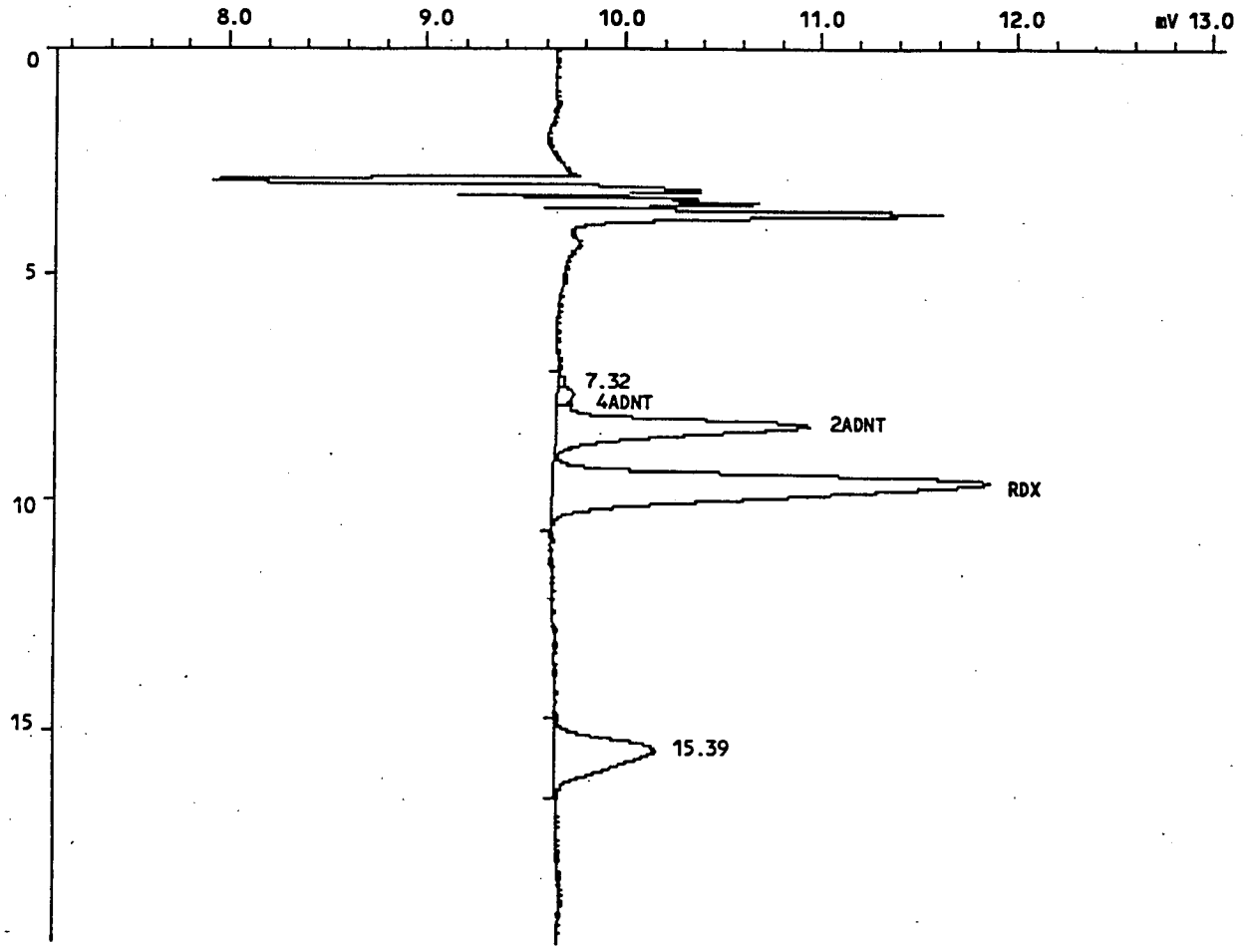
Peak	RT mins	Hght uV	Area uVs	ug/Kg	Peak name
1	7.317	41	501	193.108	TNT
3	8.357	1287	32076	19241.801	3,4DNT
5	15.387	508	21926	19824.992	HMX
Total		1836	54503	39259.898	
Residual		2319	73806	0.000	

LONG PLOT

Injection F: <MC3> 3 3CN1027,14,1

Sample name.....: CRA/BPSAD8 10X
Sample ID.....: 31436.10
INST ID: IN10, COL. TYPE: LC-CN, ID: LC-CN-07, INJ:100, UV

Acquired on 28-Oct-97 at 05:44:14
Reported on 05-Nov-97 at 11:51:20



INJECTION REPORT

Injection F: <MC3> 3 3CN1027,14,1

Acquired on 28-Oct-97 at 05:44:14
Modified on 05-Nov-81 at 11:44:38
Reported on 05-Nov-97 at 11:44:38

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN10, COL. TYPE: LC-CN, ID: LC-CN-07, INJ:100, UV
Number of samples.: 56
Calibration file..: 3CN1027B Last modified on 05-Nov-81 at 10:42:36
Method file.....: LCCN Last modified on 05-Nov-81 at 11:44:20
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: CRA/BPSAD8 10X
Sample ID.....: 31436.10
Sample type.....: Sample
Sample amount.....: 2.0000
Number of injections.....: 1
Bottle Number.....: 1

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 20.000
Dilution.....: 10.000
Amount (g or mL).....: 2.000

PEAK INFORMATION

Peak	RT mins	Hght uV	Area uVs	ug/Kg	Peak name
2	7.648	91	1862	983.727	4ADNT
3	8.357	1287	32076	12365.490	2ADNT
4	9.605	2228	71944	49788.207	RDX
Total		3606	105882	63137.422	
Residual		549	22427	0.000	

1D
EXPLOSIVE ANALYSIS DATA SHEET

EPA SAMPLE NO:

CRA/BPSAD9 10X

b Name: SWL-TULSA Contract:

Lab Code: SWOK Case No:MKF-OH SDG No: 31436

Matrix: (soil/water) SOIL Lab Sample ID: 31436.11

Sample Amt: 2gm % Moisture 14.90 Date Received: 10/14/97

Extraction Volume: 10ml Date Extracted: 10/14/97

Extraction Method: SONC Date Analyzed: 10/24/97

GPC Cleanup: (Y/N) N Dilution Factor: 10.00

CAS NO.	COMPOUND	CONCENTRATION UNITS: (ug/L or ug/kg) ug/kg	Q
2691-41-0	HMX	14500	
121-82-4	RDX	60900	
99-35-4	TNB	1250	U
99-65-0	DNB	1250	U
479-45-8	TETRYL	3250	U
98-95-3	NB	1300	U
118-96-7	TNT	823	PJ
1946-51-0	4ADNT	1210	PJ
35572-78-2	2ADNT	1250	U
606-20-2	26DNT	1300	U
121-14-2	24DNT	1250	U
88-72-2	2NT	1250	U
99-99-0	4NT	1250	U
99-08-1	3NT	1250	U

SURROGATE AMOUNT SPIKED (ug/kg dry wt.) 20000
3,4-DNT

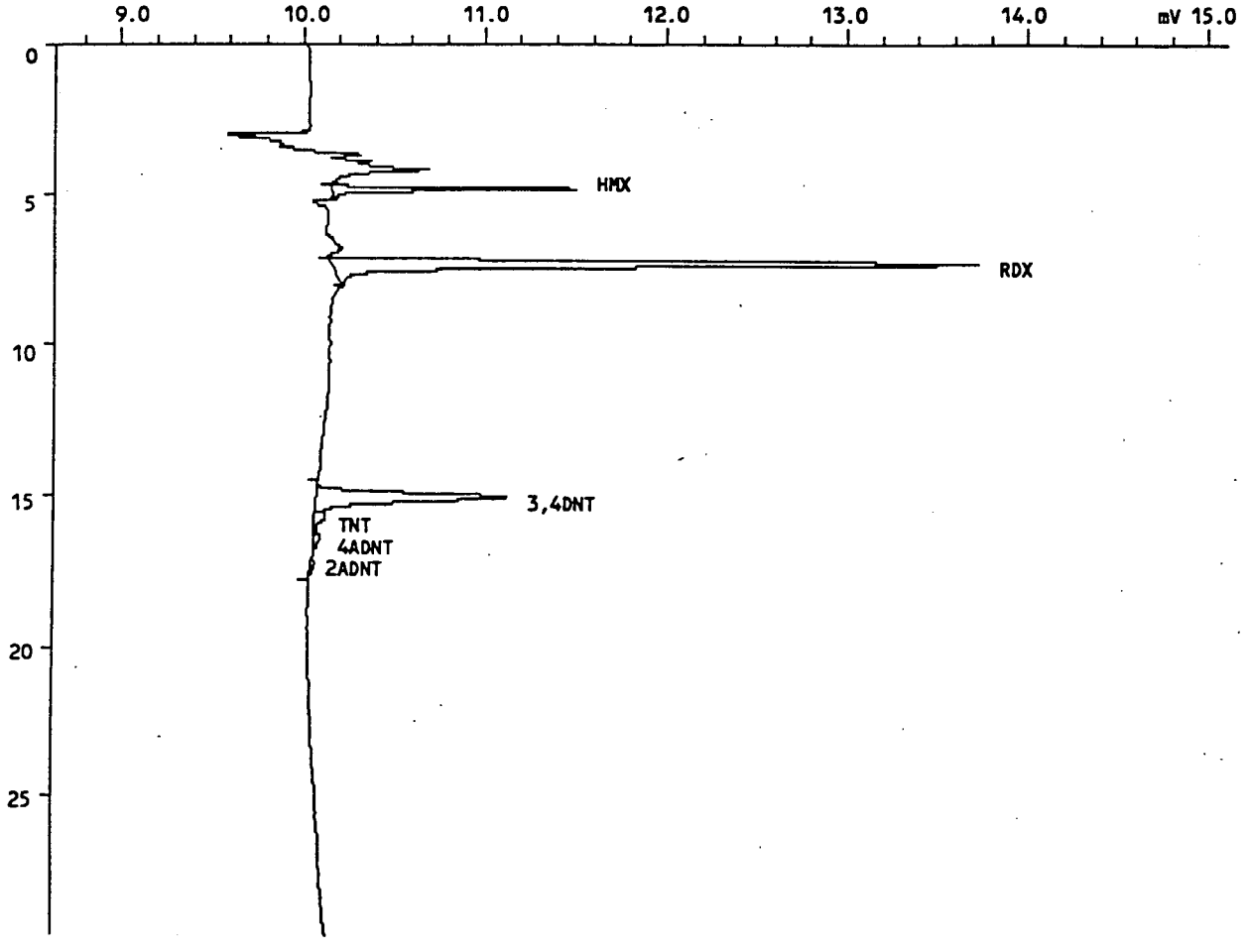
FORM I

LONG PLOT

Injection F: <MC3> 5 5EX1021E,4,1

Sample name.....: CRA/BPSAD9 10X
Sample ID.....: 31436.11
INST ID: IN13, COL. TYPE: ODS-II, ID: CARB-05, INJ:200,COL#1

Acquired on 24-Oct-97 at 20:21:49
Reported on 05-Nov-97 at 08:48:40



INJECTION REPORT

Injection F: <MC3> 5 5EX1021E,4,1

Acquired on 24-Oct-97 at 20:21:49
Modified on 05-Nov-81 at 08:44:28
Reported on 05-Nov-97 at 08:44:28

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN13, COL. TYPE: ODS-II, ID: CARB-05, INJ:200,COL#1
Number of samples.: 34
Calibration file..: 5EX1021 Last modified on 30-Oct-81 at 18:08:22
Method file.....: EXPLOS Last modified on 05-Nov-81 at 08:43:52
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: CRA/BPSAD9 10X
Sample ID.....: 31436.11
Sample type.....: Sample
Sample amount.....: 2.0000
Number of injections.....: 1
Bottle Number.....: 1

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 20.000
Dilution.....: 10.000
Amount (g or mL).....: 2.000

PEAK INFORMATION

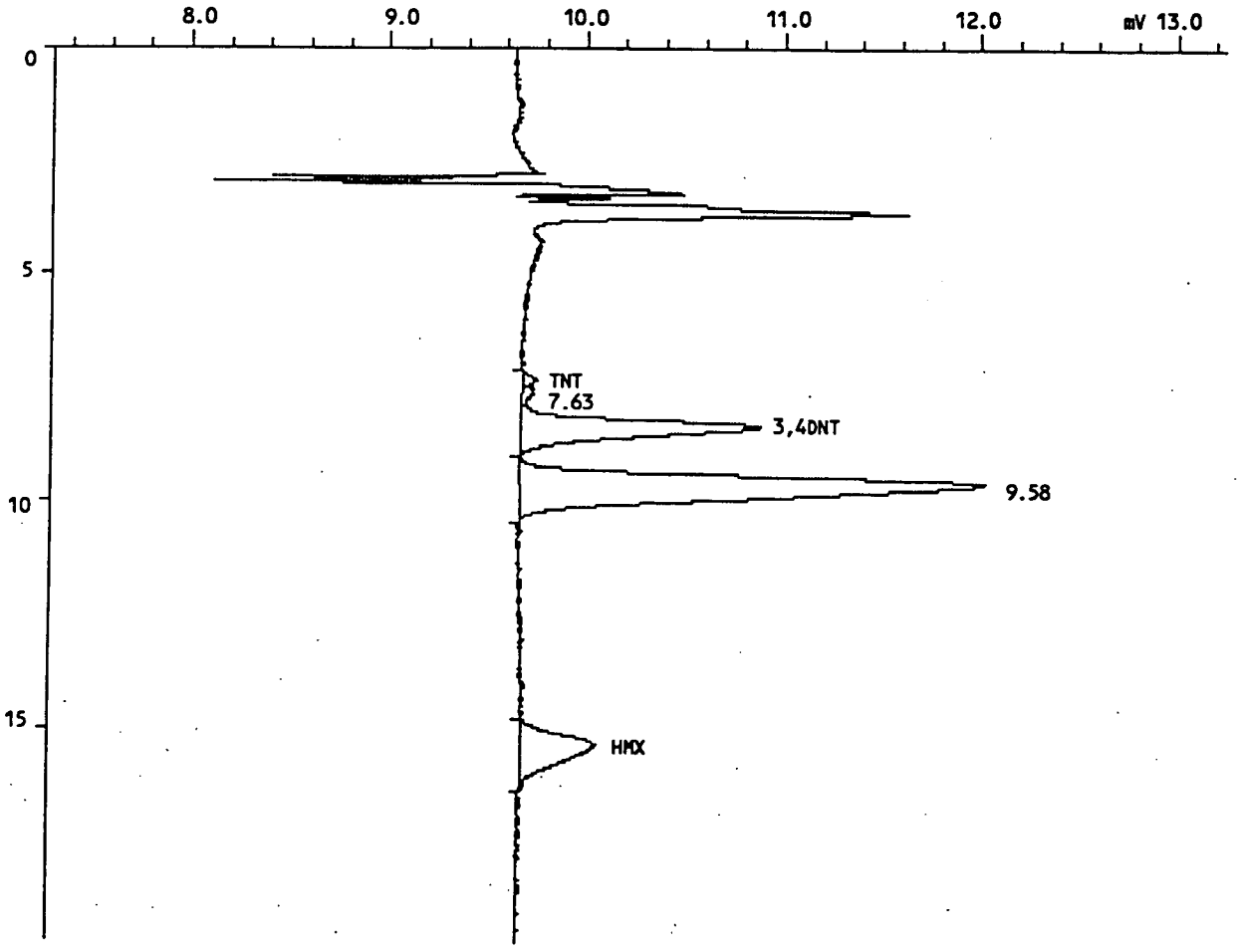
Peak	RT mins	Hght uV	Area uVs	ug/Kg	Peak name
1	4.859	1344	9875	14479.481	HMX *
2	7.371	3571	51636	60891.621	RDX *
3	15.067	1052	21757	19530.586	3,4DNT 98
4	15.723	64	1500	823.147	TNT *PJ
5	16.437	45	1508	1209.947	4ADNT *PJ
6	17.371	24	636	354.710	2ADNT
Total		6101	86911	97289.492	
Residual		0	0	0.000	

LONG PLOT

Injection F: <MC3> 3 3CN1027,15,1

Sample name.....: CRA/BPSAD9 10X
Sample ID.....: 31436.11
INST ID: IN10, COL. TYPE: LC-CN, ID: LC-CN-07, INJ:100, UV

Acquired on 28-Oct-97 at 06:25:35
Reported on 05-Nov-97 at 11:43:36



INJECTION REPORT

Injection F: <MC3> 3 3CN1027,15,1

Acquired on 28-Oct-97 at 06:25:35
Modified on 05-Nov-81 at 10:53:14
Reported on 05-Nov-97 at 10:53:14

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN10, COL. TYPE: LC-CN, ID: LC-CN-07, INJ:100, UV
Number of samples.: 56
Calibration file..: 3CN1027A Last modified on 05-Nov-81 at 10:52:08
Method file.....: LCCN Last modified on 05-Nov-81 at 10:52:26
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: CRA/BPSAD9 10X
Sample ID.....: 31436.11
Sample type.....: Sample
Sample amount.....: 2.0000
Number of injections.....: 1
Bottle Number.....: 1

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 20.000
Dilution.....: 10.000
Amount (g or mL).....: 2.000

PEAK INFORMATION

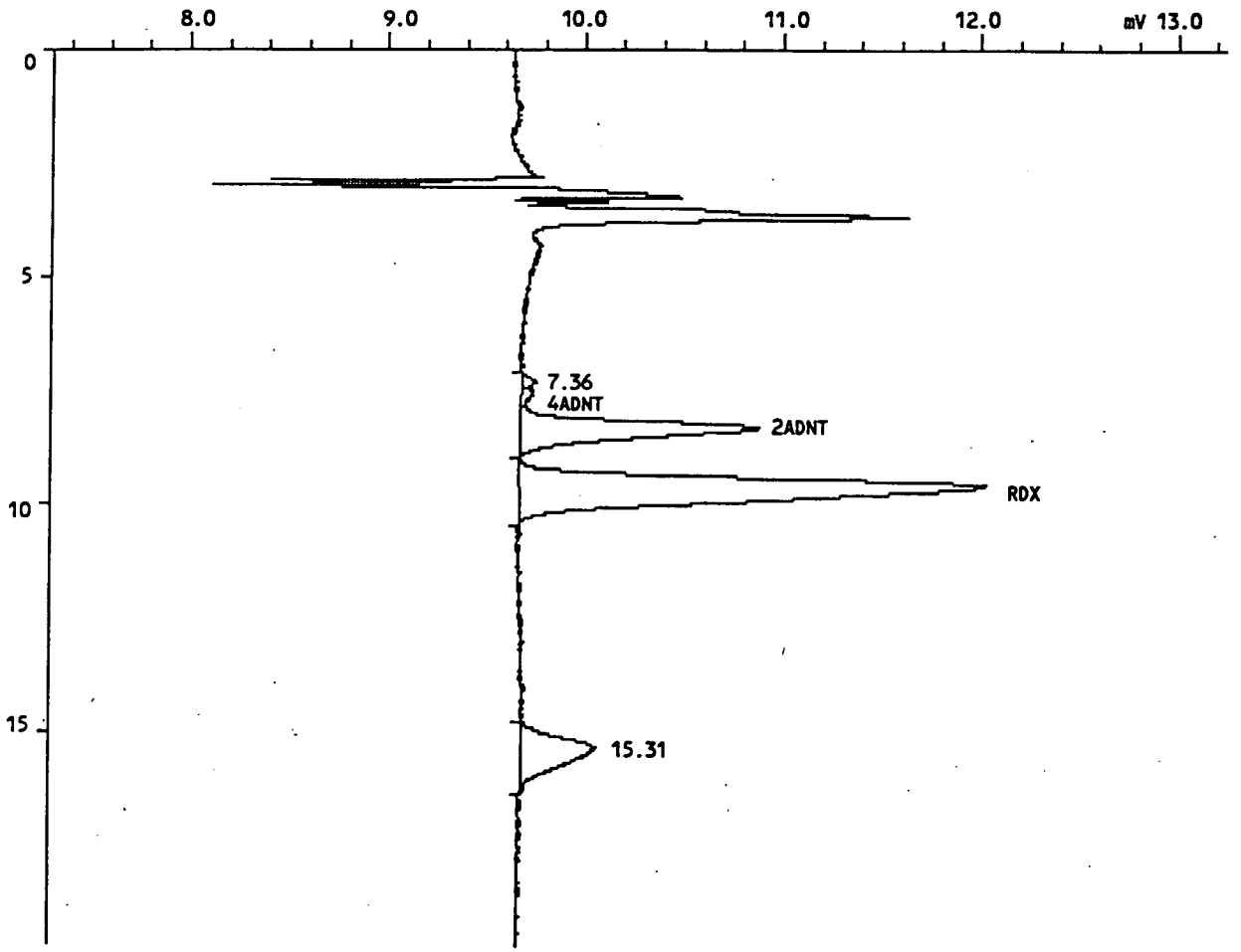
Peak	RT mins	Hght uV	Area uVs	ug/Kg	Peak name
1	7.360	74	813	313.307	TNT
3	8.331	1209	29096	17454.082	3,4DNT
5	15.307	377	15490	14005.203	HMX
Total		1661	45398	31772.592	
Residual		2429	75849	0.000	

LONG PLOT

Injection F: <MC3> 3 3CN1027,15,1

Sample name.....: CRA/BPSAD9 10X
Sample ID.....: 31436.11
INST ID: IN10, COL. TYPE: LC-CN, ID: LC-CN-07, INJ:100, UV

Acquired on 28-Oct-97 at 06:25:35
Reported on 05-Nov-97 at 11:50:56



INJECTION REPORT

Injection F: <MC3> 3 3CN1027,15,1

Acquired on 28-Oct-97 at 06:25:35
Modified on 05-Nov-81 at 11:44:50
Reported on 05-Nov-97 at 11:44:51

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN10, COL. TYPE: LC-CN, ID: LC-CN-07, INJ:100, UV
Number of samples.: 56
Calibration file..: 3CN1027B Last modified on 05-Nov-81 at 10:42:36
Method file.....: LCCN Last modified on 05-Nov-81 at 11:44:20
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: CRA/BPSAD9 10X
Sample ID.....: 31436.11
Sample type.....: Sample
Sample amount.....: 2.0000
Number of injections.....: 1
Bottle Number.....: 1

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 20.000
Dilution.....: 10.000
Amount (g or mL).....: 2.000

PEAK INFORMATION

Peak	RT mins	Hght uV	Area uVs	ug/Kg	Peak name
2	7.632	64	1094	578.012	4ADNT
3	8.331	1209	29096	11216.636	2ADNT
4	9.579	2366	74755	51733.520	RDX
Total		3639	104945	63528.168	
Residual		451	16302	0.000	

1D

EXPLOSIVE ANALYSIS DATA SHEET

EPA SAMPLE NO:

CRA/BPSAD10 50X

Lab Name: SWL-TULSA Contract:

Lab Code: SWOK Case No:MKF-OH SDG No: 31436

Matrix: (soil/water) SOIL Lab Sample ID: 31436.12

Sample Amt: 2gm % Moisture 15.76 Date Received: 10/14/97

Extraction Volume: 10ml Date Extracted: 10/14/97

Extraction Method: SONC Date Analyzed: 10/24/97

GPC Cleanup: (Y/N) N Dilution Factor: 50.00

CAS NO.	COMPOUND	CONCENTRATION UNITS: (ug/L or ug/kg) ug/kg	Q
2691-41-0	HMX	35000	J
121-82-4	RDX	310000	
99-35-4	TNB	6250	U
99-65-0	DNB	6250	U
479-45-8	TETRYL	16200	U
98-95-3	NB	6500	U
118-96-7	TNT	18700	
1946-51-0	4ADNT	6250	U
35572-78-2	2ADNT	6250	U
606-20-2	26DNT	6500	U
121-14-2	24DNT	6250	U
88-72-2	2NT	6250	U
99-99-0	4NT	6250	U
99-08-1	3NT	6250	U

SURROGATE AMOUNT SPIKED (ug/kg dry wt.) 20000
3,4-DNT

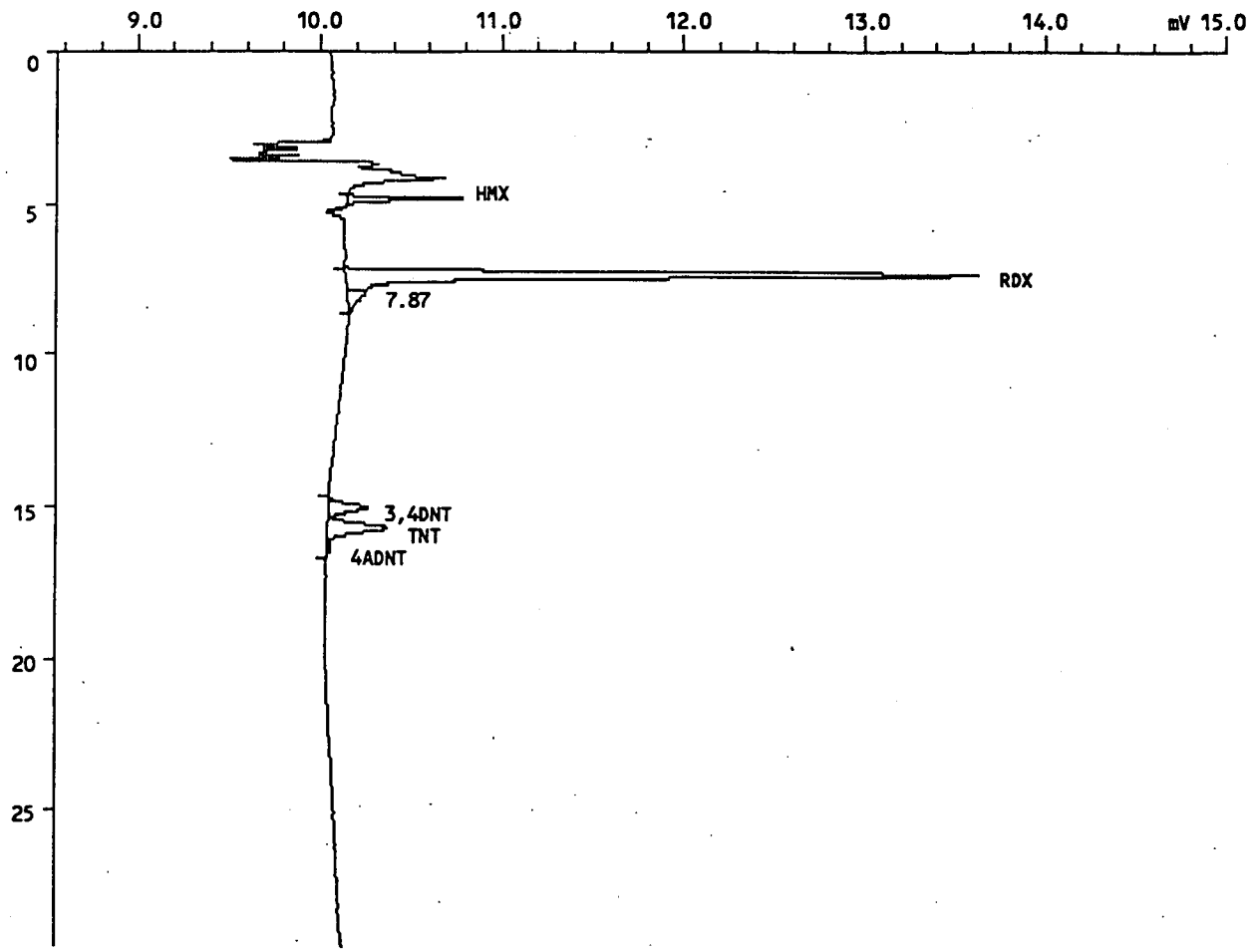
FORM I

LONG PLOT

Injection F: <MC3> 5 5EX1021E,5,1

Sample name.....: CRA/BPSAD10 50X
Sample ID.....: 31436.12
INST ID: IN13, COL. TYPE: ODS-II, ID: CARB-05, INJ:200,COL#1

Acquired on 24-Oct-97 at 21:06:29
Reported on 05-Nov-97 at 08:48:22



INJECTION REPORT

Injection F: <MC3> 5 5EX1021E,5,1

Acquired on 24-Oct-97 at 21:06:29
Modified on 05-Nov-81 at 08:44:44
Reported on 05-Nov-97 at 08:44:44

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN13, COL. TYPE: ODS-II, ID: CARB-05, INJ:200,COL#1
Number of samples.: 34
Calibration file..: 5EX1021 Last modified on 30-Oct-81 at 18:08:22
Method file.....: EXPLOS Last modified on 05-Nov-81 at 08:43:52
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: CRA/BPSAD10 50X
Sample ID.....: 31436.12
Sample type.....: Sample
Sample amount.....: 2.0000
Number of injections.....: 1
Bottle Number.....: 1

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 20.000
Dilution.....: 50.000
Amount (g or mL).....: 2.000

PEAK INFORMATION

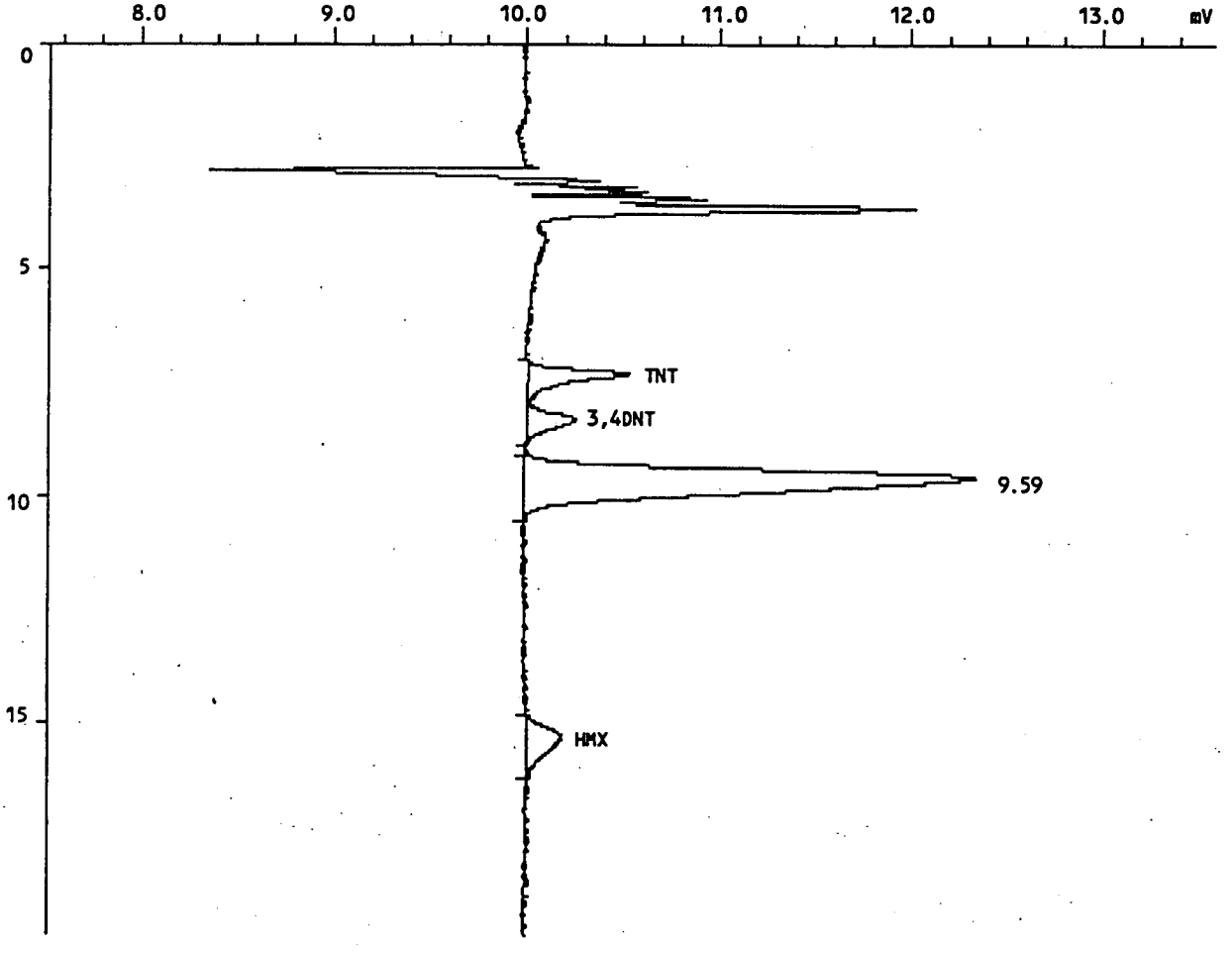
Peak	RT mins	Hght uV	Area uVs	ug/Kg	Peak name
1	4.859	637	4770	34968.023	HMX *
2	7.371	3507	52676	310591.656	RDX *
4	15.061	214	4155	18646.879	3,4DNT 23
5	15.739	322	6824	18727.934	TNT *
6	16.475	21	459	1843.673	4ADNT
Total		4701	68884	384778.188	
Residual		111	2600	15328.545	

LONG PLOT

Injection F: <MC3> 3 3CN1027,16,1

Sample name.....: CRA/BPSAD10 50X
Sample ID.....: 31436.12
INST ID: IN10, COL. TYPE: LC-CN, ID: LC-CN-07, INJ:100, UV

Acquired on 28-Oct-97 at 07:06:56
Reported on 05-Nov-97 at 14:32:04



INJECTION REPORT

Injection F: <MC3> 3 3CN1027,16,1

Acquired on 28-Oct-97 at 07:06:56
Modified on 05-Nov-81 at 14:31:16
Reported on 05-Nov-97 at 14:31:23

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN10, COL. TYPE: LC-CN, ID: LC-CN-07, INJ:100, UV
Number of samples.: 56
Calibration file..: 3CN1027A Last modified on 05-Nov-81 at 10:52:08
Method file.....: LCCN Last modified on 05-Nov-81 at 11:44:20
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: CRA/BPSAD10 50X
Sample ID.....: 31436.12
Sample type.....: Sample
Sample amount.....: 2.0000
Number of injections.....: 1
Bottle Number.....: 1

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 20.000
Dilution.....: 50.000
Amount (g or mL).....: 2.000

PEAK INFORMATION

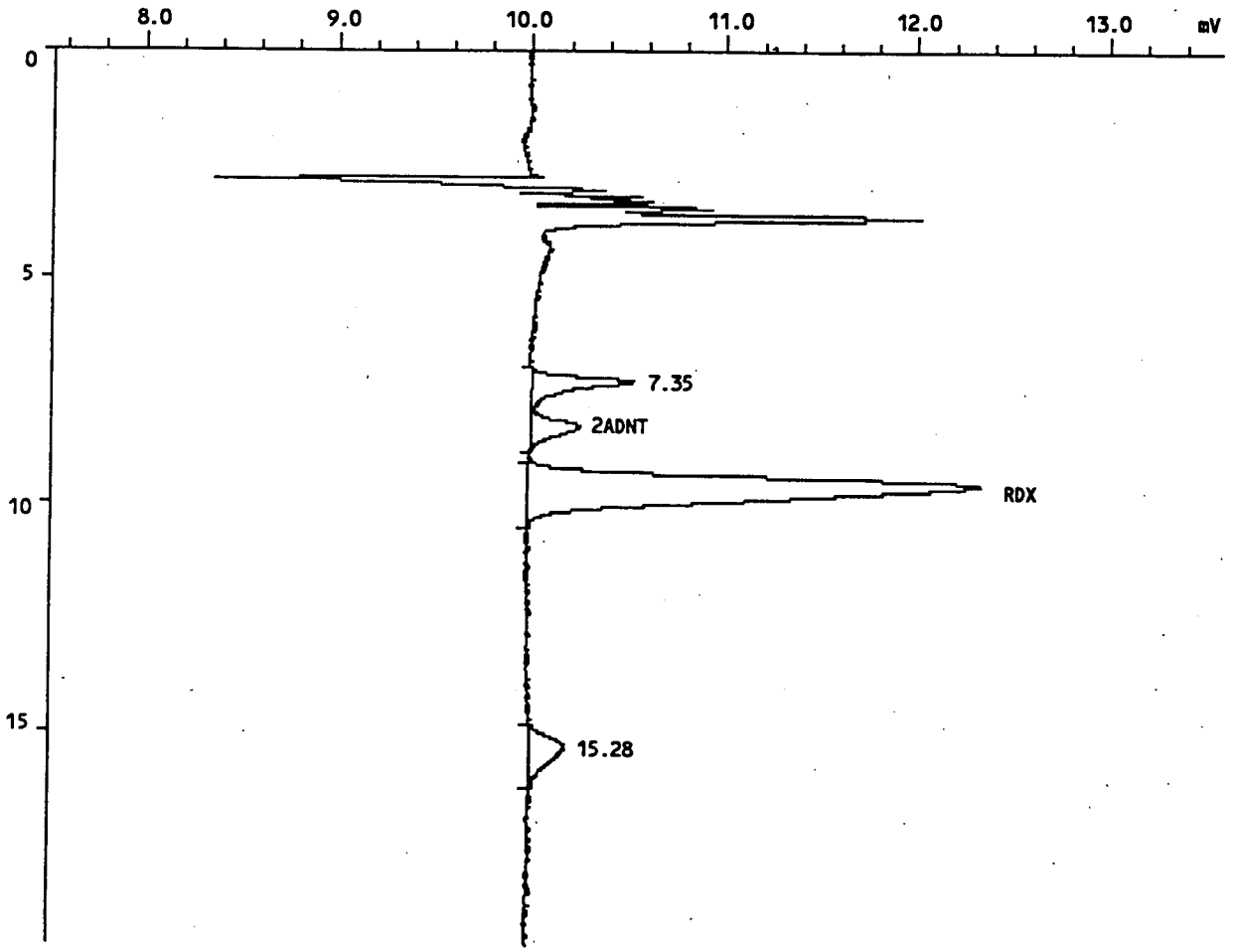
Peak	RT mins	Hght uV	Area uVs	ug/Kg	Peak name
1	7.349	525	9529	18368.229	TNT
2	8.309	258	5858	17571.121	3,4DNT
4	15.280	184	7255	32799.254	HMX
Total		967	22643	68738.609	
Residual		2355	75050	0.000	

LONG PLOT

Injection F: <MC3> 3 3CN1027,16,1

Sample name.....: CRA/BPSAD10 50X
Sample ID.....: 31436.12
INST ID: IN10, COL. TYPE: LC-CN, ID: LC-CN-07, INJ:100, UV

Acquired on 28-Oct-97 at 07:06:56
Reported on 05-Nov-97 at 14:33:09



INJECTION REPORT

Injection F: <MC3> 3 3CN1027,16,1

Acquired on 28-Oct-97 at 07:06:56
Modified on 05-Nov-81 at 14:32:36
Reported on 05-Nov-97 at 14:32:42

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN10, COL. TYPE: LC-CN, ID: LC-CN-07, INJ:100, UV
Number of samples.: 56
Calibration file..: 3CN1027B Last modified on 05-Nov-81 at 10:42:36
Method file.....: LCCN Last modified on 05-Nov-81 at 11:44:20
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: CRA/BPSAD10 50X
Sample ID.....: 31436.12
Sample type.....: Sample
Sample amount.....: 2.0000
Number of injections.....: 1
Bottle Number.....: 1

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 20.000
Dilution.....: 50.000
Amount (g or mL).....: 2.000

PEAK INFORMATION

Peak	RT mins	Hght uV	Area uVs	ug/Kg	Peak name
2	8.309	258	5858	11291.849	2ADNT
3	9.589	2355	75050	259687.625	RDX
Total		2613	80908	270979.469	
Residual		709	16785	0.000	

1D
EXPLOSIVE ANALYSIS DATA SHEET

EPA SAMPLE NO:

CRA/BPSAD12 250X

`b Name: SWL-TULSA Contract:

Lab Code: SWOK Case No:MKF-OH SDG No: 31436

Matrix: (soil/water) SOIL Lab Sample ID: 31436.13

Sample Amt: 2gm % Moisture 14.86 Date Received: 10/14/97

Extraction Volume: 10ml Date Extracted: 10/14/97

Extraction Method: SONC Date Analyzed: 10/24/97

GPC Cleanup: (Y/N) N Dilution Factor: 250.00

CAS NO.	COMPOUND	CONCENTRATION UNITS: (ug/L or ug/kg) ug/kg	Q
2691-41-0	HMX	374000	
121-82-4	RDX	3480000	
99-35-4	TNB	31200	U
99-65-0	DNB	31200	U
479-45-8	TETRYL	81200	U
98-95-3	NB	32500	U
118-96-7	TNT	547000	
1946-51-0	4ADNT	31200	U
35572-78-2	2ADNT	31200	U
606-20-2	26DNT	32500	U
121-14-2	24DNT	31200	U
88-72-2	2NT	31200	U
99-99-0	4NT	31200	U
99-08-1	3NT	31200	U

SURROGATE AMOUNT SPIKED (ug/kg dry wt.) 20000
3,4-DNT

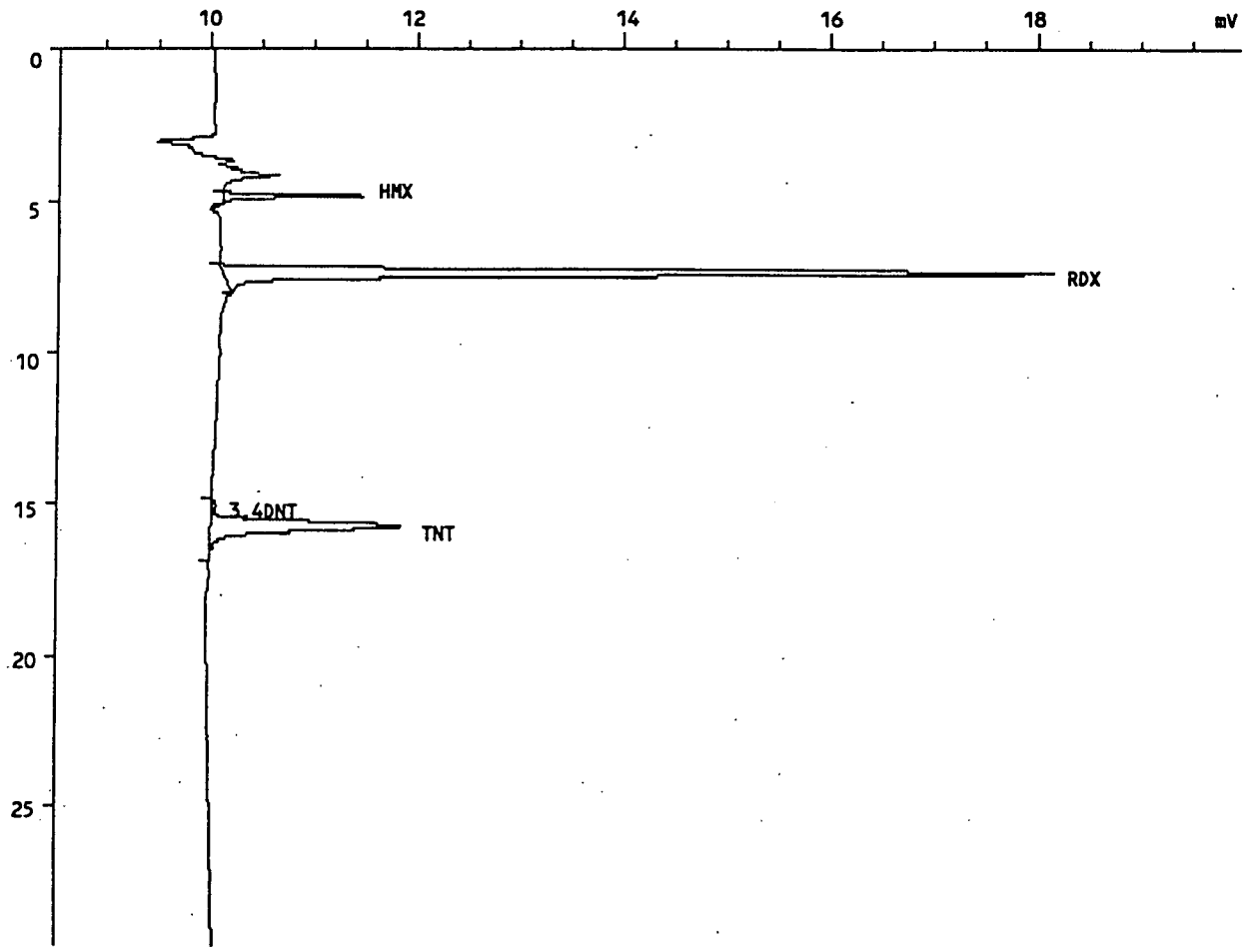
FORM I

LONG PLOT

Injection F: <MC3> 5 5EX1021E,6,1

Sample name.....: CRA/BPSAD12 250X
Sample ID.....: 31436.13
INST ID: IN13, COL. TYPE: ODS-II, ID: CARB-05, INJ:200,COL#1

Acquired on 24-Oct-97 at 21:51:10
Reported on 05-Nov-97 at 08:47:59



INJECTION REPORT

Injection F: <MC3> 5 5EX1021E,6,1

Acquired on 24-Oct-97 at 21:51:10
Modified on 05-Nov-81 at 08:45:00
Reported on 05-Nov-97 at 08:45:00

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN13, COL. TYPE: ODS-II, ID: CARB-05, INJ:200,COL#1
Number of samples.: 34
Calibration file..: 5EX1021 Last modified on 30-Oct-81 at 18:08:22
Method file.....: EXPLOS Last modified on 05-Nov-81 at 08:43:52
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: CRA/BPSAD12 250X
Sample ID.....: 31436.13
Sample type.....: Sample
Sample amount.....: 2.0000
Number of injections.....: 1
Bottle Number.....: 1

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 20.000
Dilution.....: 250.000
Amount (g or mL).....: 2.000

PEAK INFORMATION

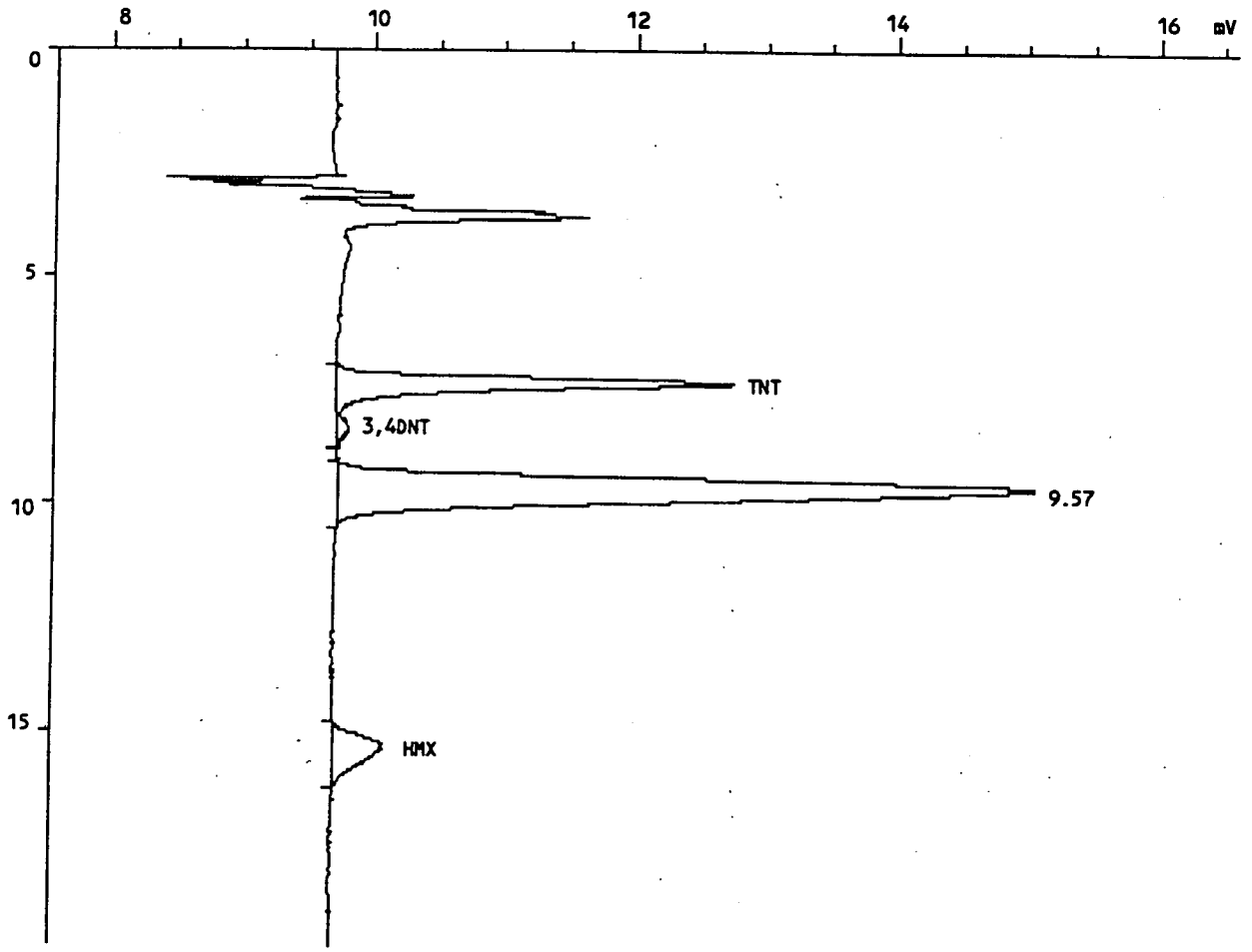
Peak	RT mins	Hght uV	Area uVs	ug/Kg	Peak name
1	4.859	1361	10204	374031.219	HMX *
2	7.376	8022	118200	3484673.000	RDX *
3	15.104	50	896	20107.625	3,4DNT 60
4	15.760	1839	39874	547113.000	TNT *
Total		11271	169173	4425925.000	
Residual		0	0	0.000	

LONG PLOT

Injection F: <MC3> 3 3CN1027,17,1

Sample name.....: CRA/BPSAD12 250X
Sample ID.....: 31436.13
INST ID: IN10, COL. TYPE: LC-CN, ID: LC-CN-07, INJ:100, UV

Acquired on 28-Oct-97 at 07:48:18
Reported on 05-Nov-97 at 11:08:49



INJECTION REPORT

Injection F: <MC3> 3 3CN1027,17,1

Acquired on 28-Oct-97 at 07:48:18
Modified on 05-Nov-81 at 10:53:42
Reported on 05-Nov-97 at 10:53:41

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN10, COL. TYPE: LC-CN, ID: LC-CN-07, INJ:100, UV
Number of samples.: 56
Calibration file..: 3CN1027A Last modified on 05-Nov-81 at 10:52:08
Method file.....: LCCN Last modified on 05-Nov-81 at 10:52:26
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: CRA/BPSAD12 250X
Sample ID.....: 31436.13
Sample type.....: Sample
Sample amount.....: 2.0000
Number of injections.....: 1
Bottle Number.....: 1

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 20.000
Dilution.....: 250.000
Amount (g or mL).....: 2.000

PEAK INFORMATION

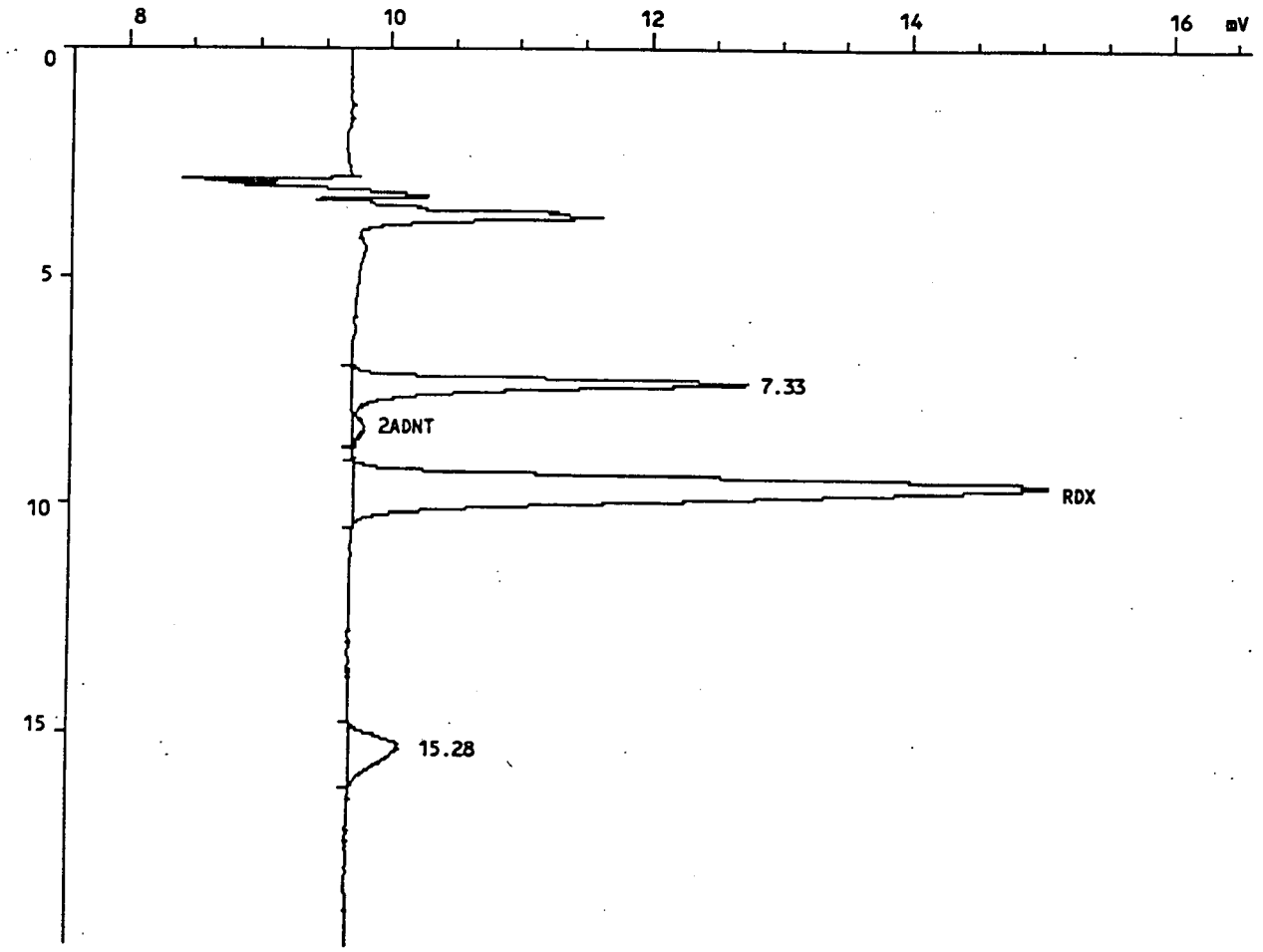
Peak	RT mins	Hght uV	Area uVs	ug/Kg	Peak name
1	7.333	3037	54123	521613.094	TNT
2	8.320	96	2368	35516.184	3,4DNT
4	15.280	382	15960	360769.625	HMX
Total		3515	72451	917898.875	
Residual		5345	170459	0.000	

LONG PLOT

Injection F: <MC3> 3 3CN1027,17,1

Sample name.....: CRA/BPSAD12 250X
Sample ID.....: 31436.13
INST ID: IN10, COL. TYPE: LC-CN, ID: LC-CN-07, INJ:100, UV

Acquired on 28-Oct-97 at 07:48:18
Reported on 05-Nov-97 at 11:50:02



INJECTION REPORT

Injection F: <MC3> 3 3CN1027,17,1

Acquired on 28-Oct-97 at 07:48:18
Modified on 05-Nov-81 at 11:45:18
Reported on 05-Nov-97 at 11:45:18

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN1C, COL. TYPE: LC-CN, ID: LC-CN-07, INJ:100, UV
Number of samples.: 56
Calibration file..: 3CN1027B Last modified on 05-Nov-81 at 10:42:36
Method file.....: LCCN Last modified on 05-Nov-81 at 11:44:20
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: CRA/BPSAD12 250X
Sample ID.....: 31436.13
Sample type.....: Sample
Sample amount.....: 2.0000
Number of injections.....: 1
Bottle Number.....: 1

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 20.000
Dilution.....: 250.000
Amount (g or mL).....: 2.000

PEAK INFORMATION

Peak	RT mins	Hght uV	Area uVs	ug/Kg	Peak name
2	8.320	96	2368	22824.010	2ADNT
3	9.573	5345	170459	2949118.250	RDX
Total		5441	172827	2971942.250	
Residual		3419	70083	0.000	

1D
EXPLOSIVE ANALYSIS DATA SHEET

EPA SAMPLE NO:

CRA/BPSAD15 100X

b Name: SWL-TULSA Contract:

Lab Code: SWOK Case No:MKF-OH SDG No: 31436

Matrix: (soil/water) SOIL Lab Sample ID: 31436.14

Sample Amt: 2gm % Moisture 14.44 Date Received: 10/14/97

Extraction Volume: 10ml Date Extracted: 10/14/97

Extraction Method: SONC Date Analyzed: 10/24/97

GPC Cleanup: (Y/N) N Dilution Factor: 100.00

CAS NO.	COMPOUND	CONCENTRATION UNITS: (ug/L or ug/kg) ug/kg	Q
2691-41-0	HMX	140000	
121-82-4	RDX	1140000	
99-35-4	TNB	12500	U
99-65-0	DNB	12500	U
479-45-8	TETRYL	32500	U
98-95-3	NB	13000	U
118-96-7	TNT	123000	
1946-51-0	4ADNT	12500	U
35572-78-2	2ADNT	12500	U
606-20-2	26DNT	13000	U
121-14-2	24DNT	12500	U
88-72-2	2NT	12500	U
99-99-0	4NT	12500	U
99-08-1	3NT	12500	U

SURROGATE AMOUNT SPIKED (ug/kg dry wt.) 20000
3,4-DNT

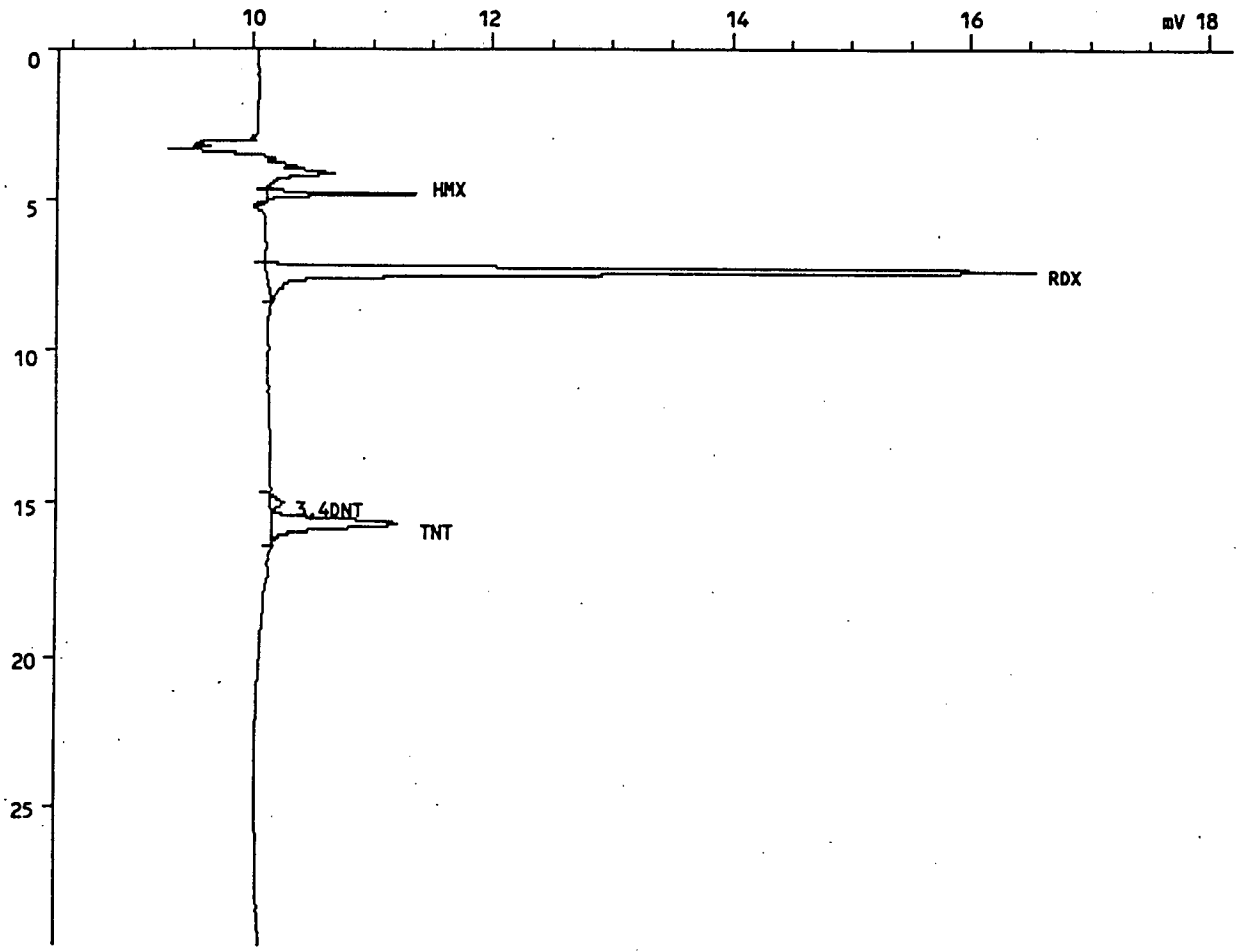
FORM I

LONG PLOT

Injection F: <MC3> 5 5EX1021E,7,1

Sample name.....: CRA/BPSAD15 100X
Sample ID.....: 31436.14
INST ID: IN13, COL. TYPE: ODS-II, ID: CARB-05, INJ:200,COL#1

Acquired on 24-Oct-97 at 22:35:50
Reported on 05-Nov-97 at 08:47:41



INJECTION REPORT

Injection F: <MC3> 5 5EX1021E,7,1

Acquired on 24-Oct-97 at 22:35:50
Modified on 05-Nov-81 at 08:45:14
Reported on 05-Nov-97 at 08:45:14

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN13, COL. TYPE: ODS-II, ID: CARB-05, INJ:200,COL#1
Number of samples.: 34
Calibration file..: 5EX1021 Last modified on 30-Oct-81 at 18:08:22
Method file.....: EXPLOS Last modified on 05-Nov-81 at 08:43:52
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: CRA/BPSAD15 100X
Sample ID.....: 31436.14
Sample type.....: Sample
Sample amount.....: 2.0000
Number of injections.....: 1
Bottle Number.....: 1

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 20.000
Dilution.....: 100.000
Amount (g or mL).....: 2.000

PEAK INFORMATION

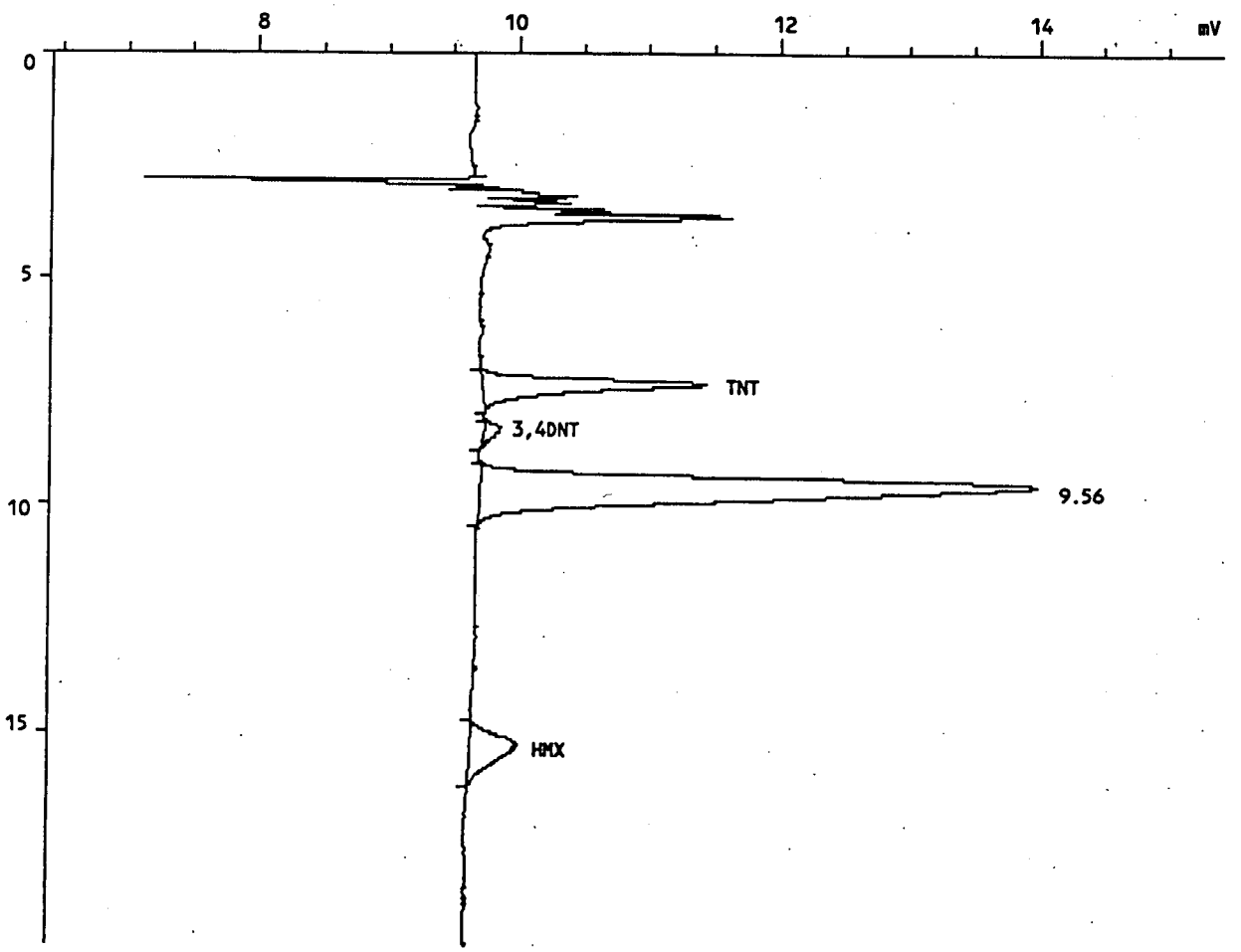
Peak	RT mins	Hght uV	Area uVs	ug/Kg	Peak name
1	4.843	1250	9524	139649.047	HMX *
2	7.355	6429	97046	1144408.750	RDX *
3	15.051	107	2155	19345.107	3,4DNT 97
4	15.723	1056	22366	122757.547	TNT *
Total		8843	131091	1426160.375	
Residual		0	0	0.000	

LONG PLOT

Injection F: <MC3> 3 3CN1027,18,1

Sample name.....: CRA/BPSAD15 100X
Sample ID.....: 31436.14
INST ID: IN10, COL. TYPE: LC-CN, ID: LC-CN-07, INJ:100, UV

Acquired on 28-Oct-97 at 08:29:39
Reported on 05-Nov-97 at 11:09:08



INJECTION REPORT

Injection F: <MC3> 3 3CN1027,18,1

Acquired on 28-Oct-97 at 08:29:39
Modified on 05-Nov-81 at 10:53:54
Reported on 05-Nov-97 at 10:53:55

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN10, COL. TYPE: LC-CN, ID: LC-CN-07, INJ:100, UV
Number of samples.: 56
Calibration file..: 3CN1027A Last modified on 05-Nov-81 at 10:52:08
Method file.....: LCCN Last modified on 05-Nov-81 at 10:52:26
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: CRA/BPSAD15 100X
Sample ID.....: 31436.14
Sample type.....: Sample
Sample amount.....: 2.0000
Number of injections.....: 1
Bottle Number.....: 1

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 20.000
Dilution.....: 100.000
Amount (g or mL).....: 2.000

PEAK INFORMATION

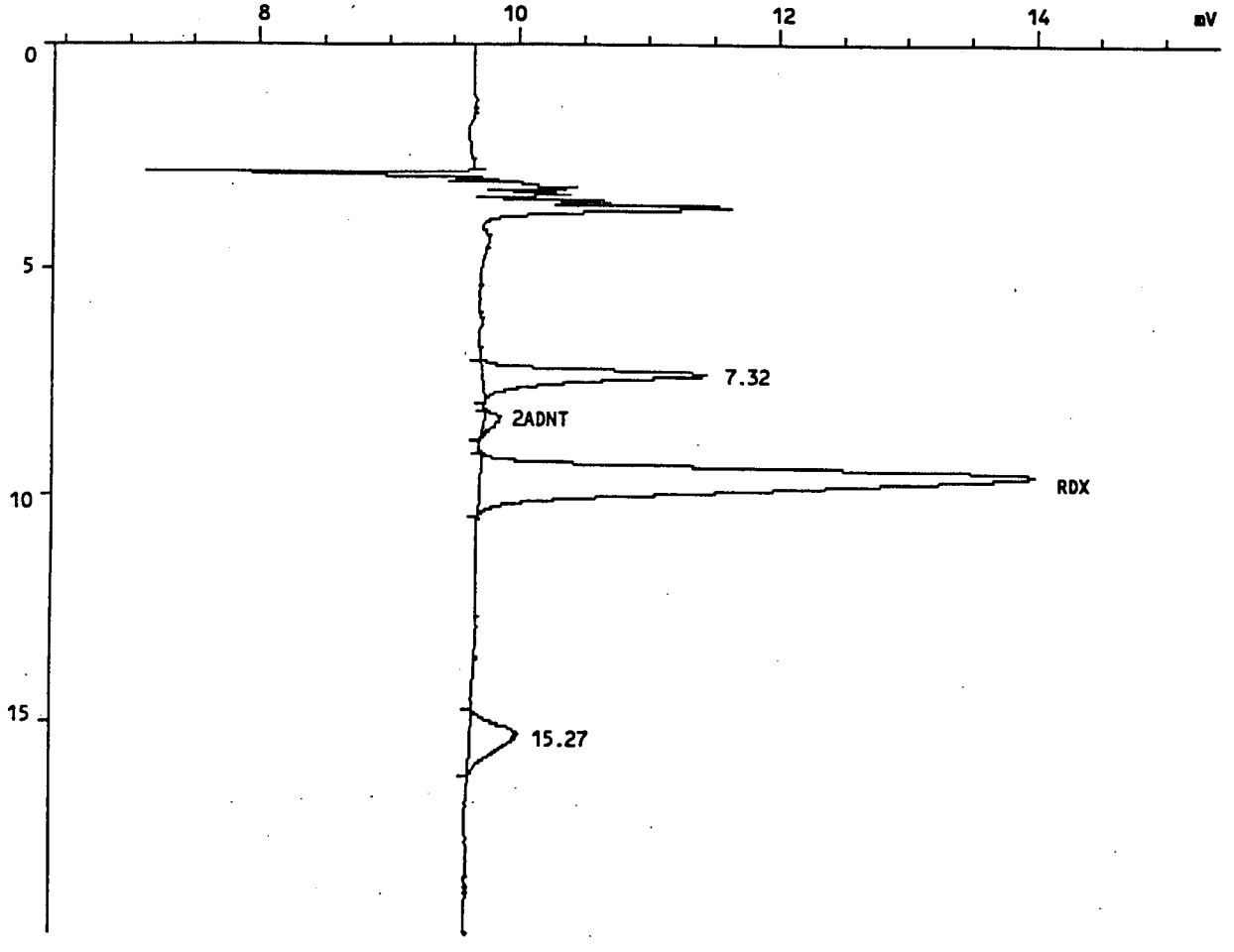
Peak	RT mins	Hght uV	Area uVs	ug/Kg	Peak name
1	7.317	1730	30528	117686.930	TNT
2	8.331	131	2587	15516.035	3,4DNT
4	15.269	358	14784	133670.313	HMX
Total		2220	47898	266873.281	
Residual		4296	136107	0.000	

LONG PLOT

Injection F: <MC3> 3 3CN1027,18,1

Sample name.....: CRA/BPSAD15 100X
Sample ID.....: 31436.14
INST ID: IN10, COL. TYPE: LC-CN, ID: LC-CN-07, INJ:100, UV

Acquired on 28-Oct-97 at 08:29:39
Reported on 05-Nov-97 at 11:47:53



INJECTION REPORT

Injection F: <MC3> 3 3CN1027,18,1

Acquired on 28-Oct-97 at 08:29:39
Modified on 05-Nov-81 at 11:45:30
Reported on 05-Nov-97 at 11:45:31

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN10, COL. TYPE: LC-CN, ID: LC-CN-07, INJ:100, UV
Number of samples.: 56
Calibration file..: 3CN1027B Last modified on 05-Nov-81 at 10:42:36
Method file.....: LCCN Last modified on 05-Nov-81 at 11:44:20
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: CRA/BPSAD15 100X
Sample ID.....: 31436.14
Sample type.....: Sample
Sample amount.....: 2.0000
Number of injections.....: 1
Bottle Number.....: 1

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 20.000
Dilution.....: 100.000
Amount (g or mL).....: 2.000

PEAK INFORMATION

Peak	RT mins	Hght uV	Area uVs	ug/Kg	Peak name
2	8.331	131	2587	9971.176	2ADNT
3	9.557	4427	136107	941916.375	RDX
Total		4427	138693	951887.563	
Residual		2088	45312	0.000	

1D
EXPLOSIVE ANALYSIS DATA SHEET

EPA SAMPLE NO:

CRA/BPSAD16 200X

Lab Name: SWL-TULSA Contract:

Lab Code: SWOK Case No:MKF-OH SDG No: 31436

Matrix: (soil/water) SOIL Lab Sample ID: 31436.15

Sample Amt: 2gm % Moisture 14.28 Date Received: 10/14/97

Extraction Volume: 10ml Date Extracted: 10/14/97

Extraction Method: SONC Date Analyzed: 10/24/97

GPC Cleanup: (Y/N) N Dilution Factor: 200.00

CAS NO.	COMPOUND	CONCENTRATION UNITS: (ug/L or ug/kg) ug/kg	Q
2691-41-0	HMX	343000	
121-82-4	RDX	3360000	
99-35-4	TNB	25000	U
99-65-0	DNB	25000	U
479-45-8	TETRYL	65000	U
98-95-3	NB	26000	U
118-96-7	TNT	367000	
1946-51-0	4ADNT	25000	U
35572-78-2	2ADNT	25000	U
606-20-2	26DNT	26000	U
121-14-2	24DNT	25000	U
88-72-2	2NT	25000	U
99-99-0	4NT	25000	U
99-08-1	3NT	25000	U

SURROGATE AMOUNT SPIKED (ug/kg dry wt.) 20000
3,4-DNT

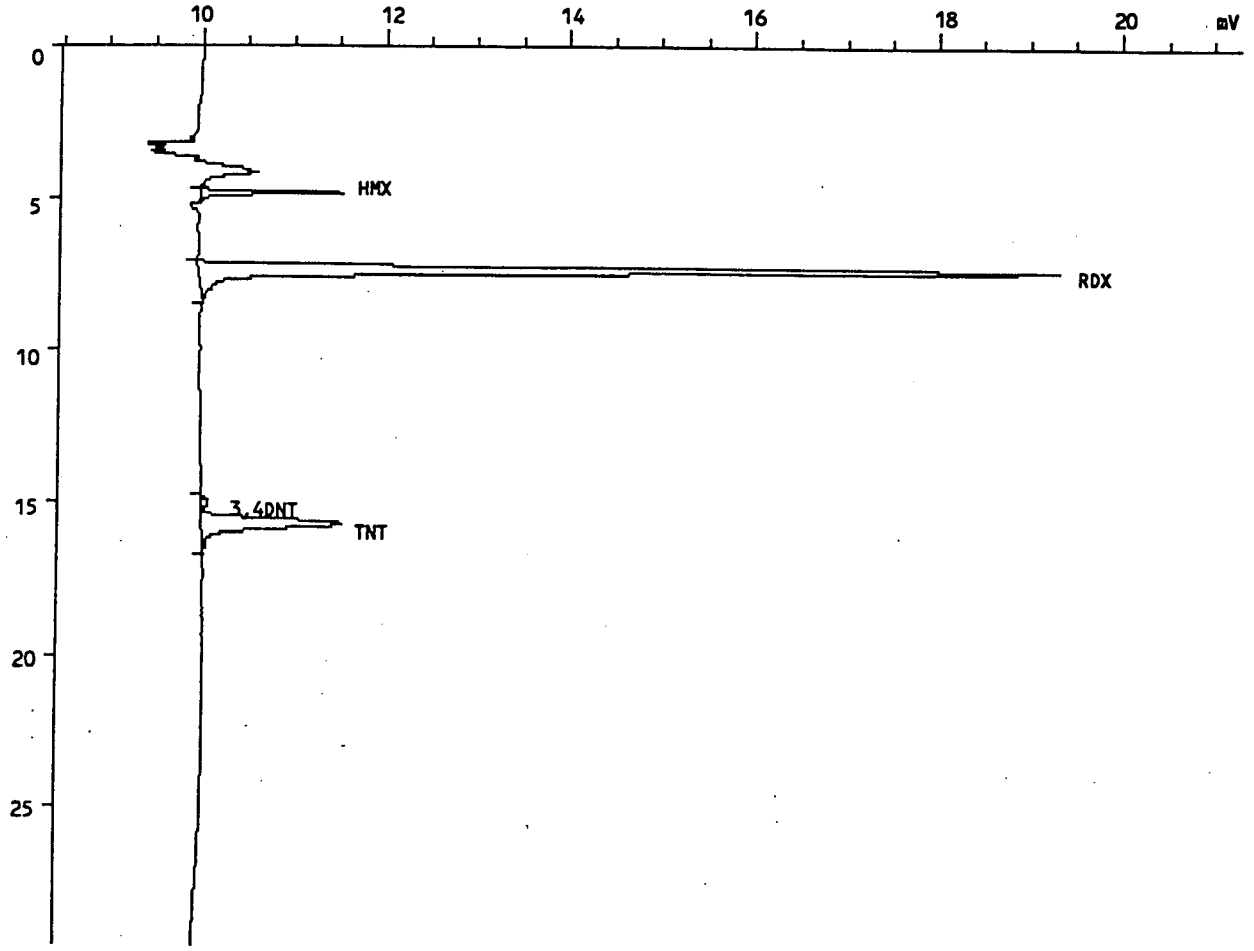
FORM I

LONG PLOT

Injection F: <MC3> 5 5EX1021E,8,1

Sample name.....: CRA/BPSAD16 200X
Sample ID.....: 31436.15
INST ID: IN13, COL. TYPE: ODS-II, ID: CARB-05, INJ:200,COL#1

Acquired on 24-Oct-97 at 23:20:31
Reported on 05-Nov-97 at 08:47:23



INJECTION REPORT

Injection F: <MC3> 5 5EX1021E,8,1

Acquired on 24-Oct-97 at 23:20:31
Modified on 05-Nov-81 at 08:45:28
Reported on 05-Nov-97 at 08:49:17

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN13, COL. TYPE: ODS-II, ID: CARB-05, INJ:200,COL#1
Number of samples.: 34
Calibration file..: 5EX1021 Last modified on 30-Oct-81 at 18:08:22
Method file.....: EXPLOS Last modified on 05-Nov-81 at 08:43:52
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: CRA/BPSAD16 200X
Sample ID.....: 31436.15
Sample type.....: Sample
Sample amount.....: 2.0000
Number of injections.....: 1
Bottle Number.....: 1

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 20.000
Dilution.....: 200.000
Amount (g or mL).....: 2.000

PEAK INFORMATION

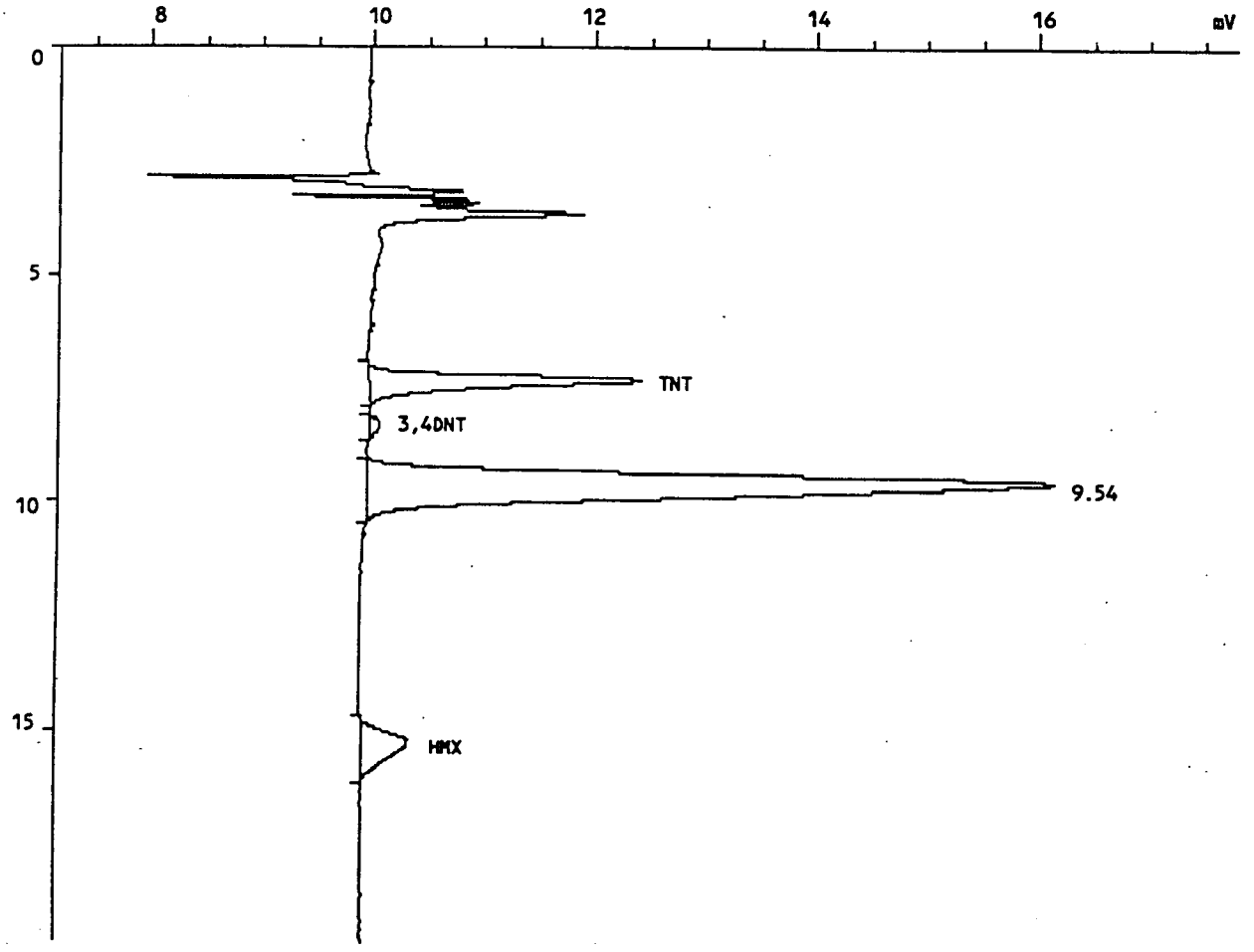
Peak	RT mins	Hght uV	Area uVs	ug/Kg	Peak name
1	4.864	1551	11702	343161.500	HMX *
2	7.376	9388	142328	3356786.000	RDX *
3	15.067	81	1556	27935.467	3,4DNT 140
4	15.733	1535	33439	367054.594	TNT *
Total		12554	189024	4094937.500	
Residual		0	0	0.000	

LONG PLOT

Injection F: <MC3> 3 3CN1027,19,1

Sample name.....: CRA/BPSAD16 200X
Sample ID.....: 31436.15
INST ID: IN10, COL. TYPE: LC-CN, ID: LC-CN-07, INJ:100, UV

Acquired on 28-Oct-97 at 09:11:00
Reported on 05-Nov-97 at 11:09:29



INJECTION REPORT

Injection F: <MC3> 3 3CN1027,19,1

Acquired on 28-Oct-97 at 09:11:00
Modified on 05-Nov-81 at 10:54:10
Reported on 05-Nov-97 at 10:54:09

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN10, COL. TYPE: LC-CN, ID: LC-CN-07, INJ:100, UV
Number of samples.: 56
Calibration file..: 3CN1027A Last modified on 05-Nov-81 at 10:52:08
Method file.....: LCCN Last modified on 05-Nov-81 at 10:52:26
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: CRA/BPSAD16 200X
Sample ID.....: 31436.15
Sample type.....: Sample
Sample amount.....: 2.0000
Number of injections.....: 1
Bottle Number.....: 1

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 20.000
Dilution.....: 200.000
Amount (g or mL).....: 2.000

PEAK INFORMATION

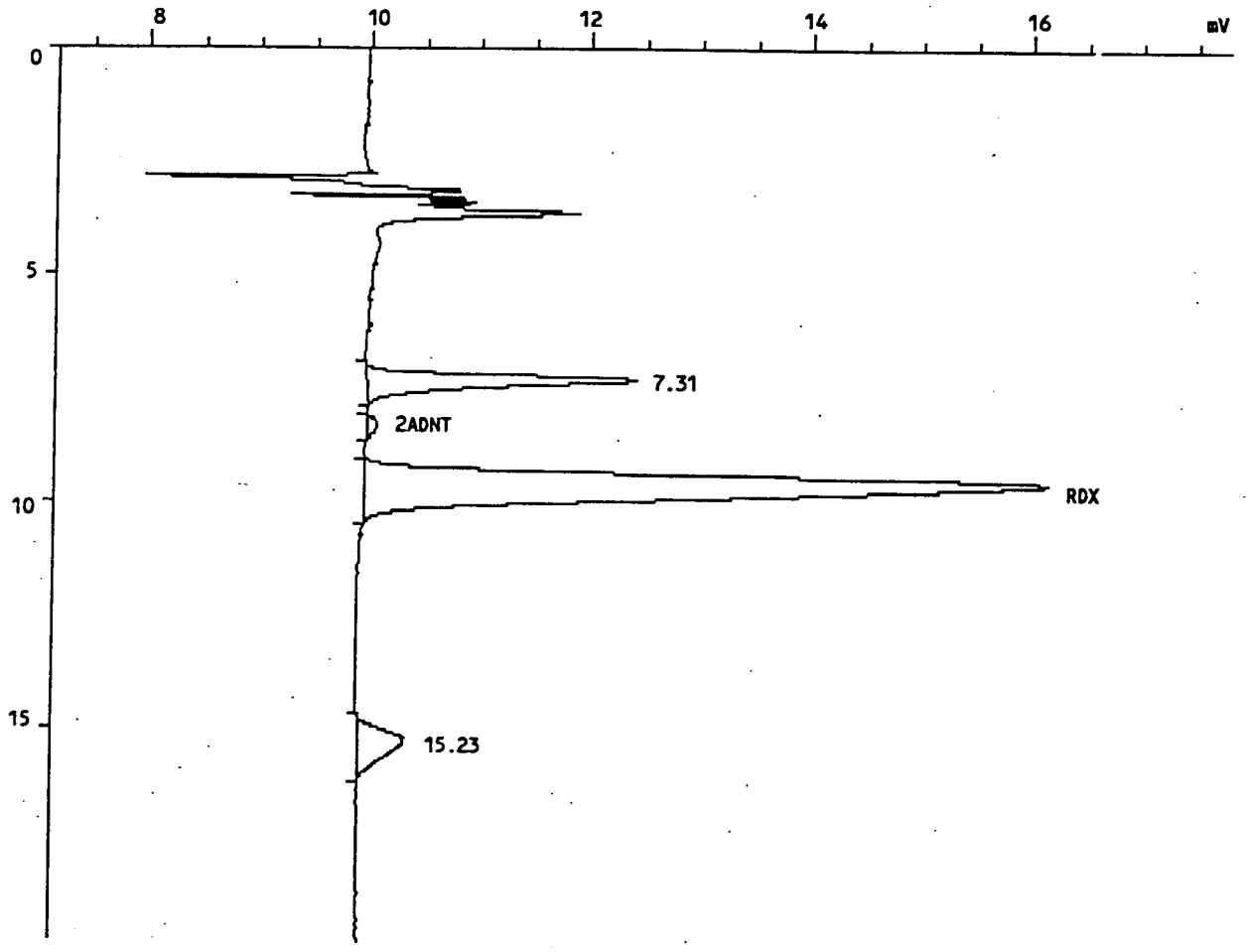
Peak	RT mins	Hght uV	Area uVs	ug/Kg	Peak name
1	7.312	2474	43658	336610.469	TNT
2	8.293	106	1976	23701.531	3,4DNT
4	15.232	428	17769	321318.219	HMX
Total		3008	63403	681630.250	
Residual		6212	198058	0.000	

LONG PLOT

Injection F: <MC3> 3 3CN1027,19,1

Sample name.....: CRA/BPSAD16 200X
Sample ID.....: 31436.15
INST ID: IN10, COL. TYPE: LC-CN, ID: LC-CN-07, INJ:100, UV

Acquired on 28-Oct-97 at 09:11:00
Reported on 05-Nov-97 at 11:47:33



INJECTION REPORT

Injection F: <MC3> 3 3CN1027,19,1

Acquired on 28-Oct-97 at 09:11:00
Modified on 05-Nov-81 at 11:45:44
Reported on 05-Nov-97 at 11:45:44

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN1C, COL. TYPE: LC-CN, ID: LC-CN-07, INJ:100, UV
Number of samples.: 56
Calibration file..: 3CN1027B Last modified on 05-Nov-81 at 10:42:36
Method file.....: LCCN Last modified on 05-Nov-81 at 11:44:20
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: CRA/BPSAD16 200X
Sample ID.....: 31436.15
Sample type.....: Sample
Sample amount.....: 2.0000
Number of injections.....: 1
Bottle Number.....: 1

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 20.000
Dilution.....: 200.000
Amount (g or mL).....: 2.000

PEAK INFORMATION

Peak	RT mins	Hght uV	Area uVs	ug/Kg	Peak name
2	8.293	106	1976	15231.478	2ADNT
3	9.541	6212	198058	2741280.250	RDX
Total		6318	200033	2756511.750	
Residual		2902	61427	0.000	

1D
EXPLOSIVE ANALYSIS DATA SHEET

EPA SAMPLE NO:

CRA/BPSAD17 50X

Lab Name: SWL-TULSA Contract:

Lab Code: SWOK Case No:MKF-OH SDG No: 31436

Matrix: (soil/water) SOIL Lab Sample ID: 31436.16

Sample Amt: 2gm % Moisture 16.04 Date Received: 10/14/97

Extraction Volume: 10ml Date Extracted: 10/14/97

Extraction Method: SONC Date Analyzed: 10/25/97

GPC Cleanup: (Y/N) N Dilution Factor: 50.00

CAS NO.	COMPOUND	CONCENTRATION UNITS: (ug/L or ug/kg) ug/kg	Q
2691-41-0	HMX	88700	
121-82-4	RDX	740000	
99-35-4	TNB	6250	U
99-65-0	DNB	6250	U
479-45-8	TETRYL	16200	U
98-95-3	NB	6500	U
118-96-7	TNT	18600	
1946-51-0	4ADNT	6250	U
35572-78-2	2ADNT	6250	U
606-20-2	26DNT	6500	U
121-14-2	24DNT	6250	U
88-72-2	2NT	6250	U
99-99-0	4NT	6250	U
99-08-1	3NT	6250	U

SURROGATE AMOUNT SPIKED (ug/kg dry wt.) 20000
3,4-DNT

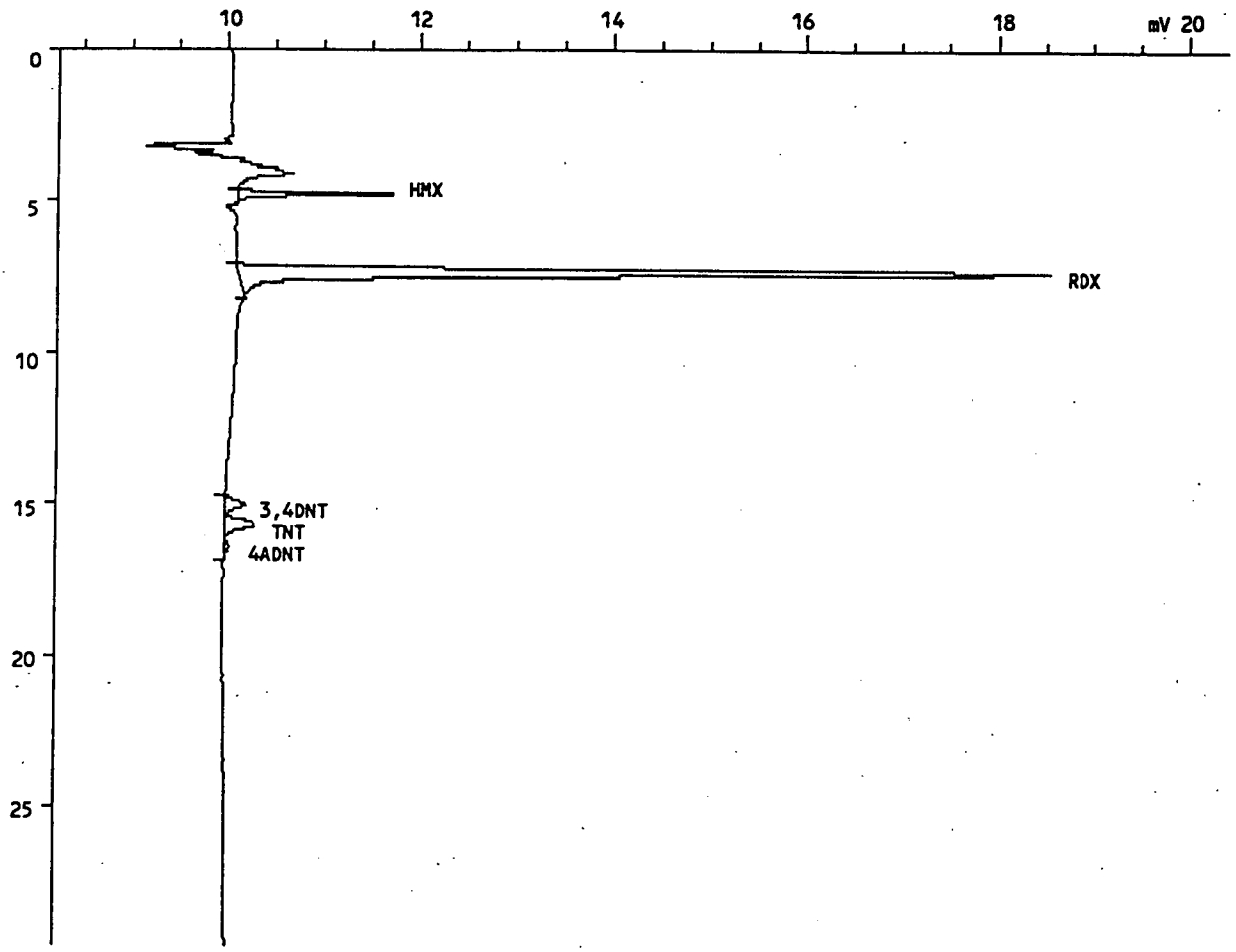
FORM I

LONG PLOT

Injection F: <MC3> 5 5EX1021E,9,1

Sample name.....: CRA/BPSAD17 50X
Sample ID.....: 31436.16
INST ID: IN13, COL. TYPE: ODS-II, ID: CARB-05, INJ:200,COL#1

Acquired on 25-Oct-97 at 00:05:13
Reported on 05-Nov-97 at 08:46:54



INJECTION REPORT

Injection F: <MC3> 5 5EX1021E,9,1

Acquired on 25-Oct-97 at 00:05:13
Modified on 05-Nov-81 at 08:46:10
Reported on 05-Nov-97 at 08:46:10

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN13, COL. TYPE: ODS-II, ID: CARB-05, INJ:200,COL#1
Number of samples.: 34
Calibration file..: 5EX1021 Last modified on 30-Oct-81 at 18:08:22
Method file.....: EXPLOS Last modified on 05-Nov-81 at 08:43:52
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: CRA/BPSAD17 50X
Sample ID.....: 31436.16
Sample type.....: Sample
Sample amount.....: 2.0000
Number of injections.....: 1
Bottle Number.....: 1

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 20.000
Dilution.....: 50.000
Amount (g or mL).....: 2.000

PEAK INFORMATION

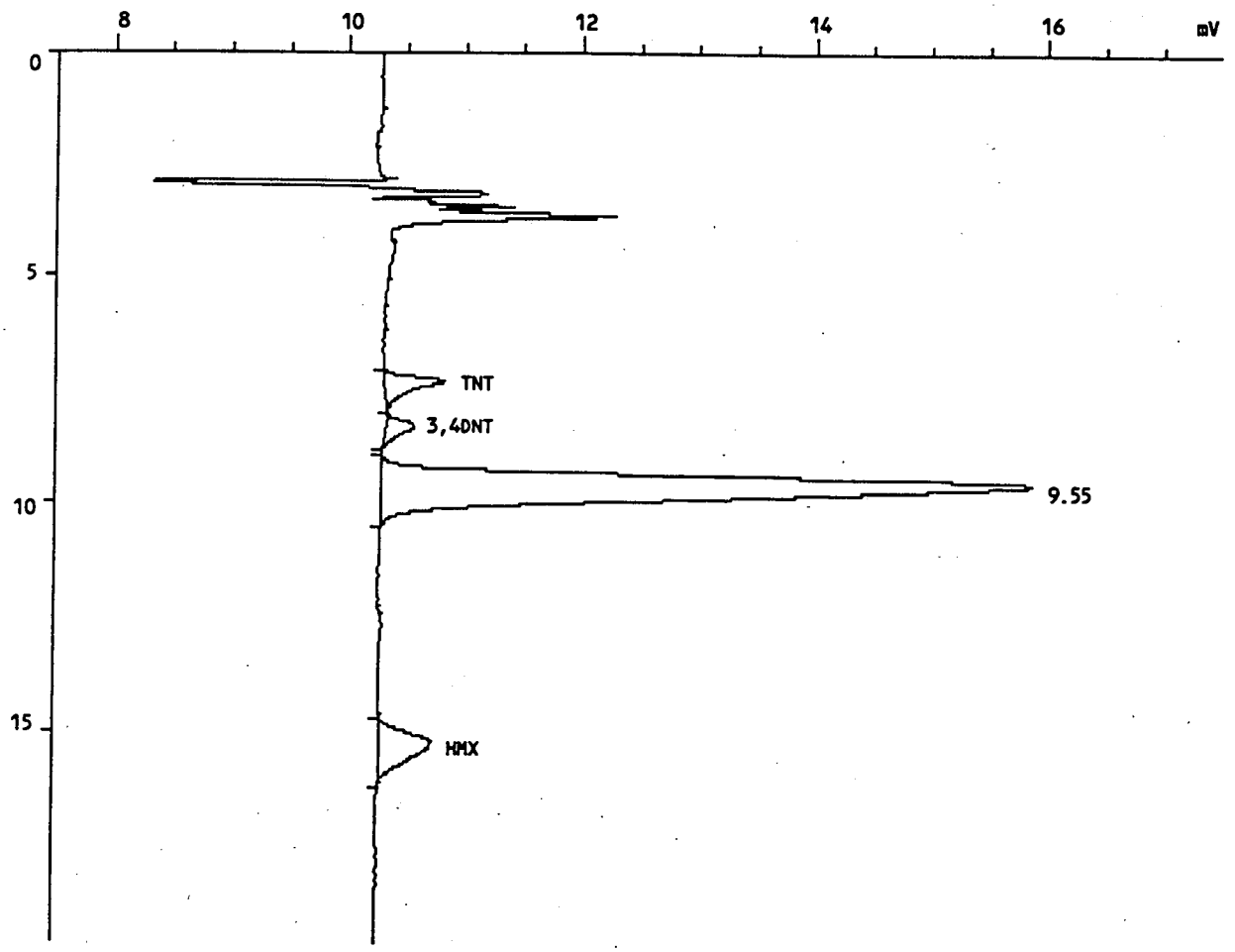
Peak	RT mins	Hght uV	Area uVs	ug/Kg	Peak name
1	4.848	1616	12100	88708.484	HMX *
2	7.365	8440	125513	740054.250	RDX *
3	15.083	213	4273	19178.939	3,4DNT 96
4	15.723	320	6798	18655.443	TNT *
5	16.395	56	1349	5411.802	4ADNT
Total		10646	150033	872008.938	
Residual		0	0	0.000	

LONG PLOT

Injection F: <MC3> 3 3CN1027,20,1

Sample name.....: CRA/BPSAD17 50X
Sample ID.....: 31436.16
INST ID: IN10, COL. TYPE: LC-CN, ID: LC-CN-07, INJ:100, UV

Acquired on 28-Oct-97 at 09:52:21
Reported on 05-Nov-97 at 11:09:52



INJECTION REPORT

Injection F: <MC3> 3 3CN1027,20,1

Acquired on 28-Oct-97 at 09:52:21
Modified on 05-Nov-81 at 10:54:24
Reported on 05-Nov-97 at 10:54:23

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN10, COL. TYPE: LC-CN, ID: LC-CN-07, INJ:100, UV
Number of samples.: 56
Calibration file..: 3CN1027A Last modified on 05-Nov-81 at 10:52:08
Method file.....: LCCN Last modified on 05-Nov-81 at 10:52:26
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: CRA/BPSAD17 50X
Sample ID.....: 31436.16
Sample type.....: Sample
Sample amount.....: 2.0000
Number of injections.....: 1
Bottle Number.....: 1

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 20.000
Dilution.....: 50.000
Amount (g or mL).....: 2.000

PEAK INFORMATION

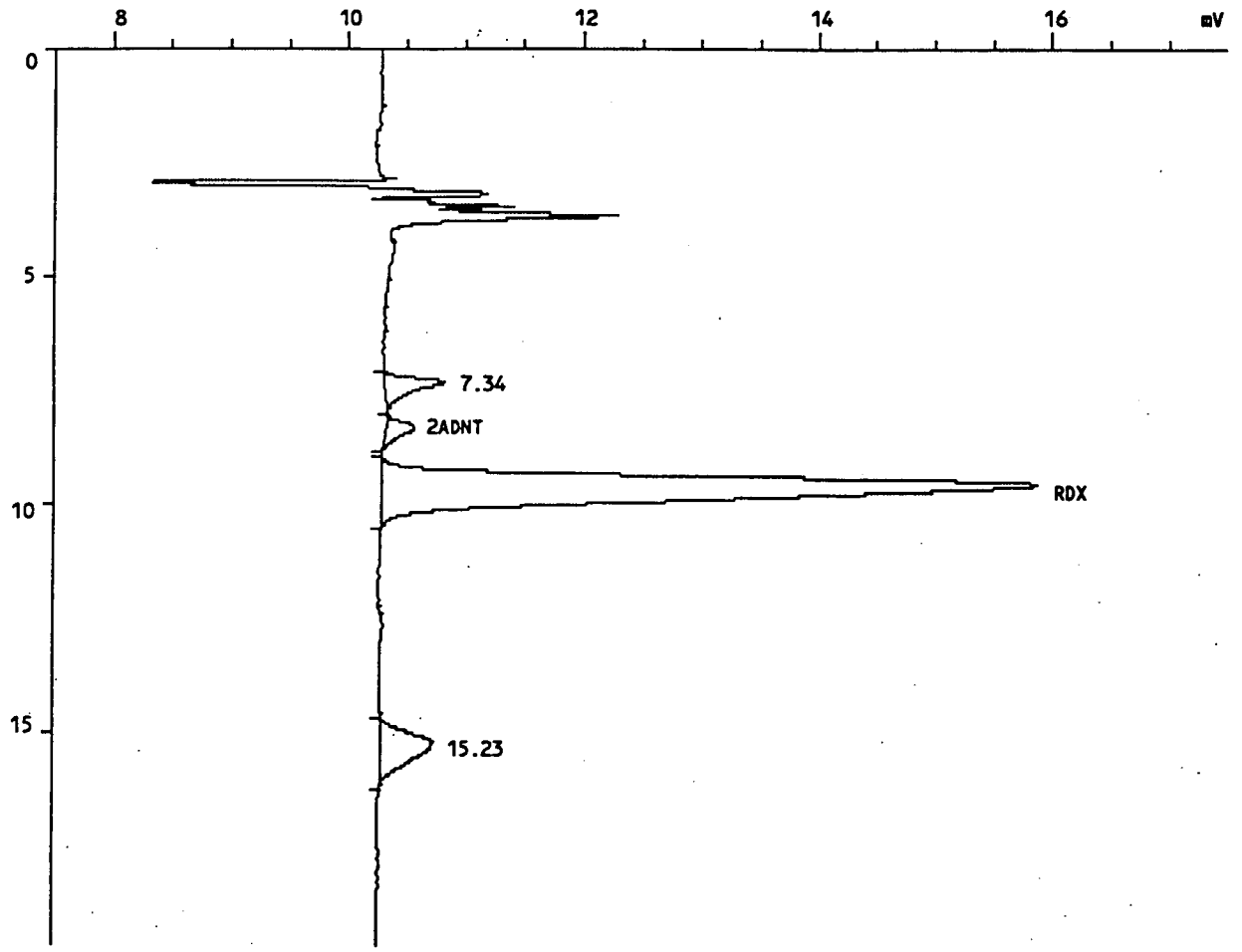
Peak	RT mins	Hght uV	Area uVs	ug/Kg	Peak name
1	7.339	512	9867	19018.879	TNT
2	8.336	248	5525	16571.732	3,4DNT
4	15.227	452	18426	83299.633	HMX
Total		1212	33818	118890.242	
Residual		5609	179238	0.000	

LONG PLOT

Injection F: <MC3> 3 3CN1027,20,1

Sample name.....: CRA/BPSAD17 50X
Sample ID.....: 31436.16
INST ID: IN10, COL. TYPE: LC-CN, ID: LC-CN-07, INJ:100, UV

Acquired on 28-Oct-97 at 09:52:21
Reported on 05-Nov-97 at 11:47:10



INJECTION REPORT

Injection F: <MC3> 3 3CN1027,20,1

Acquired on 28-Oct-97 at 09:52:21
Modified on 05-Nov-81 at 11:45:58
Reported on 05-Nov-97 at 11:45:57

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN10, COL. TYPE: LC-CN, ID: LC-CN-07, INJ:100, UV
Number of samples.: 56
Calibration file..: 3CN1027B Last modified on 05-Nov-81 at 10:42:36
Method file.....: LCCN Last modified on 05-Nov-81 at 11:44:20
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: CRA/BPSAD17 50X
Sample ID.....: 31436.16
Sample type.....: Sample
Sample amount.....: 2.0000
Number of injections.....: 1
Bottle Number.....: 1

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 20.000
Dilution.....: 50.000
Amount (g or mL).....: 2.000

PEAK INFORMATION

Peak	RT mins	Hght uV	Area uVs	ug/Kg	Peak name
2	8.336	248	5525	10649.607	2ADNT
3	9.552	5609	179238	620201.000	RDX
Total		5857	184763	630850.625	
Residual		964	28293	0.000	

1D
EXPLOSIVE ANALYSIS DATA SHEET

EPA SAMPLE NO:

CRA/BPSAD18

Lab Name: SWL-TULSA Contract:

Lab Code: SWOK Case No:MKF-OH SDG No: 31436

Matrix: (soil/water) SOIL Lab Sample ID: 31436.17

Sample Amt: 2gm % Moisture 2.13 Date Received: 10/14/97

Extraction Volume: 10ml Date Extracted: 10/14/97

Extraction Method: SONC Date Analyzed: 11/06/97

GPC Cleanup: (Y/N) N Dilution Factor: 2.00

CAS NO.	COMPOUND	CONCENTRATION UNITS: (ug/L or ug/kg) ug/kg	Q
2691-41-0	HMX	2200	U
121-82-4	RDX	1000	U
99-35-4	TNB	250	U
99-65-0	DNB	250	U
479-45-8	TETRYL	650	U
98-95-3	NB	260	U
118-96-7	TNT	250	U
1946-51-0	4ADNT	250	U
35572-78-2	2ADNT	250	U
606-20-2	26DNT	260	U
121-14-2	24DNT	250	U
88-72-2	2NT	250	U
99-99-0	4NT	250	U
99-08-1	3NT	250	U

SURROGATE AMOUNT SPIKED (ug/kg dry wt.) 20000
3,4-DNT

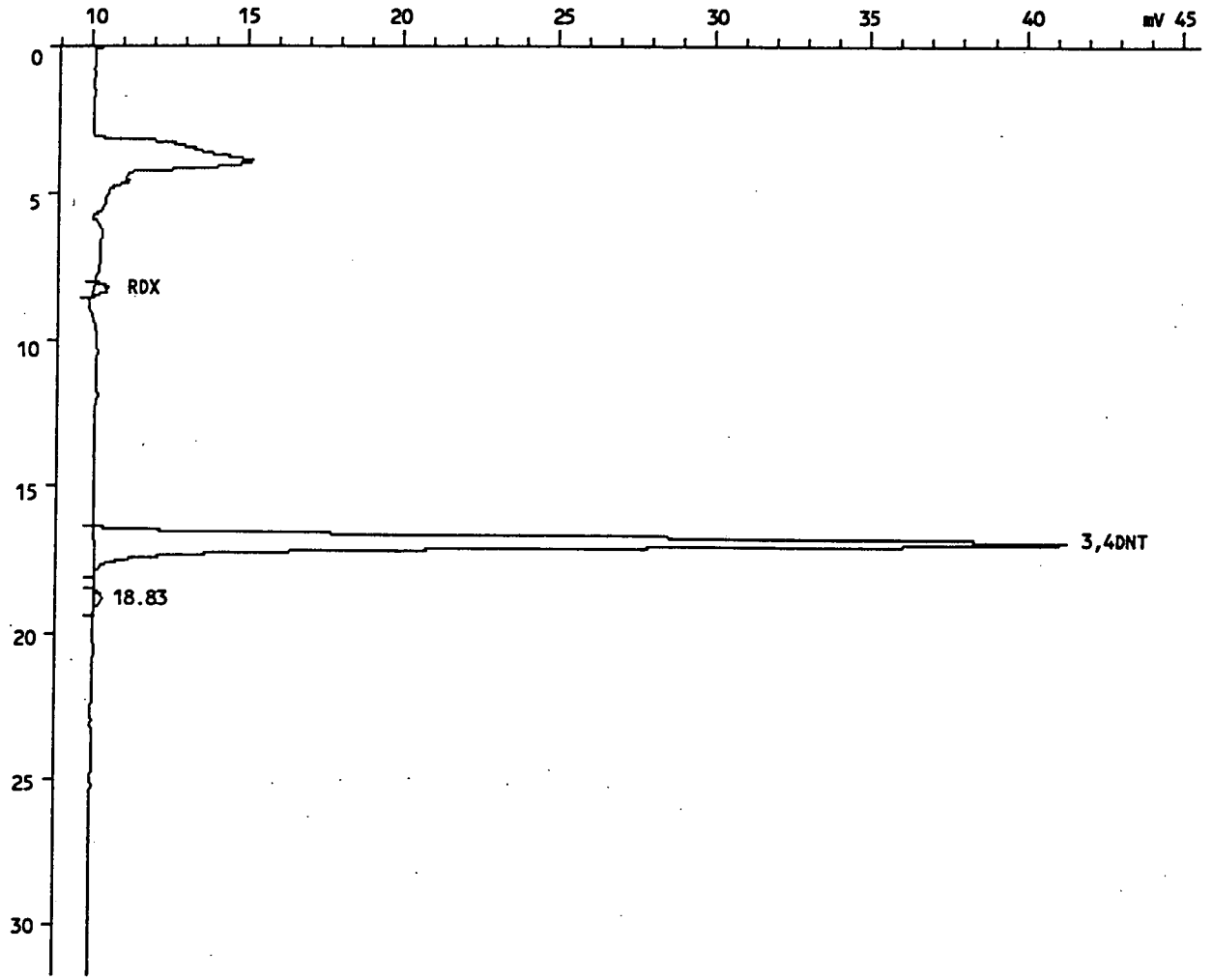
FORM I

LONG PLOT

Injection F: <MC3> 6 6EX1104B,1,1

Sample name.....: CRA/BPSAD18
Sample ID.....: 31436.17
INST ID: IN12, COL. TYPE: ODS-II, ID: CARB-04, INJ:200,COL#1

Acquired on 06-Nov-97 at 18:23:31
Reported on 25-Nov-97 at 11:15:55



INJECTION REPORT

Injection F: <MC3> 6 6EX1104B,1,1

Acquired on 06-Nov-97 at 18:23:31
Modified on 25-Nov-81 at 11:15:00
Reported on 25-Nov-97 at 11:15:26

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN12, COL. TYPE: ODS-II, ID: CARB-04, INJ:200,COL#1
Number of samples.: 40
Calibration file..: 6EX1104 Last modified on 18-Nov-81 at 14:26:56
Method file.....: EXPLOS Last modified on 25-Nov-81 at 10:24:50
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: CRA/BPSAD18
Sample ID.....: 31436.17
Sample type.....: Sample
Sample amount.....: 2.0000
Number of injections.....: 1
Bottle Number.....: 1

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 10.000
Dilution.....: 2.000
Amount (g or mL).....: 2.000

PEAK INFORMATION

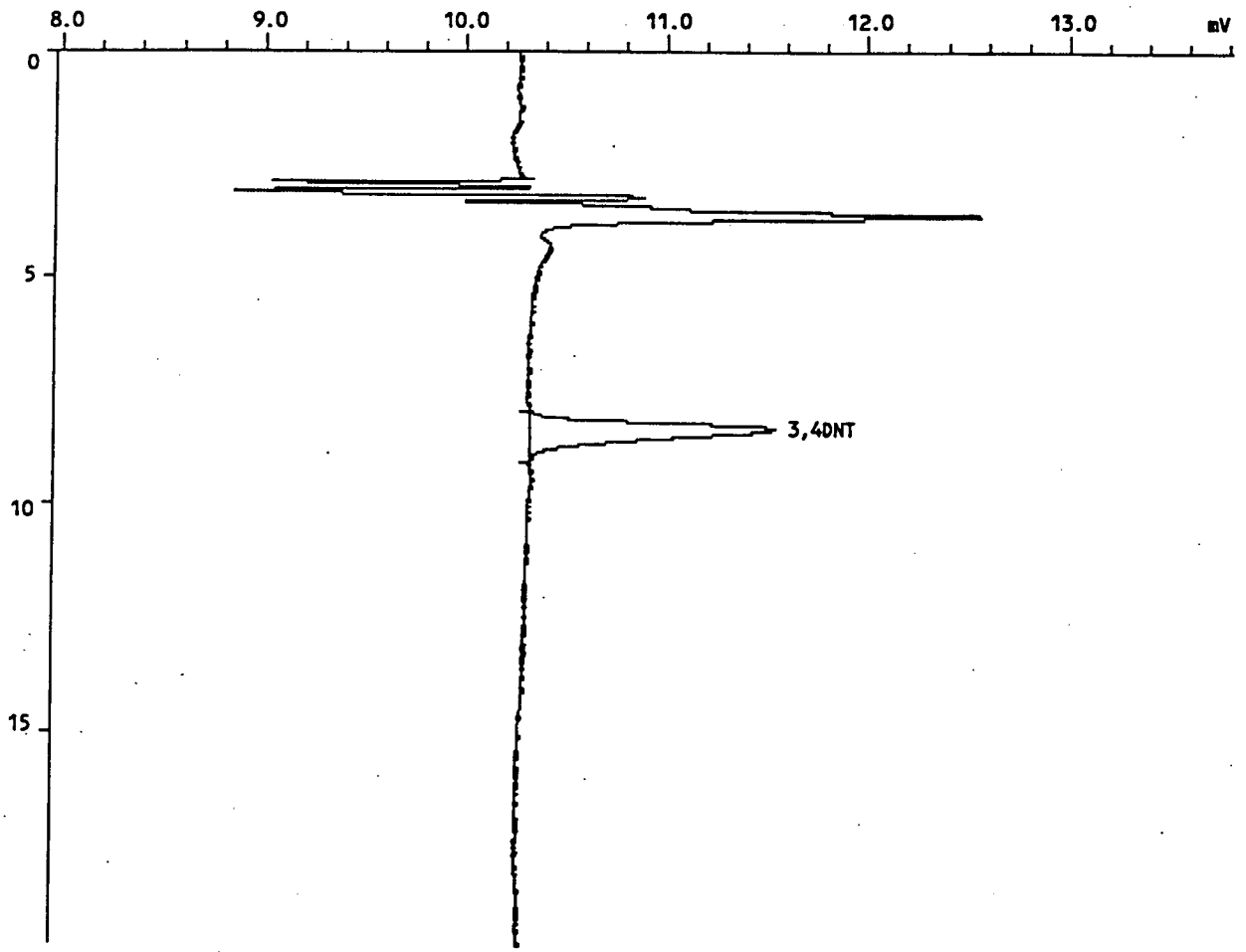
Peak	RT mins	Hght uV	Area uVs	ug/Kg	Peak name
1	8.283	488	9080	310.532	RDX
2	16.805	31254	830648	20449.246	3,4DNT 102%
Total		31742	839728	20759.779	
Residual		277	7078	174.255	

LONG PLOT

Injection F: <MC3> 3 3CN1027,21,1

Sample name.....: CRA/BPSAD18 10X
Sample ID.....: 31436.17
INST ID: IN10, COL. TYPE: LC-CN, ID: LC-CN-07, INJ:100, UV

Acquired on 28-Oct-97 at 10:33:43
Reported on 05-Nov-97 at 11:10:11



INJECTION REPORT

Injection F: <MC3> 3 3CN1027,21,1

Acquired on 28-Oct-97 at 10:33:43
Modified on 05-Nov-81 at 10:54:36
Reported on 05-Nov-97 at 10:54:37

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN10, COL. TYPE: LC-CN, ID: LC-CN-07, INJ:100, UV
Number of samples.: 56
Calibration file..: 3CN1027A Last modified on 05-Nov-81 at 10:52:08
Method file.....: LCCN Last modified on 05-Nov-81 at 10:52:26
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: CRA/BPSAD18 10X
Sample ID.....: 31436.17
Sample type.....: Sample
Sample amount.....: 2.0000
Number of injections.....: 1
Bottle Number.....: 1

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 20.000
Dilution.....: 10.000
Amount (g or mL).....: 2.000

PEAK INFORMATION

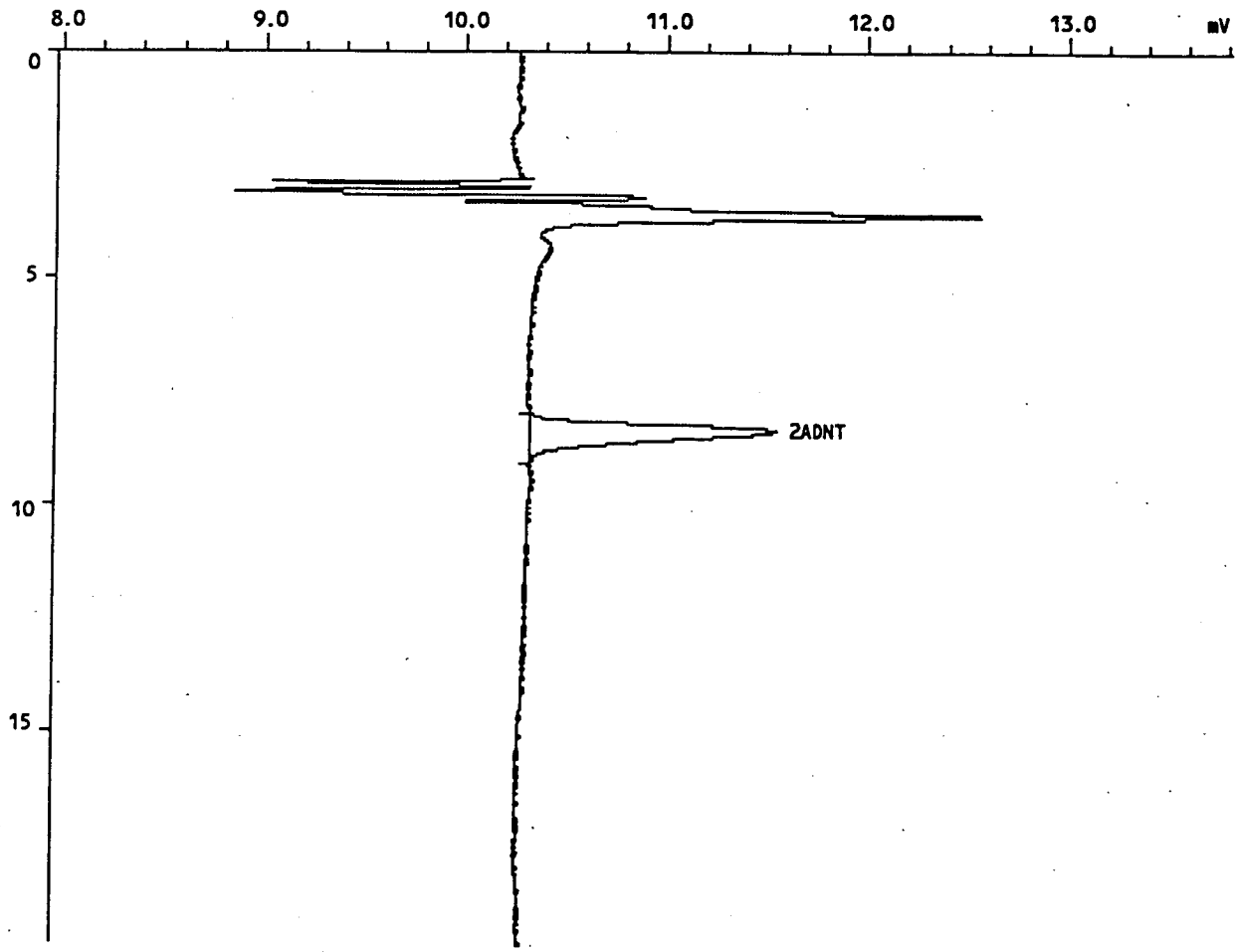
Peak	RT mins	Hght uV	Area uVs	ug/Kg	Peak name
1	8.325	1225	28971	17379.148	3,4DNT
Total		1225	28971	17379.148	
Residual		0	0	0.000	

LONG PLOT

Injection F: <MC3> 3 3CN1027,21,1

Sample name.....: CRA/BPSAD18 10X
Sample ID.....: 31436.17
INST ID: IN10, COL. TYPE: LC-CN, ID: LC-CN-07, INJ:100, UV

Acquired on 28-Oct-97 at 10:33:43
Reported on 05-Nov-97 at 11:46:48



INJECTION REPORT

Injection F: <MC3> 3 3CN1027,21,1

Acquired on 28-Oct-97 at 10:33:43
Modified on 05-Nov-81 at 11:46:10
Reported on 05-Nov-97 at 11:46:10

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN10, COL. TYPE: LC-CN, ID: LC-CN-07, INJ:100, UV
Number of samples.: 56
Calibration file..: 3CN1027B Last modified on 05-Nov-81 at 10:42:36
Method file.....: LCCN Last modified on 05-Nov-81 at 11:44:20
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: CRA/BPSAD18 10X
Sample ID.....: 31436.17
Sample type.....: Sample
Sample amount.....: 2.0000
Number of injections.....: 1
Bottle Number.....: 1

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 20.000
Dilution.....: 10.000
Amount (g or mL).....: 2.000

PEAK INFORMATION

Peak	RT mins	Hght uV	Area uVs	ug/Kg	Peak name
1	8.325	1225	28971	11168.480	2ADNT
Total		1225	28971	11168.480	
Residual		0	0	0.000	

CALIBRATION FACTOR DATA
FORM 9A-1

EXPLOSIVE CALIBRATION - METHOD SW846-8330
Instrument ID: IN13
lumn ID : CARB-05

Inj 1

Injection File Name Level 1 : 5EX1015,4
Injection File Name Level 2 : 5EX1015,5
Injection File Name Level 3 : 5EX1015,6
Injection File Name Level 4 : 5EX1015,7
Injection File Name Level 5 : 5EX1015,8

Calibration Date : 10/16/97
Number of Calibration Levels: 5
Line forced thru zero : Yes
Calculation Method : Area

COMPONENT NAME	Area Level 1	CAL FACTOR Level 1	Area Level 2	CAL FACTOR Level 2	Area Level 3	CAL FACTOR Level 3	Area Level 4	CAL FACTOR Level 4	Area Level 5	CAL FACTOR Level 5
HMX	3100	306	87098	343	241304	366	361201	365	474220	359
RDX	1407	333	44198	419	127286	461	194237	471	252957	460
TNB	2543	842	70083	927	199122	1011	295165	997	387022	982
DNB	1966	1214	49417	1217	139220	1318	208786	1318	274774	1296
TETRYL	2550	685	64321	692	184728	763	274386	754	351877	727
NB	2458	745	68441	831	192947	902	288148	895	383871	893
3,4DNT	2878	567	69295	546	196966	597	294803	594	388200	588
TNT	2858	1059	60431	894	175729	998	260352	986	340312	967
4ADNT	2023	857	36542	617	108749	706	160178	690	207134	673
2ADNT	2247	952	52477	886	154622	1004	228220	984	295953	961
26DNT	2167	559	57549	592	162166	638	242576	638	319941	632
24DNT	2561	1085	72151	1219	204932	1331	305298	1316	404206	1312
2NT	1402	415	42487	502	117654	535	177742	539	236076	537
	1224	362	35743	422	100350	456	151559	459	200573	456
	1530	475	44785	557	121618	579	183582	585	244786	586

CALIBRATION FACTOR DATA
FORM 9B

EXPLOSIVE CALIBRATION - METHOD SW846-8330
Instrument ID: IN13
Column ID : CARB-05

Inj 1

Injection File Name Level 1 : 5EX1015,4
Injection File Name Level 2 : 5EX1015,5
Injection File Name Level 3 : 5EX1015,6
Injection File Name Level 4 : 5EX1015,7
Injection File Name Level 5 : 5EX1015,8

Calibration Date : 10/16/97
Number of Calibration Levels: 5
Line forced thru zero : Yes
Calculation Method : Area

Component Name	Mean Cal Factor	% RSD	Corr. Coeff.	Slope area/amt	Intercept
HMX	348	7.24	0.99985	362	0.000
RDX	429	13.31	0.99966	463	0.000
TNB	952	7.26	0.99975	990	0.000
DNB	1273	4.17	0.99980	1304	0.000
TETRYL	724	4.87	0.99933	740	0.000
NB	853	7.84	0.99989	893	0.000
3,4DNT	578	3.72	0.99983	590	0.000
TNT	981	6.06	0.99961	976	0.000
[illegible]NT	709	12.63	0.99932	682	0.000
2[illegible]DNT	957	4.65	0.99944	973	0.000
26DNT	612	5.82	0.99988	634	0.000
24DNT	1253	8.26	0.99986	1314	0.000
2NT	505	10.45	0.99993	536	0.000
4NT	431	9.61	0.99989	456	0.000
3NT	556	8.42	0.99996	584	0.000

CALIBRATION FACTOR DATA
FORM 9C

Initial Calibration Date: 10/16/97
C tinuing Calibration #: 4
cinuing Cal Date : 10/17/97
Continuing Cal Level : 3
Instrument ID : IN13
Column ID : CARB-05
Injection File Name : 5EX1015B,6
Calculation Mode : Area

Component Name	Response Cont Std	Cont CF	Init CF Mean	%D	Init CF Slope	%D
HMX	234934	356	348	2.38	362	1.54
RDX	125286	454	429	5.84	463	1.94
TNB	192668	978	952	2.75	990	1.19
DNB	135369	1282	1273	0.73	1304	1.72
TETRYL	171719	710	724	2.03	740	4.08
NB	189332	885	853	3.73	893	0.97
3,4DNT	189686	575	578	0.64	590	2.66
TNT	164145	933	981	4.91	976	4.42
4ADNT	100248	651	709	8.15	682	4.52
2ADNT	141567	919	957	3.99	973	5.48
26DNT	157430	620	612	1.29	634	2.28
24DNT	197131	1280	1253	2.19	1314	2.60
2NT	117390	534	505	5.58	536	0.50
4NT	100090	455	431	5.51	456	0.30
T	122151	582	556	4.56	584	0.38

CALIBRATION FACTOR DATA
FORM 9C

Initial Calibration Date: 10/16/97
Continuing Calibration #: 5
Continuing Cal Date : 10/18/97
Continuing Cal Level : 3
Instrument ID : IN13
Column ID : CARB-05
Injection File Name : 5EX1015C,9
Calculation Mode : Area

Component Name	Response Cont Std	Cont CF	Init CF Mean	%D	Init CF Slope	%D
HMX	249718	378	348	8.83	362	4.66
RDX	134479	487	429	13.60	463	5.26
TNB	202583	1028	952	8.03	990	3.90
DNB	143737	1361	1273	6.95	1304	4.35
TETRYL	178575	738	724	1.89	740	0.25
NB	191055	893	853	4.67	893	0.07
3,4DNT	200170	607	578	4.86	590	2.72
TNT	174788	993	981	1.26	976	1.77
4ADNT	106475	691	709	2.44	682	1.41
2ADNT	151593	984	957	2.81	973	1.21
26DNT	166415	655	612	7.07	634	3.30
24DNT	208979	1357	1253	8.34	1314	3.26
2NT	119687	544	505	7.65	536	1.44
4NT	102562	466	431	8.12	456	2.16
[illegible]T	124268	592	556	6.37	584	1.35

CALIBRATION FACTOR DATA
FORM 9C

Initial Calibration Date: 10/16/97
Continuing Calibration #: 6
Continuing Cal Date : 10/18/97
Continuing Cal Level : 3
Instrument ID : IN13
Column ID : CARB-05
Injection File Name : 5EX1015D,2
Calculation Mode : Area

Component Name	Response Cont Std	Cont CF	Init CF Mean	%D	Init CF Slope	%D
HMX	238453	361	348	3.92	362	0.07
RDX	130545	473	429	10.28	463	2.18
TNB	195660	993	952	4.34	990	0.35
DNB	136600	1294	1273	1.64	1304	0.83
TETRYL	174591	721	724	0.39	740	2.47
NB	189903	887	853	4.04	893	0.68
3,4DNT	192679	584	578	0.93	590	1.12
TNT	166843	948	981	3.35	976	2.85
4ADNT	101555	659	709	6.95	682	3.28
2ADNT	145992	948	957	0.99	973	2.53
26DNT	159806	629	612	2.81	634	0.80
24DNT	200663	1303	1253	4.02	1314	0.85
2NT	118465	538	505	6.55	536	0.41
4NT	100932	459	431	6.40	456	0.54
[illegible]	123362	587	556	5.59	584	0.61

CALIBRATION FACTOR DATA
FORM 9C

Initial Calibration Date: 10/16/97
Continuing Calibration #: 7
Continuing Cal Date : 10/18/97
Continuing Cal Level : 3
Instrument ID : IN13
Column ID : CARB-05
Injection File Name : 5EX1015D,9
Calculation Mode : Area

Component Name	Response Cont Std	Cont CF	Init CF Mean	%D	Init CF Slope	%D
HMX	237512	360	348	3.51	362	0.46
RDX	130257	472	429	10.04	463	1.95
TNB	194101	985	952	3.51	990	0.45
DNB	136175	1290	1273	1.33	1304	1.14
TETRYL	173156	716	724	1.21	740	3.28
NB	188999	883	853	3.55	893	1.15
3,4DNT	191656	581	578	0.40	590	1.65
TNT	167039	949	981	3.23	976	2.74
4ADNT	101562	659	709	6.94	682	3.27
2ADNT	145148	943	957	1.56	973	3.09
26DNT	158920	626	612	2.24	634	1.35
24DNT	200229	1300	1253	3.80	1314	1.07
2NT	117991	536	505	6.12	536	0.01
4NT	100408	456	431	5.85	456	0.02
3NT	122581	584	556	4.93	584	0.03

CALIBRATION FACTOR DATA
FORM 9C

Initial Calibration Date: 10/16/97
C tinuing Calibration #: 8
cinuing Cal Date : 10/19/97
Continuing Cal Level : 3
Instrument ID : IN13
Column ID : CARB-05
Injection File Name : 5EX1015D,19
Calculation Mode : Area

Component Name	Response Cont Std	Cont CF	Init CF Mean	%D	Init CF Slope	%D
HMX	238270	361	348	3.84	362	0.14
RDX	130381	472	429	10.14	463	2.05
TNB	194838	989	952	3.90	990	0.07
DNB	136549	1293	1273	1.60	1304	0.87
TETRYL	173655	718	724	0.92	740	3.00
NB	188690	882	853	3.38	893	1.31
3,4DNT	192303	583	578	0.73	590	1.31
TNT	168198	956	981	2.56	976	2.06
4ADNT	101820	661	709	6.71	682	3.02
2ADNT	146552	952	957	0.61	973	2.16
26DNT	160171	631	612	3.05	634	0.58
24DNT	200944	1305	1253	4.17	1314	0.71
2NT	118782	540	505	6.83	536	0.68
4NT	100963	459	431	6.44	456	0.57
T	122349	583	556	4.73	584	0.22

CALIBRATION FACTOR DATA
FORM 9C

Initial Calibration Date: 10/16/97
Continuing Calibration #: 9
Continuing Cal Date : 10/19/97
Continuing Cal Level : 3
Instrument ID : IN13
Column ID : CARB-05
Injection File Name : 5EX1015E,1
Calculation Mode : Area

Component Name	Response Cont Std	Cont CF	Init CF Mean	%D	Init CF Slope	%D
HMX	239757	363	348	4.49	362	0.48
RDX	124426	451	429	5.11	463	2.61
TNB	197895	1005	952	5.53	990	1.49
DNB	140500	1330	1273	4.54	1304	2.00
TETRYL	181010	748	724	3.28	740	1.11
NB	196448	918	853	7.63	893	2.75
3,4DNT	199677	605	578	4.60	590	2.47
TNT	175351	996	981	1.58	976	2.10
4ADNT	104792	680	709	3.99	682	0.19
2ADNT	151897	986	957	3.02	973	1.41
26DNT	160277	631	612	3.12	634	0.51
24DNT	208082	1351	1253	7.87	1314	2.81
2NT	119882	545	505	7.82	536	1.61
4NT	102364	465	431	7.91	456	1.97
T	124072	591	556	6.20	584	1.19

CALIBRATION FACTOR DATA
FORM 9C

Initial Calibration Date: 10/16/97
Continuing Calibration #: 10
Continuing Cal Date : 10/20/97
Continuing Cal Level : 3
Instrument ID : IN13
Column ID : CARB-05
Injection File Name : 5EX1015E,9
Calculation Mode : Area

Component Name	Response Cont Std	Cont CF	Init CF Mean	%D	Init CF Slope	%D
HMX	243625	369	348	6.17	362	2.10
RDX	133004	482	429	12.36	463	4.10
TNB	198019	1005	952	5.60	990	1.56
DNB	139579	1322	1273	3.86	1304	1.33
TETRYL	174529	721	724	0.42	740	2.51
NB	190764	891	853	4.51	893	0.22
3,4DNT	196974	597	578	3.18	590	1.08
TNT	170886	971	981	1.00	976	0.50
4ADNT	104117	676	709	4.60	682	0.83
2ADNT	148644	965	957	0.81	973	0.76
26DNT	162755	641	612	4.71	634	1.03
24DNT	205072	1332	1253	6.31	1314	1.32
2NT	120273	547	505	8.17	536	1.94
4NT	102788	467	431	8.36	456	2.39
[illegible]T	123652	589	556	5.84	584	0.84

CALIBRATION FACTOR DATA
FORM 9D

EXPLOSIVE CALIBRATION - METHOD SW846-8330

jection volume = 200uL

Amount Units = ng

COMPONENT NAME	AMT ON COL Level 1	AMT ON COL Level 2	AMT ON COL Level 3	AMT ON COL Level 4	AMT ON COL Level 5
HMX	10.140	254.000	660.000	990.000	1320.000
RDX	4.220	105.600	276.000	412.000	550.000
TNB	3.020	75.600	197.000	296.000	394.000
DNB	1.620	40.600	105.600	158.400	212.000
TETRYL	3.720	93.000	242.000	364.000	484.000
NB	3.300	82.400	214.000	322.000	430.000
3,4DNT	5.080	126.800	330.000	496.000	660.000
TNT	2.700	67.600	176.000	264.000	352.000
4ADNT	2.360	59.200	154.000	232.000	308.000
2ADNT	2.360	59.200	154.000	232.000	308.000
26DNT	3.880	97.200	254.000	380.000	506.000
24DNT	2.360	59.200	154.000	232.000	308.000
2NT	3.380	84.600	220.000	330.000	440.000
4NT	3.380	84.600	220.000	330.000	440.000
3NT	3.220	80.400	210.000	314.000	418.000

CALIBRATION FACTOR DATA
FORM 9E

EXPLOSIVE CALIBRATION - METHOD SW846-8330

ɜtrument ID : IN13
Column ID : CARB-05
Calibration Date : 10/16/97
Number of Calibration Levels: 5

COMPONENT NAME	RT Level 1	RT Level 2	RT Level 3	RT Level 4	RT Level 5
HMX	4.85	4.85	4.85	4.84	4.84
RDX	7.38	7.37	7.37	7.37	7.36
TNB	10.01	10.01	10.01	10.00	10.00
DNB	12.27	12.27	12.28	12.27	12.27
TETRYL	13.04	13.04	13.05	13.04	13.04
NB	13.96	13.96	13.97	13.96	13.96
3,4DNT	15.16	15.14	15.16	15.15	15.15
TNT	15.80	15.78	15.80	15.79	15.79
4ADNT	16.49	16.49	16.51	16.50	16.50
2ADNT	17.38	17.37	17.40	17.38	17.38
26DNT	18.51	18.48	18.52	18.50	18.50
24DNT	19.17	19.14	19.18	19.16	19.16
2NT	22.53	22.50	22.57	22.55	22.54
2NT					
4NT	24.12	24.06	24.14	24.11	24.11
3NT	26.03	25.94	26.01	25.99	25.98

COMPONENT NAME	RT Cont CA1	RT Cont CA2	RT Cont CA3	RT Cont CA4	RT Cont CA5
HMX	4.84	4.84	4.85	4.86	4.84
RDX	7.36	7.36	7.38	7.39	7.38
TNB	9.99	9.99	10.01	10.02	10.02
DNB	12.25	12.26	12.28	12.29	12.29
TETRYL	13.00	13.04	13.07	13.11	13.08
NB	13.94	13.94	13.95	13.95	13.98
3,4DNT	15.11	15.13	15.16	15.17	15.18
TNT	15.76	15.77	15.80	15.82	15.82
4ADNT	16.43	16.49	16.52	16.56	16.56
2ADNT	17.30	17.39	17.42	17.47	17.45
26DNT	18.44	18.48	18.51	18.53	18.54
24DNT	19.10	19.14	19.17	19.20	19.21
2NT	22.48	22.49	22.55	22.53	22.58
4NT	24.03	24.04	24.11	24.08	24.14
3NT	25.89	25.92	25.99	25.96	26.02

COMPONENT NAME	AVE RT	STD_DEV	3X SD	AVE+-3X SD
HMX	4.84	0.016	0.048	4.79 - 4.89
X	7.36	0.028	0.083	7.28 - 7.44
NB	10.00	0.025	0.076	9.92 - 10.07
DNB	12.26	0.028	0.083	12.18 - 12.35
TETRYL	13.03	0.051	0.152	12.88 - 13.18
NB	13.95	0.022	0.067	13.89 - 14.02
3,4DNT	15.14	0.033	0.099	15.04 - 15.24

TNT	15.78	0.035	0.104	15.68 - 15.88
4ADNT	16.48	0.053	0.160	16.32 - 16.64
2ADNT	17.37	0.062	0.187	17.18 - 17.56
26DNT	18.49	0.040	0.119	18.37 - 18.61
24DNT	19.15	0.042	0.125	19.03 - 19.28
2NT	22.53	0.037	0.111	22.42 - 22.64
[illegible]	24.09	0.040	0.121	23.97 - 24.21
[illegible]NT	25.97	0.046	0.139	25.83 - 26.11

CALIBRATION FACTOR DAT
FORM 9E

EXPLOSIVE CALIBRATION - METHOD SW846-8330

Instrument ID : IN13
Column ID : CARB-05
Calibration Date : 10/16/97
Number of Calibration Levels: 5

COMPONENT NAME	RT Level 1	RT Level 2	RT Level 3	RT Level 4	RT Level 5
HMX	4.85	4.85	4.85	4.84	4.84
RDX	7.38	7.37	7.37	7.37	7.36
TNB	10.01	10.01	10.01	10.00	10.00
DNB	12.27	12.27	12.28	12.27	12.27
TETRYL	13.04	13.04	13.05	13.04	13.04
NB	13.96	13.96	13.97	13.96	13.96
3,4DNT	15.16	15.14	15.16	15.15	15.15
TNT	15.80	15.78	15.80	15.79	15.79
4ADNT	16.49	16.49	16.51	16.50	16.50
2ADNT	17.38	17.37	17.40	17.38	17.38
26DNT	18.51	18.48	18.52	18.50	18.50
24DNT	19.17	19.14	19.18	19.16	19.16
2NT	22.53	22.50	22.57	22.55	22.54
2NT					
4NT	24.12	24.06	24.14	24.11	24.11
3NT	26.03	25.94	26.01	25.99	25.98

COMPONENT NAME	RT Cont CA6	RT Cont CA7	RT Cont CA8	RT Cont CA9	RT Cont CA10
HMX	4.82	4.86	4.83	4.80	4.84
RDX	7.33	7.38	7.34	7.27	7.36
TNB	9.97	10.02	9.98	9.92	10.01
DNB	12.23	12.29	12.25	12.19	12.28
TETRYL	12.98	13.05	12.98	12.90	13.04
NB	13.93	13.98	13.95	13.90	13.98
3,4DNT	15.10	15.17	15.13	15.06	15.17
TNT	15.74	15.80	15.76	15.69	15.80
4ADNT	16.42	16.50	16.44	16.36	16.51
2ADNT	17.29	17.38	17.31	17.23	17.39
26DNT	18.44	18.51	18.46	18.40	18.52
24DNT	19.09	19.17	19.12	19.06	19.18
2NT	22.49	22.56	22.53	22.48	22.59
4NT	24.06	24.12	24.10	24.03	24.16
3NT	25.93	25.99	25.97	25.90	26.03

COMPONENT NAME	AVE RT	STD_DEV	3X SD	AVE+-3X SD
HMX	4.84	0.016	0.048	4.79 - 4.89
RDX	7.36	0.028	0.083	7.28 - 7.44
TNB	10.00	0.025	0.076	9.92 - 10.07
DNB	12.26	0.028	0.083	12.18 - 12.35
TETRYL	13.03	0.051	0.152	12.88 - 13.18
NB	13.95	0.022	0.067	13.89 - 14.02
3,4DNT	15.14	0.033	0.099	15.04 - 15.24
TNT	15.78	0.035	0.104	15.68 - 15.88

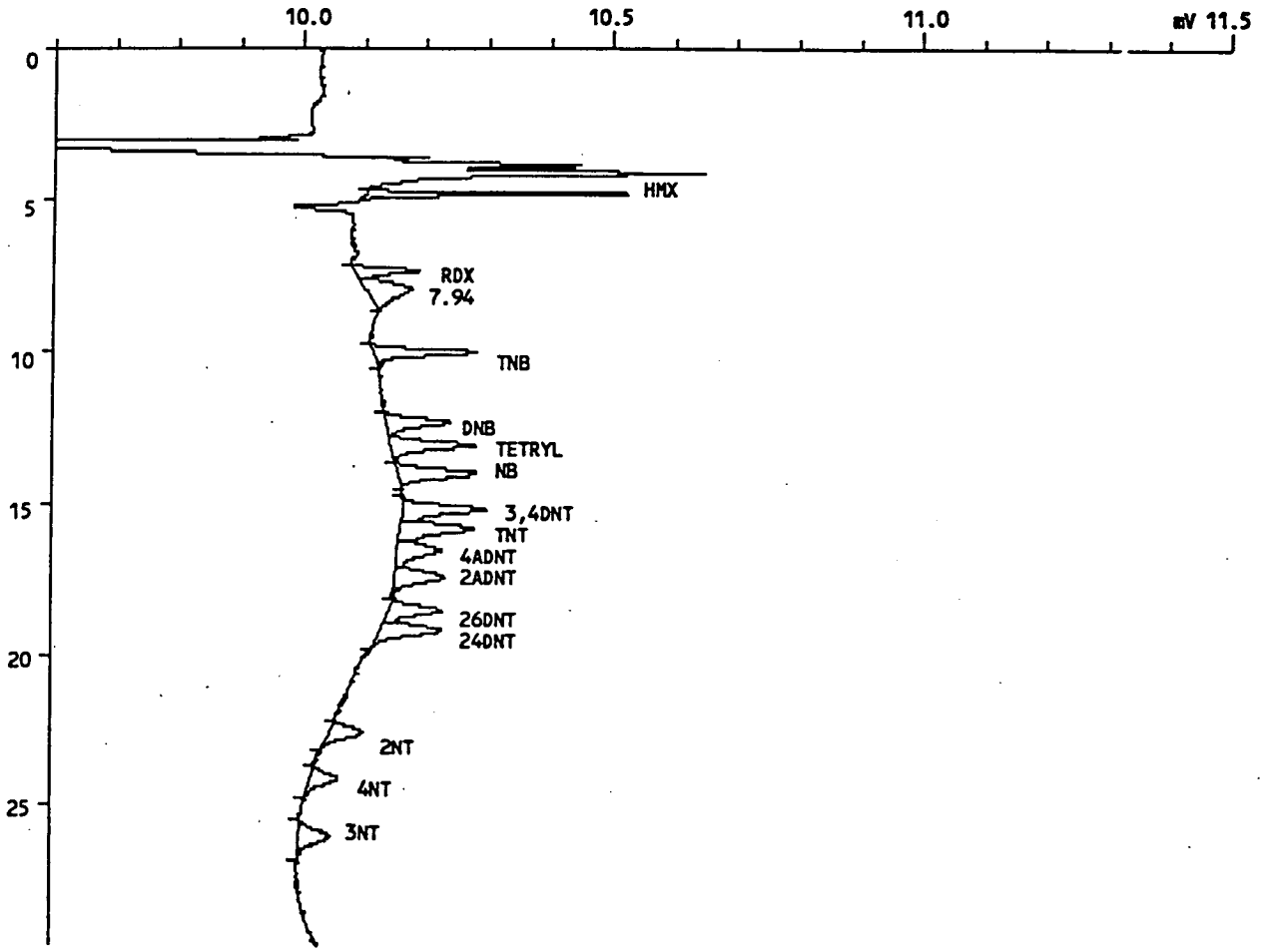
4ADNT	16.48	0.053	0.160	16.32 - 16.64
2ADNT	17.37	0.062	0.187	17.18 - 17.56
26DNT	18.49	0.040	0.119	18.37 - 18.61
24DNT	19.15	0.042	0.125	19.03 - 19.28
2NT	22.53	0.037	0.111	22.42 - 22.64
4NT	24.09	0.040	0.121	23.97 - 24.21
[illegible]	25.97	0.046	0.139	25.83 - 26.11

LONG PLOT

Injection F: <MC3> 5 5EX1015,4,1

Sample name.....: EXP L1 SOIL STD
Sample ID.....: 1-90:IA1
INST ID: IN13, COL. TYPE: ODS-II, ID: CARB-05, INJ:200,COL#1

Acquired on 16-Oct-97 at 02:01:39
Reported on 19-Nov-97 at 09:56:48



INJECTION REPORT

Injection F: <MC3> 5 5EX1015,4,1

Acquired on 16-Oct-97 at 02:01:39
Modified on 23-Oct-81 at 13:38:40
Reported on 23-Oct-97 at 13:40:29

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN13, COL. TYPE: ODS-II, ID: CARB-05, INJ:200,COL#1
Number of samples.: 31
Calibration file..: 5EX1015 Last modified on 23-Oct-81 at 13:39:04
Method file.....: EXPLOS Last modified on 23-Oct-81 at 13:40:14
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: EXP L1 SOIL STD
Sample ID.....: 1-90:IA1
Sample type.....: Standard
Sample amount.....: 1.0000
Number of injections.....: 1
Bottle Number.....: 1

Retention time update.....: No update
Calibration level identifier.....: 1
Response factor update.....: Initialise

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 1.000
Dilution.....: 1.000
Amount (g or mL).....: 1.000

PEAK INFORMATION

Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
1	4.848	423	3100	0.043	HMX
2	7.376	104	1407	0.015	RDX
4	10.005	169	2543	0.013	TNB
5	12.267	102	1966	7.704E-003	DNB
6	13.035	135	2550	0.017	TETRYL
7	13.957	126	2458	0.014	NB
8	15.157	138	2878	0.025	3,4DNT
9	15.797	120	2858	0.015	TNT
10	16.491	70	2023	0.018	4ADNT
11	17.381	82	2247	0.012	2ADNT

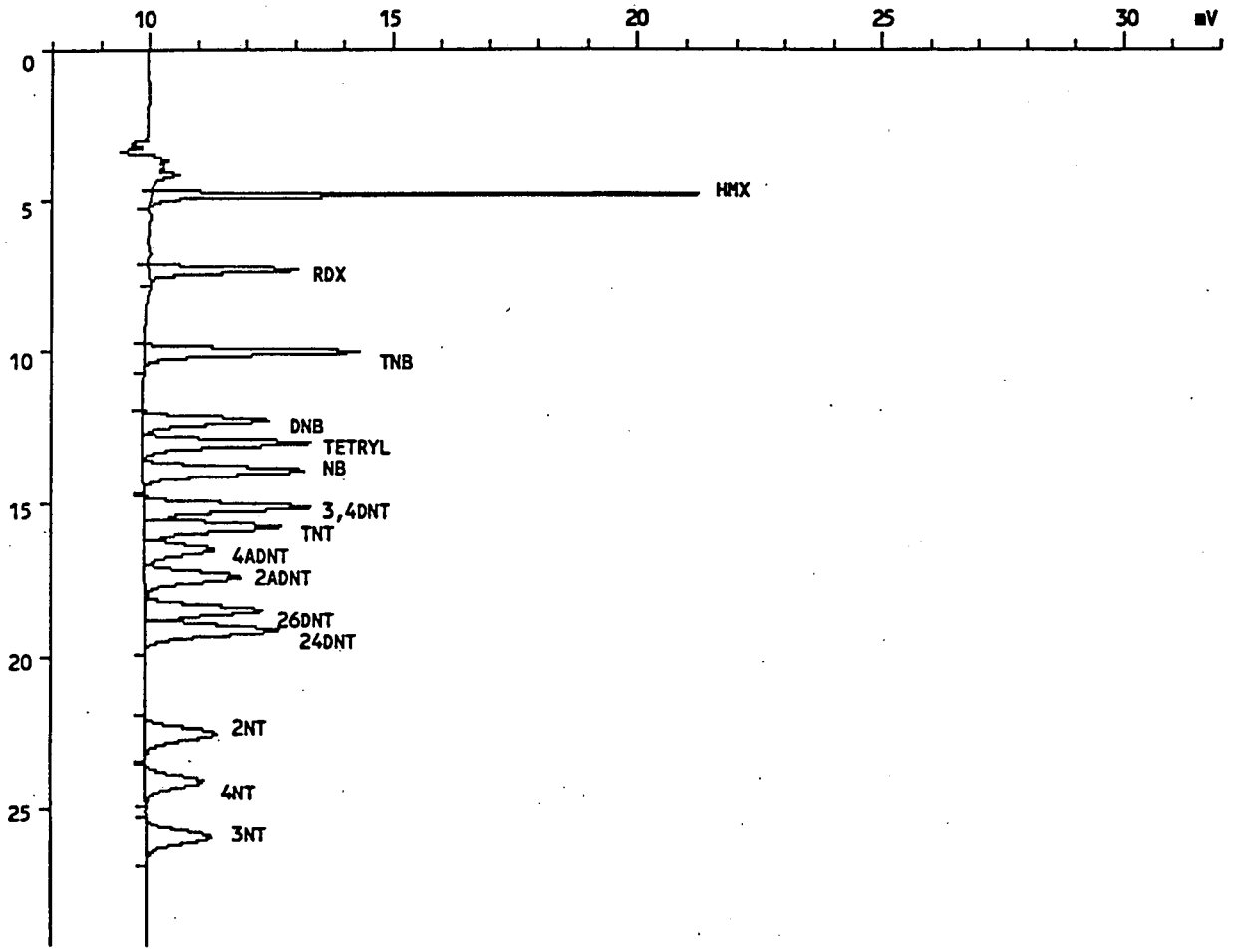
Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
12	18.512	91	2167	0.017	26DNT
13	19.173	103	2561	9.964E-003	24DNT
14	22.533	54	1402	0.013	2NT
15	24.117	46	1224	0.014	4NT
16	26.032	49	1530	0.013	3NT
Total		1811	32915	0.248	
Residual		74	2429	0.026	

LONG PLOT

Injection F: <MC3> 5 5EX1015,5,1

Sample name.....: EXP L2 SOIL STD
Sample ID.....: 1-89:IA2
INST ID: IN13, COL. TYPE: ODS-II, ID: CARB-05, INJ:200,COL#1

Acquired on 16-Oct-97 at 02:46:21
Reported on 19-Nov-97 at 09:57:29



INJECTION REPORT

Injection F: <MC3> 5 5EX1015,5,1

Acquired on 16-Oct-97 at 02:46:21
Modified on 23-Oct-81 at 13:38:46
Reported on 23-Oct-97 at 13:40:51

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN13, COL. TYPE: ODS-II, ID: CARB-05, INJ:200,COL#1
Number of samples.: 31
Calibration file..: 5EX1015 Last modified on 23-Oct-81 at 13:39:04
Method file.....: EXPLOS Last modified on 23-Oct-81 at 13:40:14
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: EXP L2 SOIL STD
Sample ID.....: 1-89:IA2
Sample type.....: Standard
Sample amount.....: 1.0000
Number of injections.....: 1
Bottle Number.....: 1

Retention time update.....: No update
Calibration level identifier.....: 2
Response factor update.....: Initialise

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 1.000
Dilution.....: 1.000
Amount (g or mL).....: 1.000

PEAK INFORMATION

Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
1	4.848	11193	87098	1.218	HMX
2	7.371	3049	44198	0.480	RDX
3	10.011	4445	70083	0.359	TNB
4	12.272	2603	49417	0.194	DNB
5	13.035	3416	64321	0.435	TETRYL
6	13.957	3289	68441	0.390	NB
7	15.141	3425	69295	0.601	3,4DNT
8	15.776	2796	60431	0.320	TNT
9	16.485	1444	36542	0.319	4ADNT
10	17.365	1984	52477	0.277	2ADNT

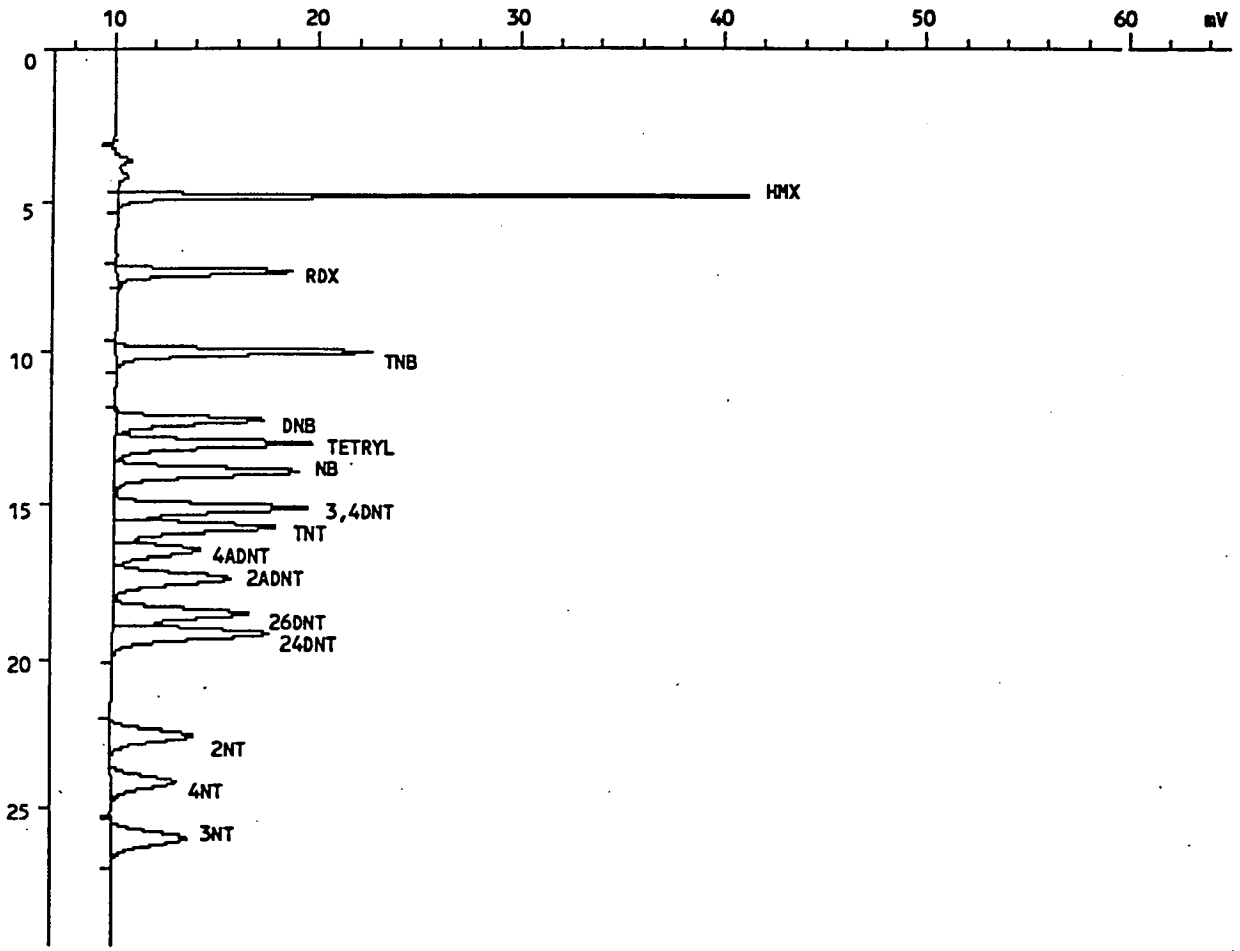
Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
11	18.480	2379	57549	0.462	26DNT
12	19.141	2750	72151	0.281	24DNT
13	22.501	1462	42487	0.400	2NT
14	24.064	1182	35743	0.395	4NT
15	25.936	1363	44785	0.386	3NT
Total		46781	855019	6.516	
Residual		0	0	0.000	

LONG PLOT

Injection F: <MC3> 5 5EX1015,6,1

Sample name.....: EXP L1 SOIL STD
Sample ID.....: 1-88:IA3
INST ID: IN13, COL. TYPE: ODS-II, ID: CARB-05, INJ:200,COL#1

Acquired on 16-Oct-97 at 03:31:02
Reported on 19-Nov-97 at 09:58:11



INJECTION REPORT

Injection F: <MC3> 5 5EX1015,6,1

Acquired on 16-Oct-97 at 03:31:02
Modified on 23-Oct-81 at 13:38:50
Reported on 23-Oct-97 at 13:41:15

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN13, COL. TYPE: ODS-II, ID: CARB-05, INJ:200,COL#1
Number of samples.: 31
Calibration file..: 5EX1015 Last modified on 23-Oct-81 at 13:39:04
Method file.....: EXPLOS Last modified on 23-Oct-81 at 13:40:14
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: EXP L1 SOIL STD
Sample ID.....: 1-88:IA3
Sample type.....: Standard
Sample amount.....: 1.0000
Number of injections.....: 1
Bottle Number.....: 1

Retention time update.....: No update
Calibration level identifier.....: 3
Response factor update.....: Initialise

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 1.000
Dilution.....: 1.000
Amount (g or mL).....: 1.000

PEAK INFORMATION

Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
1	4.848	30987	241304	3.358	HMX
2	7.371	8642	127286	1.372	RDX
3	10.011	12531	199122	1.011	TNB
4	12.277	7273	139220	0.542	DNB
5	13.045	9610	184728	1.241	TETRYL
6	13.968	8983	192947	1.092	NB
7	15.163	9497	196966	1.697	3,4DNT
8	15.797	7944	175729	0.921	TNT
9	16.507	4211	108749	0.938	4ADNT
10	17.397	5731	154622	0.809	2ADNT

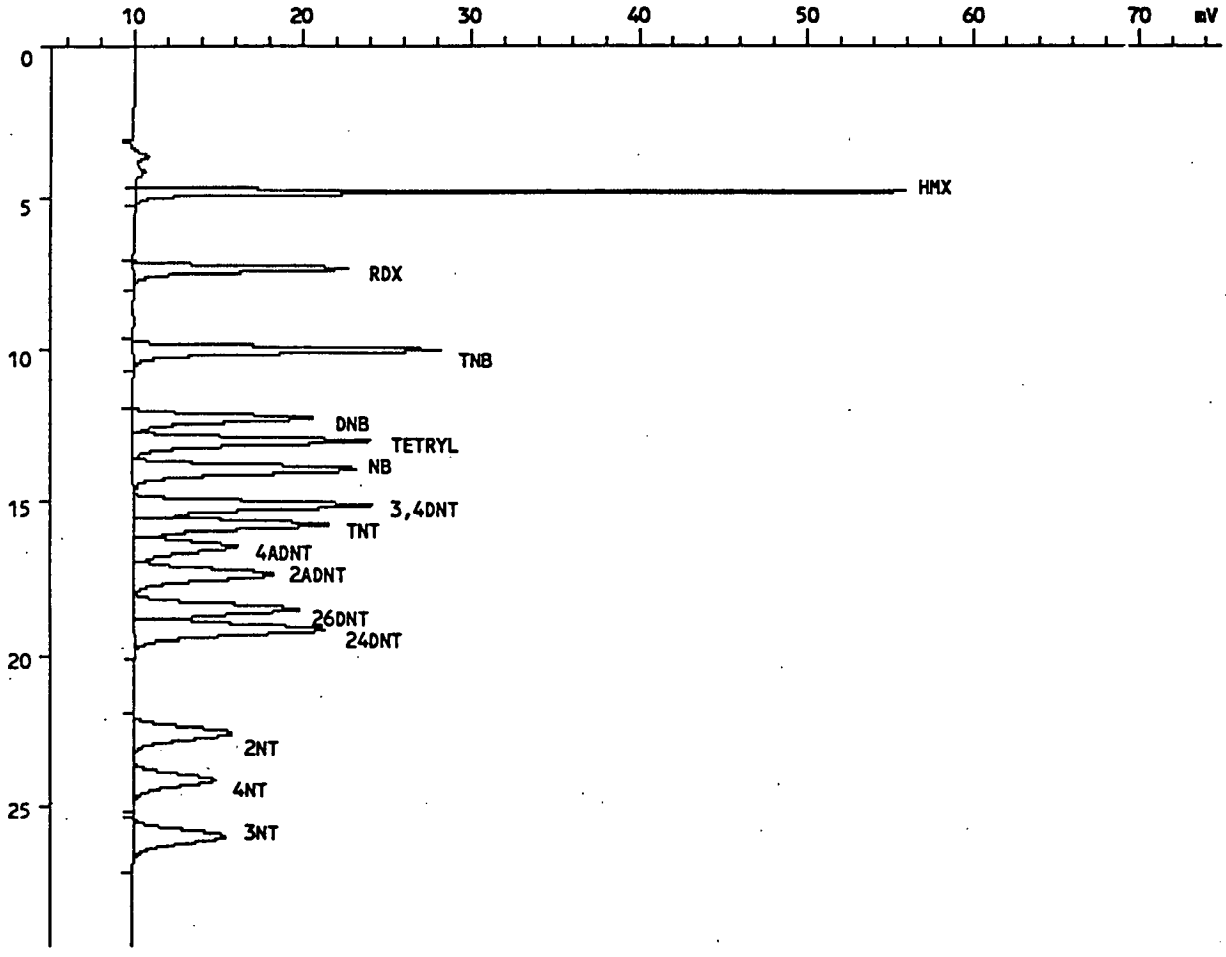
Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
11	18.517	6605	162166	1.294	26DNT
12	19.179	7649	204932	0.792	24DNT
13	22.571	3991	117654	1.104	2NT
14	24.139	3258	100350	1.106	4NT
15	26.011	3717	121618	1.047	3NT
Total		130629	2427393	18.326	
Residual		0	0	0.000	

LONG PLOT

Injection F: <MC3> 5 5EX1015,7,1

Sample name.....: EXP L4 SOIL STD
Sample ID.....: 1-87:IA4
INST ID: IN13, COL. TYPE: ODS-II, ID: CARB-05, INJ:200,COL#1

Acquired on 16-Oct-97 at 04:15:45
Reported on 23-Oct-97 at 13:44:55



INJECTION REPORT

Injection F: <MC3> 5 5EX1015,7,1

Acquired on 16-Oct-97 at 04:15:45
Modified on 23-Oct-81 at 13:38:58
Reported on 23-Oct-97 at 13:41:39

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN13, COL. TYPE: ODS-II, ID: CARB-05, INJ:200,COL#1
Number of samples.: 31
Calibration file..: 5EX1015 Last modified on 23-Oct-81 at 13:39:04
Method file.....: EXPLOS Last modified on 23-Oct-81 at 13:40:14
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: EXP L4 SOIL STD
Sample ID.....: 1-87:IA4
Sample type.....: Standard
Sample amount.....: 1.0000
Number of injections.....: 1
Bottle Number.....: 1

Retention time update.....: No update
Calibration level identifier.....: 4
Response factor update.....: Initialise

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 1.000
Dilution.....: 1.000
Amount (g or mL).....: 1.000

PEAK INFORMATION

Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
1	4.837	45910	361201	4.979	HMX
2	7.365	12750	194237	2.084	RDX
3	10.000	18361	295165	1.487	TNB
4	12.272	10741	208786	0.804	DNB
5	13.040	14130	274386	1.828	TETRYL
6	13.963	13324	288148	1.614	NB
7	15.152	14141	294803	2.503	3,4DNT
8	15.787	11692	260352	1.341	TNT
9	16.496	6165	160178	1.347	4ADNT
10	17.381	8416	228220	1.173	2ADNT

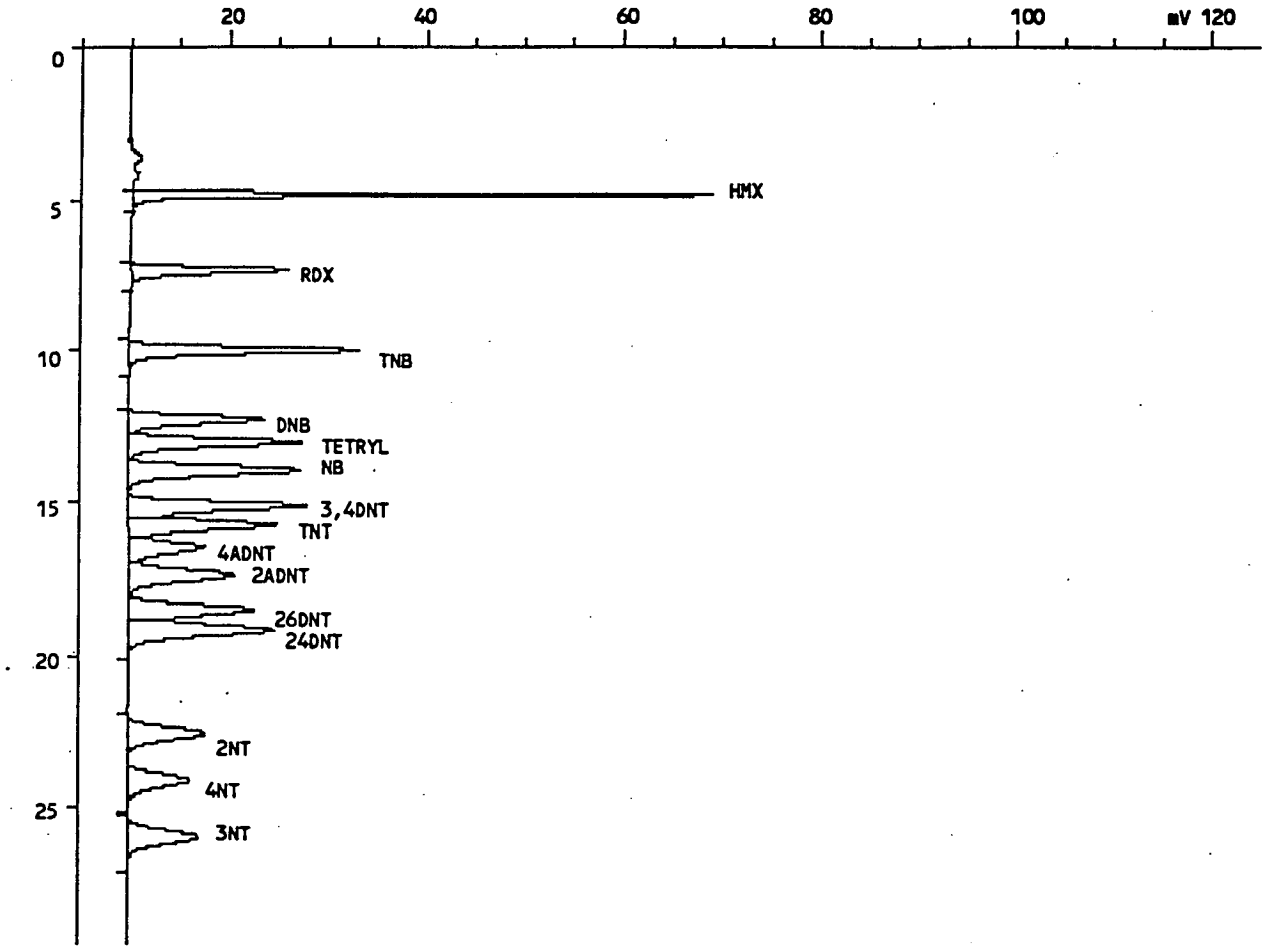
Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
11	18.501	9827	242576	1.918	26DNT
12	19.163	11359	305298	1.168	24DNT
13	22.549	5968	177742	1.660	2NT
14	24.112	4867	151559	1.663	4NT
15	25.989	5541	183582	1.575	3NT
Total		193190	3626234	27.146	
Residual		0	0	0.000	

LONG PLOT

Injection F: <MC3> 5 5EX1015,8,1

Sample name.....: EXP L5 SOIL STD
Sample ID.....: 1-86:IA5
INST ID: IN13, COL. TYPE: ODS-II, ID: CARB-05, INJ:200,COL#1

Acquired on 16-Oct-97 at 05:00:27
Reported on 23-Oct-97 at 13:43:01



INJECTION REPORT

Injection F: <MC3> 5 5EX1015,8,1

Acquired on 16-Oct-97 at 05:00:27
Modified on 23-Oct-81 at 13:39:06
Reported on 23-Oct-97 at 13:42:02

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN13, COL. TYPE: ODS-II, ID: CARB-05, INJ:200,COL#1
Number of samples.: 31
Calibration file..: 5EX1015 Last modified on 23-Oct-81 at 13:39:04
Method file.....: EXPLOS Last modified on 23-Oct-81 at 13:40:14
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: EXP L5 SOIL STD
Sample ID.....: 1-86:IA5
Sample type.....: Standard
Sample amount.....: 1.0000
Number of injections.....: 1
Bottle Number.....: 1

Retention time update.....: No update
Calibration level identifier.....: 5
Response factor update.....: Initialise

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 1.000
Dilution.....: 1.000
Amount (g or mL).....: 1.000

PEAK INFORMATION

Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
1	4.837	58758	474220	6.559	HMX
2	7.360	15916	252957	2.733	RDX
3	10.000	23194	387022	1.956	TNB
4	12.272	13731	274774	1.053	DNB
5	13.035	17620	351877	2.380	TETRYL
6	13.963	17277	383871	2.149	NB
7	15.152	18179	388200	3.288	3,4DNT
8	15.787	14932	340312	1.744	TNT
9	16.496	7765	207134	1.735	4ADNT
10	17.376	10631	295953	1.522	2ADNT

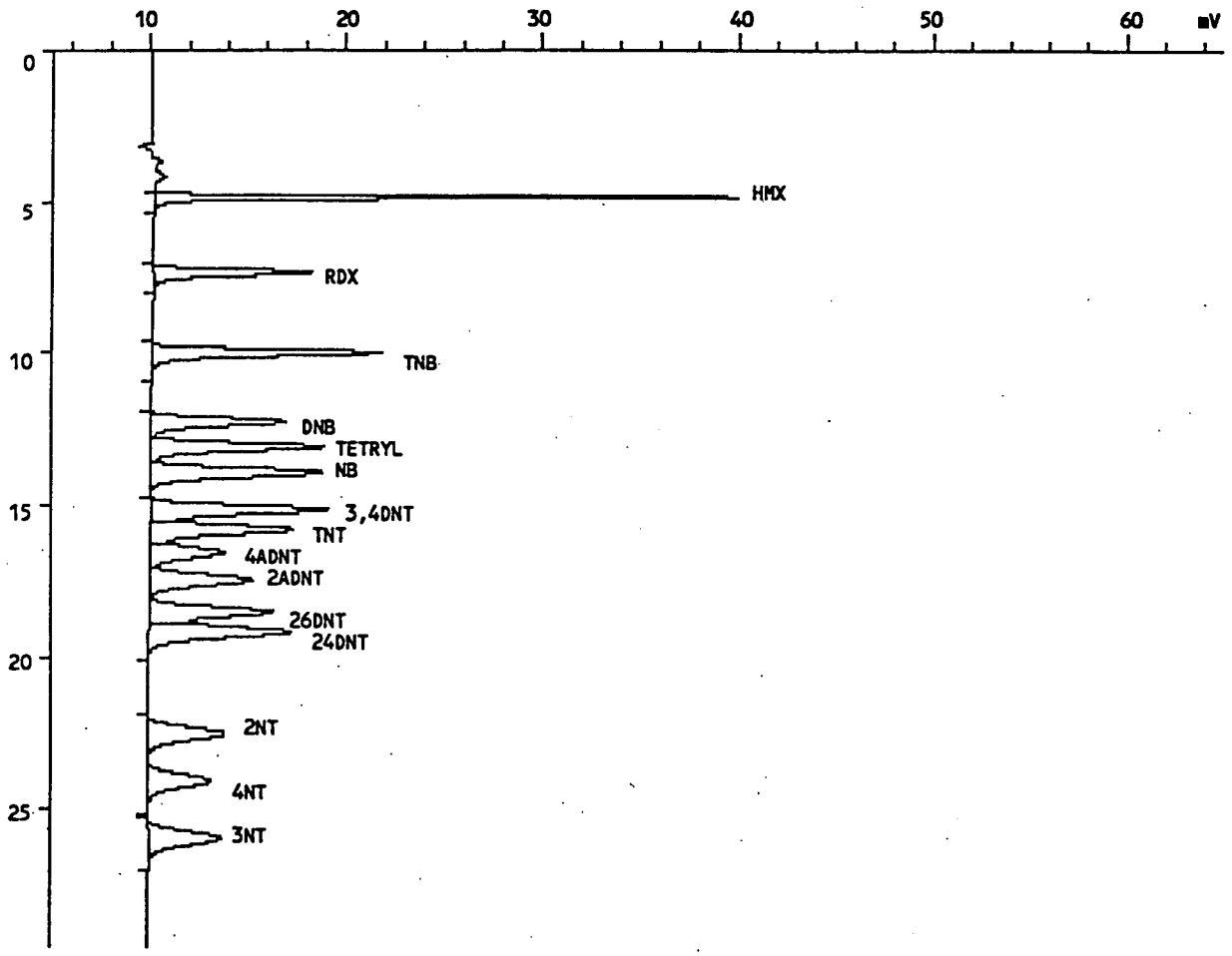
Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
11	18.496	12685	319941	2.523	26DNT
12	19.157	14672	404206	1.538	24DNT
13	22.544	7804	236076	2.201	2NT
14	24.107	6361	200573	2.198	4NT
15	25.979	7298	244786	2.096	3NT
Total		246822	4761903	35.675	
Residual		0	0	0.000	

LONG PLOT

Injection F: <MC3> 5 5EX1015B,6,1

Sample name.....: EXP L3 SOIL STD
Sample ID.....: 1-111:CA4
INST ID: IN13, COL. TYPE: ODS-II, ID: CARB-05, INJ:200,COL#1

Acquired on 17-Oct-97 at 19:26:19
Reported on 19-Nov-97 at 09:49:04



INJECTION REPORT

Injection F: <MC3> 5 5EX1015B,6,1

Acquired on 17-Oct-97 at 19:26:19
Modified on 18-Oct-81 at 12:20:50
Reported on 19-Nov-97 at 09:48:36

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN13, COL. TYPE: ODS-II, ID: CARB-05, INJ:200,COL#1
Number of samples.: 7
Calibration file..: 5EX1015 Last modified on 17-Nov-81 at 21:07:32
Method file.....: EXPLOS Last modified on 19-Nov-81 at 09:45:48
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: EXP L3 SOIL STD
Sample ID.....: 1-111:CA4
Sample type.....: Standard
Sample amount.....: 1.0000
Number of injections.....: 1
Bottle Number.....: 1

Retention time update.....: No update
Calibration level identifier.....: 3
Response factor update.....: No update

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 1.000
Dilution.....: 1.000
Amount (g or mL).....: 1000.000

PEAK INFORMATION

Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
1	4.859	29826	234934	3.415	HMX
2	7.387	8121	125286	1.520	RDX
3	10.016	11795	192668	1.027	TNB
4	12.288	6899	135369	0.552	DNB
5	13.109	8809	171719	1.199	TETRYL
6	13.952	8800	189332	1.128	NB
7	15.168	9050	189686	1.706	3,4DNT
8	15.819	7375	164145	0.905	TNT
9	16.555	3813	100248	0.794	4ADNT
10	17.467	5217	141567	0.782	2ADNT

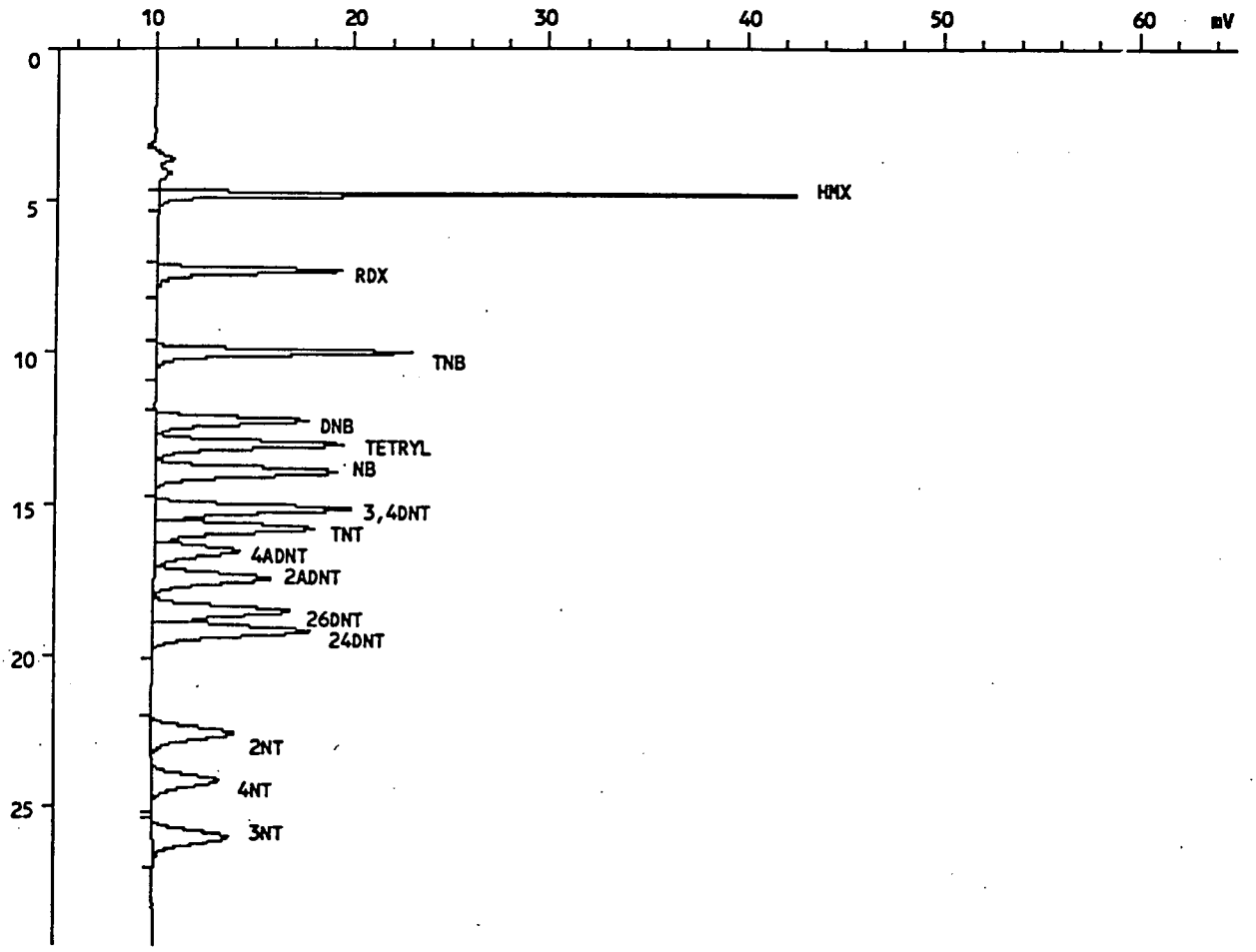
Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
11	18.533	6335	157430	1.316	26DNT
12	19.195	7311	197131	0.798	24DNT
13	22.528	3953	117390	1.151	2NT
14	24.080	3221	100090	1.145	4NT
15	25.957	3700	122151	1.112	3NT
Total		124226	2339148	18.552	
Residual		0	0	0.000	

LONG PLOT

Injection F: <MC3> 5 5EX1015C,9,1

Sample name.....: EXP L3 SOIL STD
Sample ID.....: 1-111:CA5
INST ID: IN13, COL. TYPE: ODS-II, ID: CARB-05, INJ:200,COL#1

Acquired on 18-Oct-97 at 02:53:13
Reported on 19-Nov-97 at 09:49:52



INJECTION REPORT

Injection F: <MC3> 5 5EX1015C,9,1

Acquired on 18-Oct-97 at 02:53:13
Modified on 18-Oct-81 at 12:22:02
Reported on 19-Nov-97 at 09:49:27

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN13, COL. TYPE: ODS-II, ID: CARB-05, INJ:200,COL#1
Number of samples.: 27
Calibration file..: 5EX1015 Last modified on 17-Nov-81 at 21:07:32
Method file.....: EXPLOS Last modified on 19-Nov-81 at 09:45:48
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: EXP L3 SOIL STD
Sample ID.....: 1-111:CA5
Sample type.....: Standard
Sample amount.....: 1.0000
Number of injections.....: 1
Bottle Number.....: 1

Retention time update.....: No update
Calibration level identifier.....: 3
Response factor update.....: No update

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 1.000
Dilution.....: 1.000
Amount (g or mL).....: 1000.000

PEAK INFORMATION

Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
1	4.843	32449	249718	3.630	HMX
2	7.381	9331	134479	1.632	RDX
3	10.016	13002	202583	1.080	TNB
4	12.293	7686	143737	0.586	DNB
5	13.083	9467	178575	1.247	TETRYL
6	13.979	9248	191055	1.139	NB
7	15.179	9914	200170	1.800	3,4DNT
8	15.819	8081	174788	0.964	TNT
9	16.555	4288	106475	0.844	4ADNT
10	17.451	5886	151593	0.838	2ADNT

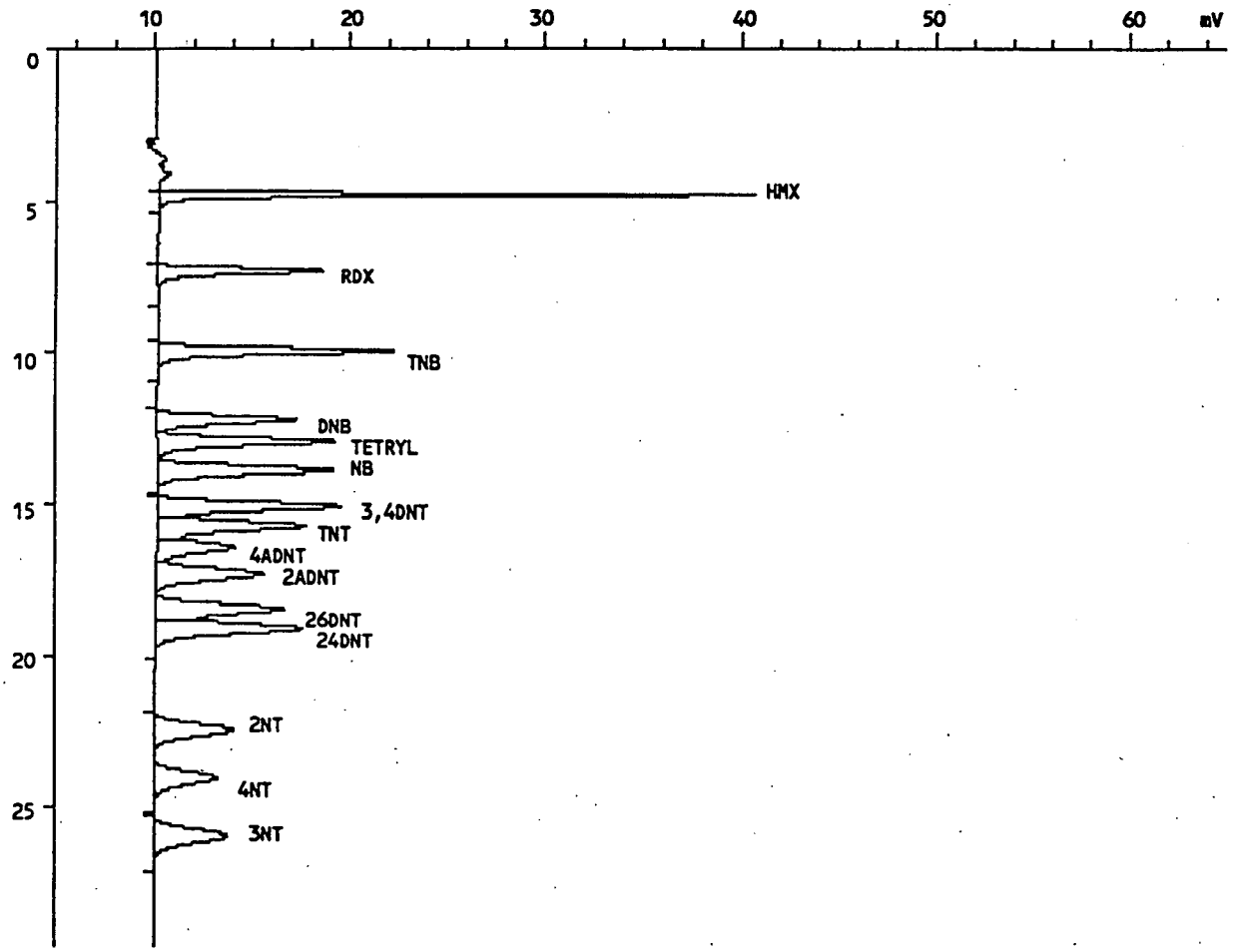
Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
11	18.544	6913	166415	1.391	26DNT
12	19.211	7982	208979	0.846	24DNT
13	22.581	4129	119687	1.173	2NT
14	24.139	3378	102562	1.173	4NT
15	26.021	3850	124268	1.132	3NT
Total		135604	2455085	19.474	
Residual		0	0	0.000	

LONG PLOT

Injection F: <MC3> 5 5EX1015D,2,1

Sample name.....: EXP L3 SOIL STD
Sample ID.....: 1-111:CA6
INST ID: IN13, COL. TYPE: ODS-II, ID: CARB-05, INJ:200,COL#1

Acquired on 18-Oct-97 at 17:16:37
Reported on 19-Nov-97 at 09:51:06



INJECTION REPORT

Injection F: <MC3> 5 5EX1015D,2,1

Acquired on 18-Oct-97 at 17:16:37
Modified on 19-Oct-81 at 19:14:26
Reported on 19-Nov-97 at 09:50:41

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN13, COL. TYPE: ODS-II, ID: CARB-05, INJ:200,COL#1
Number of samples.: 26
Calibration file..: 5EX1015 Last modified on 17-Nov-81 at 21:07:32
Method file.....: EXPLOS Last modified on 19-Nov-81 at 09:45:48
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: EXP L3 SOIL STD
Sample ID.....: 1-111:CA6
Sample type.....: Standard
Sample amount.....: 1.0000
Number of injections.....: 1
Bottle Number.....: 1

Retention time update.....: No update
Calibration level identifier.....: 3
Response factor update.....: No update

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 1.000
Dilution.....: 1.000
Amount (g or mL).....: 1000.000

PEAK INFORMATION

Peak	RT mins	Hght uV	Area uVs	ug/Kg	Peak name
1	4.821	30462	238453	3.466	HMX
2	7.333	8406	130545	1.584	RDX
3	9.973	12099	195660	1.043	TNB
4	12.229	7094	136600	0.557	DNB
5	12.976	9082	174591	1.219	TETRYL
6	13.931	8941	189903	1.132	NB
7	15.104	9366	192679	1.733	3,4DNT
8	15.744	7583	166843	0.920	TNT
9	16.416	3956	101555	0.805	4ADNT
10	17.291	5442	145992	0.807	2ADNT

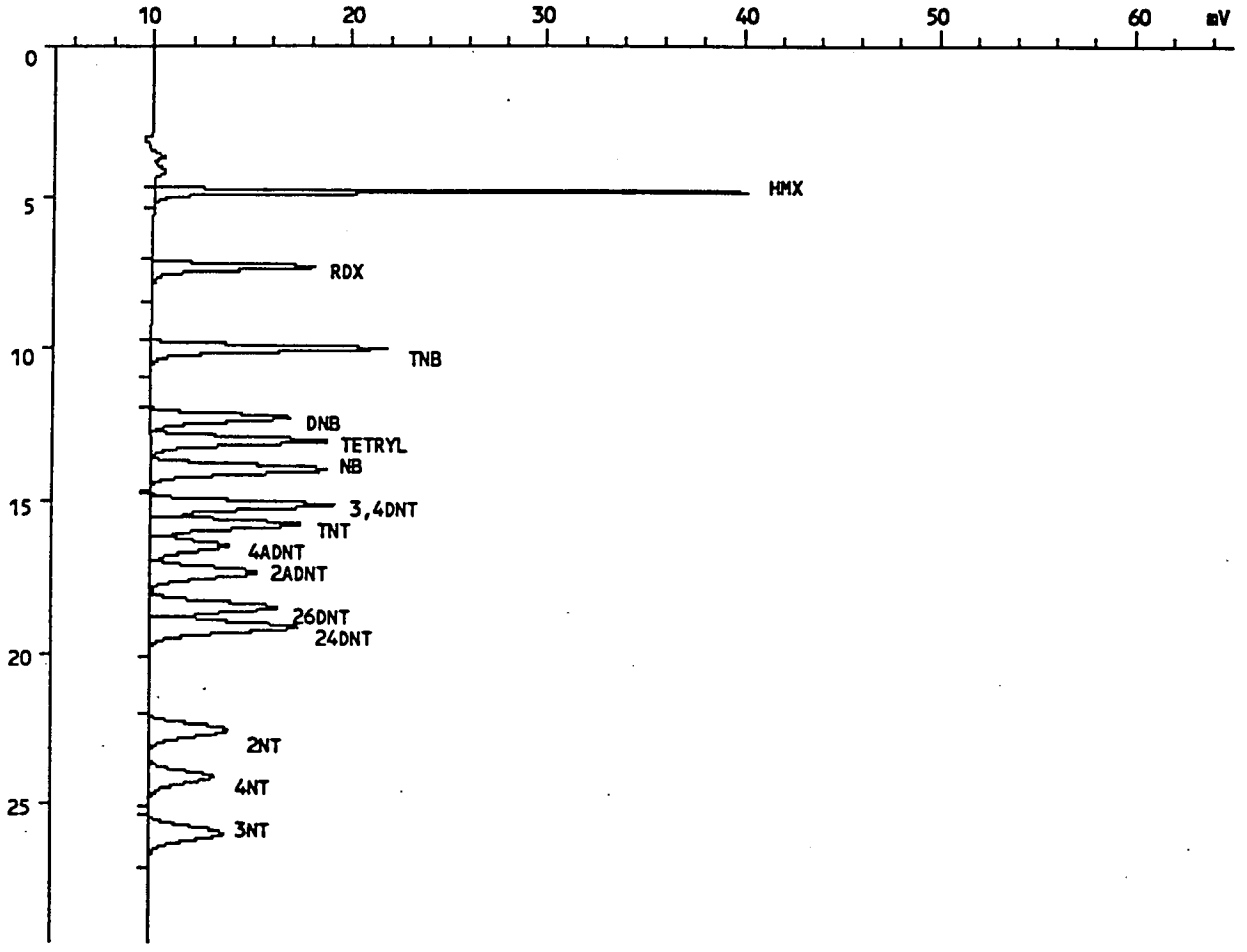
Peak	RT mins	Hght uV	Area uVs	ug/Kg	Peak name
11	18.437	6506	159806	1.336	26DNT
12	19.093	7492	200663	0.812	24DNT
13	22.491	3997	118465	1.161	2NT
14	24.059	3256	100932	1.155	4NT
15	25.925	3748	123362	1.124	3NT
Total		127429	2376050	18.853	
Residual		0	0	0.000	

LONG PLOT

Injection F: <MC3> 5 5EX1015D,9,1

Sample name.....: EXP L3 SOIL STD
Sample ID.....: 1-111:CA7
INST ID: IN13, COL. TYPE: ODS-II, ID: CARB-05, INJ:200,COL#1

Acquired on 18-Oct-97 at 22:29:21
Reported on 19-Nov-97 at 09:52:47



INJECTION REPORT

Injection F: <MC3> 5 5EX1015D,9,1

Acquired on 18-Oct-97 at 22:29:21
Modified on 19-Oct-81 at 19:15:44
Reported on 19-Nov-97 at 09:52:16

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN13, COL. TYPE: ODS-II, ID: CARB-05, INJ:200,COL#1
Number of samples.: 26
Calibration file..: 5EX1015 Last modified on 17-Nov-81 at 21:07:32
Method file.....: EXPLOS Last modified on 19-Nov-81 at 09:45:48
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: EXP L3 SOIL STD
Sample ID.....: 1-111:CA7
Sample type.....: Standard
Sample amount.....: 1.0000
Number of injections.....: 1
Bottle Number.....: 1

Retention time update.....: No update
Calibration level identifier.....: 3
Response factor update.....: No update

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 1.000
Dilution.....: 1.000
Amount (g or mL).....: 1000.000

PEAK INFORMATION

Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
1	4.859	30214	237512	3.452	HMX
2	7.376	8341	130257	1.581	RDX
3	10.021	11977	194101	1.035	TNB
4	12.288	7044	136175	0.555	DNB
5	13.045	8989	173156	1.209	TETRYL
6	13.984	8894	188999	1.126	NB
7	15.168	9270	191656	1.724	3,4DNT
8	15.803	7553	167039	0.921	TNT
9	16.501	3937	101562	0.805	4ADNT
10	17.376	5393	145148	0.802	2ADNT

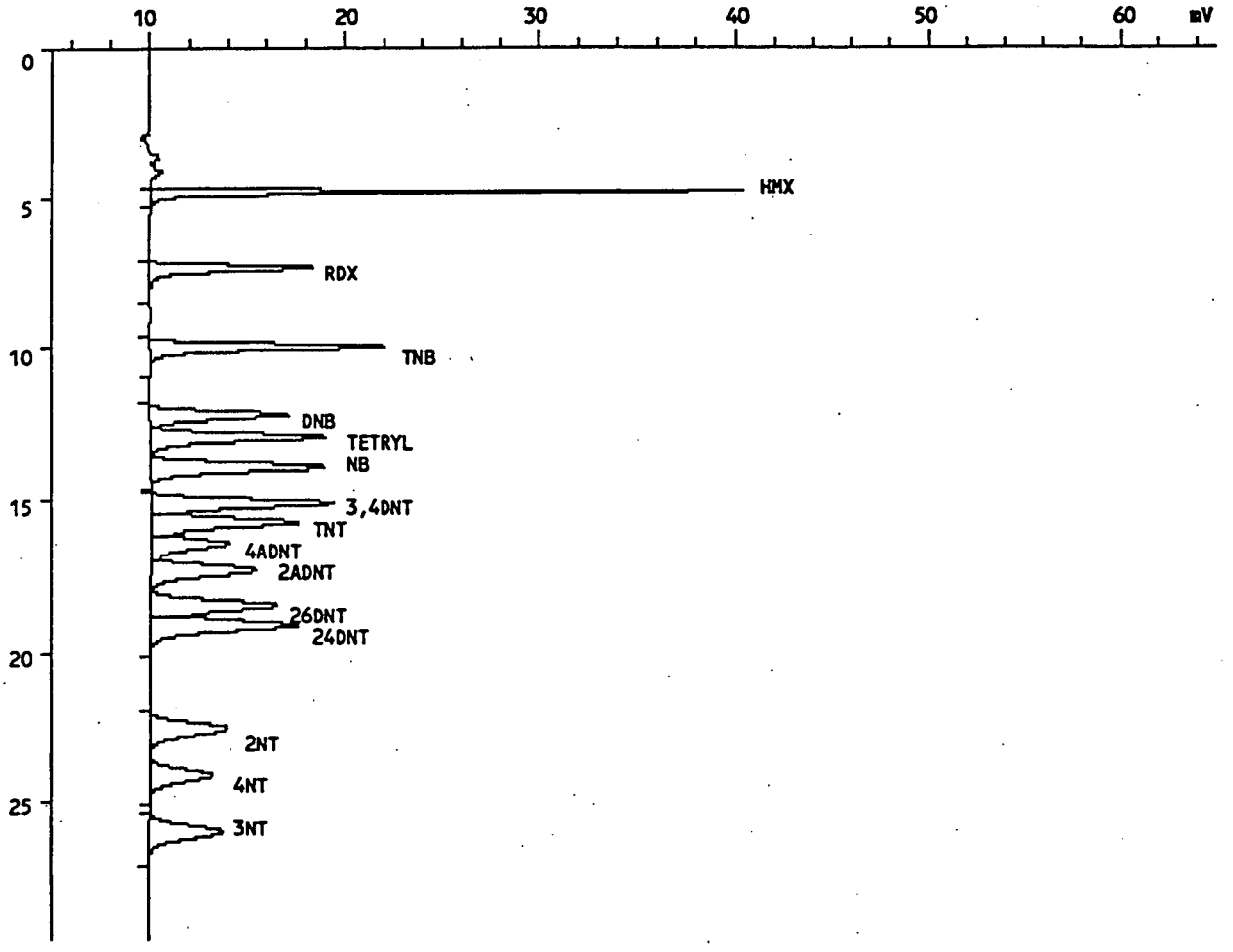
Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
11	18.512	6471	158920	1.329	26DNT
12	19.168	7460	200229	0.811	24DNT
13	22.555	3981	117991	1.157	2NT
14	24.117	3243	100408	1.149	4NT
15	25.989	3718	122581	1.116	3NT
Total		126485	2365734	18.771	
Residual		0	0	0.000	

LONG PLOT

Injection F: <MC3> 5 5EX1015D,19,1

Sample name.....: EXP L3 SOIL STD
Sample ID.....: 1-111:CA8
INST ID: IN13, COL. TYPE: ODS-II, ID: CARB-05, INJ:200,COL#1

Acquired on 19-Oct-97 at 05:56:35
Reported on 19-Nov-97 at 09:55:17



INJECTION REPORT

Injection F: <MC3> 5 5EX1015D,19,1

Acquired on 19-Oct-97 at 05:56:35
Modified on 19-Oct-81 at 19:15:56
Reported on 19-Nov-97 at 09:53:12

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN13, COL. TYPE: ODS-II, ID: CARB-05, INJ:200,COL#1
Number of samples.: 26
Calibration file..: 5EX1015 Last modified on 17-Nov-81 at 21:07:32
Method file.....: EXPLOS Last modified on 19-Nov-81 at 09:45:48
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: EXP L3 SOIL STD
Sample ID.....: 1-111:CA8
Sample type.....: Standard
Sample amount.....: 1.0000
Number of injections.....: 1
Bottle Number.....: 1

Retention time update.....: No update
Calibration level identifier.....: 3
Response factor update.....: No update

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 1.000
Dilution.....: 1.000
Amount (g or mL).....: 1000.000

PEAK INFORMATION

Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
1	4.827	30378	238270	3.463	HMX
2	7.339	8375	130381	1.582	RDX
3	9.979	12057	194838	1.039	TNB
4	12.245	7072	136549	0.557	DNB
5	12.976	8988	173655	1.213	TETRYL
6	13.952	8868	188690	1.124	NB
7	15.131	9301	192303	1.729	3,4DNT
8	15.755	7594	168198	0.927	TNT
9	16.437	3973	101820	0.807	4ADNT
10	17.312	5432	146552	0.810	2ADNT

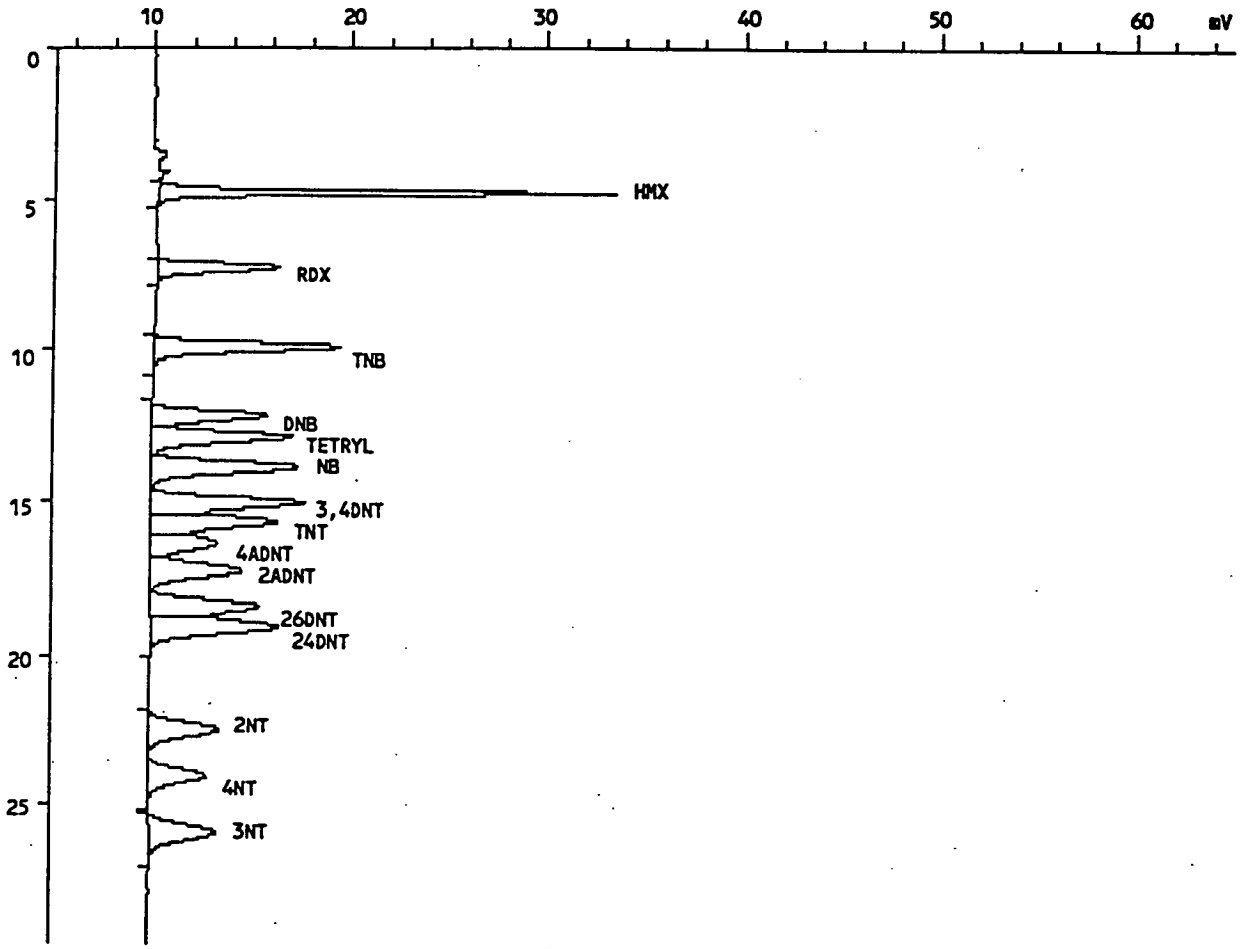
Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
11	18.464	6511	160171	1.339	26DNT
12	19.120	7493	200944	0.814	24DNT
13	22.533	3982	118782	1.165	2NT
14	24.101	3235	100963	1.155	4NT
15	25.973	3696	122349	1.114	3NT
Total		126955	2374465	18.838	
Residual		0	0	0.000	

LONG PLOT

Injection F: <MC3> 5 5EX1015E,1,1

Sample name.....: EXP L3 SOIL STD
Sample ID.....: 1-111:CA9
INST ID: IN13, COL. TYPE: ODS-II, ID: CARB-05, INJ:200,COL#1

Acquired on 19-Oct-97 at 19:42:54
Reported on 19-Nov-97 at 09:54:23



INJECTION REPORT

Injection F: <MC3> 5 5EX1015E,1,1

Acquired on 19-Oct-97 at 19:42:54
Modified on 20-Oct-81 at 20:35:56
Reported on 19-Nov-97 at 09:53:52

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN13, COL. TYPE: ODS-II, ID: CARB-05, INJ:200,COL#1
Number of samples.: 9
Calibration file..: 5EX1015 Last modified on 17-Nov-81 at 21:07:32
Method file.....: EXPLOS Last modified on 19-Nov-81 at 09:45:48
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: EXP L3 SOIL STD
Sample ID.....: 1-111:CA9
Sample type.....: Standard
Sample amount.....: 1.0000
Number of injections.....: 1
Bottle Number.....: 1

Retention time update.....: No update
Calibration level identifier.....: 3
Response factor update.....: No update

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 1.000
Dilution.....: 1.000
Amount (g or mL).....: 1000.000

PEAK INFORMATION

Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
1	4.795	23193	239757	3.485	HMX
2	7.275	6165	124426	1.510	RDX
3	9.920	9403	197895	1.055	TNB
4	12.187	5805	140500	0.573	DNB
5	12.896	7120	181010	1.264	TETRYL
6	13.899	7448	196448	1.171	NB
7	15.056	7753	199677	1.796	3,4DNT
8	15.685	6429	175351	0.967	TNT
9	16.357	3352	104792	0.830	4ADNT
10	17.232	4592	151897	0.839	2ADNT

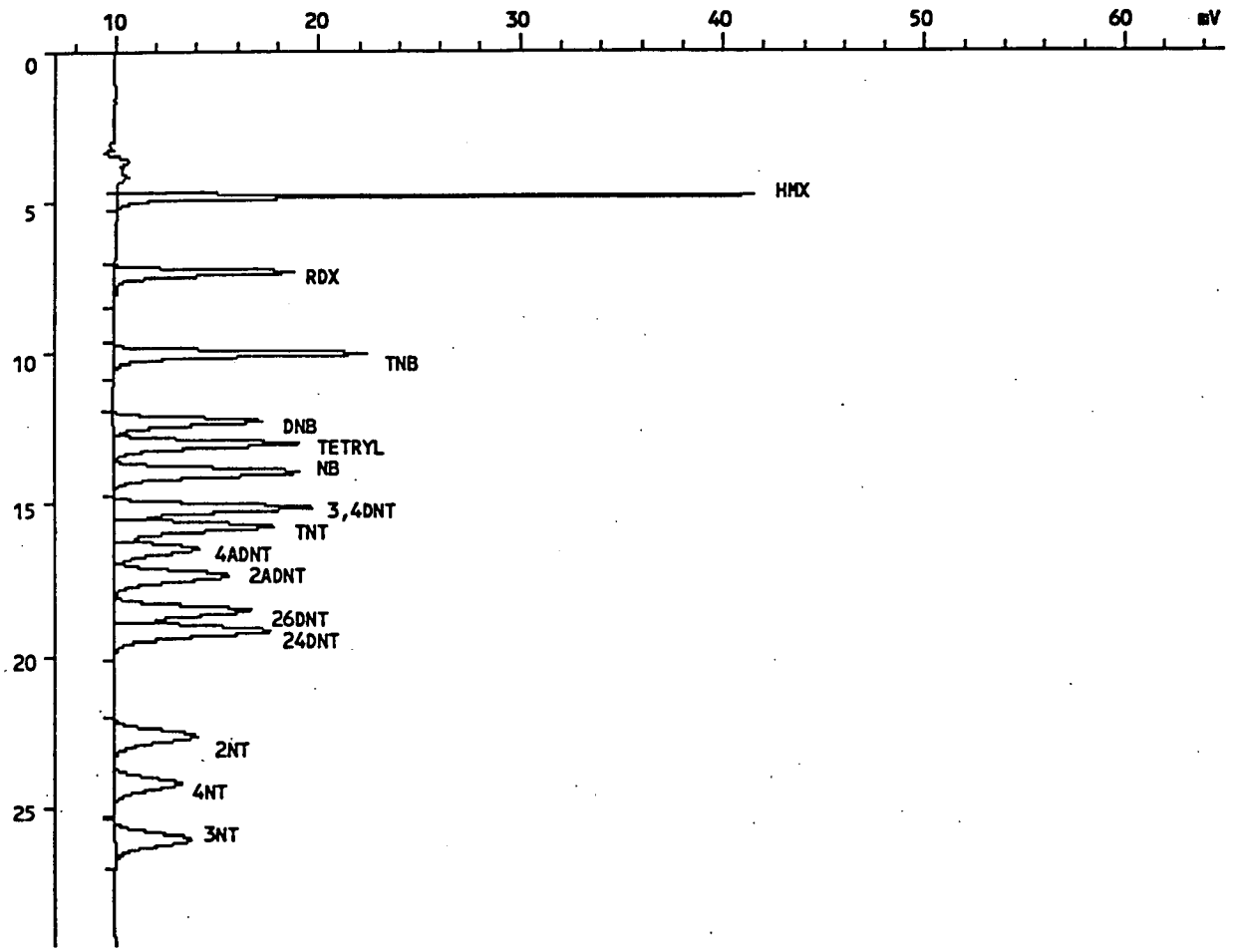
Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
11	18.400	5503	160277	1.340	26DNT
12	19.061	6492	208082	0.842	24DNT
13	22.475	3531	119882	1.175	2NT
14	24.032	2937	102364	1.171	4NT
15	25.899	3383	124072	1.130	3NT
Total		103106	2426430	19.148	
Residual		0	0	0.000	

LONG PLOT

Injection F: <MC3> 5 5EX1015E,9,1

Sample name.....: EXP L3 SOIL STD
Sample ID.....: 1-111:CA10
INST ID: IN13, COL. TYPE: ODS-II, ID: CARB-05, INJ:200,COL#1

Acquired on 20-Oct-97 at 01:40:17
Reported on 19-Nov-97 at 09:58:58



INJECTION REPORT

Injection F: <MC3> 5 5EX1015E,9,1

Acquired on 20-Oct-97 at 01:40:17
Modified on 26-Oct-81 at 20:06:20
Reported on 19-Nov-97 at 09:54:44

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN13, COL. TYPE: ODS-II, ID: CARB-05, INJ:200,COL#1
Number of samples.: 9
Calibration file..: 5EX1015 Last modified on 17-Nov-81 at 21:07:32
Method file.....: EXPLOS Last modified on 19-Nov-81 at 09:45:48
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: EXP L3 SOIL STD
Sample ID.....: 1-111:CA10
Sample type.....: Standard
Sample amount.....: 1.0000
Number of injections.....: 1
Bottle Number.....: 1

Retention time update.....: No update
Calibration level identifier.....: 3
Response factor update.....: No update

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 1.000
Dilution.....: 1.000
Amount (g or mL).....: 1000.000

PEAK INFORMATION

Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
1	4.837	31456	243625	3.500	HMX
2	7.360	8864	133004	1.550	RDX
3	10.005	12530	198019	1.040	TNB
4	12.277	7373	139579	0.548	DNB
5	13.035	9173	174529	1.205	TETRYL
6	13.984	9137	190764	1.118	NB
7	15.173	9664	196974	1.704	3,4DNT
8	15.803	7822	170886	0.871	TNT
9	16.512	4132	104117	0.734	4ADNT
10	17.392	5647	148644	0.777	2ADNT

Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
11	18.523	6731	162755	1.330	26DNT
12	19.184	7743	205072	0.818	24DNT
13	22.587	4097	120273	1.191	2NT
14	24.155	3342	102788	1.192	4NT
15	26.027	3807	123652	1.112	3NT
Total		131519	2414680	18.691	
Residual		0	0	0.000	

CALIBRATION FACTOR DATA
FORM 9A-1

EXPLOSIVE CALIBRATION - METHOD SW846-8330
Instrument ID: IN13
Column ID : CARB-05

Inj 1

Injection File Name Level 1 : 5EX1021,4
Injection File Name Level 2 : 5EX1021,5
Injection File Name Level 3 : 5EX1021,6
Injection File Name Level 4 : 5EX1021,7
Injection File Name Level 5 : 5EX1021,8

Calibration Date : 10/22/97
Number of Calibration Levels: 5
Line forced thru zero : Yes
Calculation Method : Area

COMPONENT NAME	Area Level 1	CAL FACTOR Level 1	Area Level 2	CAL FACTOR Level 2	Area Level 3	CAL FACTOR Level 3	Area Level 4	CAL FACTOR Level 4	Area Level 5	CAL FACTOR Level 5
HMX	3066	302	88544	349	231457	351	350142	354	464379	352
RDX	1355	321	44475	421	126587	459	190203	462	250351	455
TNB	2537	840	71239	942	190291	966	287092	970	379725	964
DNB	1895	1170	50409	1242	133184	1261	200295	1264	266051	1255
TETRYL	2288	615	64075	689	168132	695	252759	694	337865	698
NB	2496	756	70775	859	186081	870	279712	869	377789	879
3,4DNT	2602	512	71147	561	188169	570	281948	568	378994	574
TNT	2265	839	61822	915	164153	933	246131	932	329992	937
4ADNT	1262	535	37415	632	100532	653	149044	642	200444	651
2ADNT	1906	808	52901	894	143077	929	213551	920	285686	928
26DNT	2320	598	58139	598	156842	617	233509	614	310082	613
24DNT	2868	1215	73431	1240	196189	1274	295983	1276	392609	1275
[illegible]	1666	493	43507	514	116034	527	175851	533	232201	528
	1099	325	36566	432	98127	446	149796	454	197242	448
	1597	496	44953	559	120328	573	181820	579	240073	574

CALIBRATION FACTOR DATA
FORM 9B

EXPLOSIVE CALIBRATION - METHOD SW846-8330
Instrument ID: IN13
Column ID : CARB-05

Inj 1

Injection File Name Level 1 : 5EX1021,4
Injection File Name Level 2 : 5EX1021,5
Injection File Name Level 3 : 5EX1021,6
Injection File Name Level 4 : 5EX1021,7
Injection File Name Level 5 : 5EX1021,8

Calibration Date : 10/22/97
Number of Calibration Levels: 5
Line forced thru zero : Yes
Calculation Method : Area

Component Name	Mean Cal Factor	% RSD	Corr. Coeff.	Slope area/amt	Intercept
HMX	341	6.42	0.99999	352	0.000
RDX	424	14.06	0.99983	457	0.000
TNB	936	5.86	0.99998	966	0.000
DNB	1238	3.18	0.99998	1258	0.000
TETRYL	678	5.23	0.99999	696	0.000
NB	846	6.00	0.99997	874	0.000
3,4DNT	557	4.60	0.99997	572	0.000
[illegible]	911	4.54	0.99999	935	0.000
[illegible]NT	623	7.99	0.99994	648	0.000
2ADNT	896	5.72	0.99996	925	0.000
26DNT	608	1.55	0.99998	614	0.000
24DNT	1256	2.17	0.99999	1274	0.000
2NT	519	3.11	0.99996	529	0.000
4NT	421	12.88	0.99993	449	0.000
3NT	556	6.21	0.99997	575	0.000

CALIBRATION FACTOR DATA
FORM 9C

Initial Calibration Date: 10/22/97
Continuing Calibration #: 8
Continuing Cal Date : 10/24/97
Continuing Cal Level : 3
Instrument ID : IN13
Column ID : CARB-05
Injection File Name : 5EX1021E,1
Calculation Mode : Area

Component Name	Response Cont Std	Cont CF	Init CF Mean	%D	Init CF Slope	%D
HMX	233946	354	341	3.82	352	0.65
RDX	120238	436	424	2.86	457	4.68
TNB	192094	975	936	4.13	966	0.99
DNB	134333	1272	1238	2.72	1258	1.09
TETRYL	171228	708	678	4.32	696	1.61
NB	186596	872	846	3.02	874	0.23
3,4DNT	189233	573	557	2.91	572	0.31
TNT	163355	928	911	1.86	935	0.71
4ADNT	100148	650	623	4.46	648	0.33
2ADNT	142674	926	896	3.44	925	0.16
26DNT	156959	618	608	1.61	614	0.70
24DNT	197328	1281	1256	2.02	1274	0.56
2NT	116837	531	519	2.32	529	0.39
4NT	100123	455	421	8.07	449	1.27
T	121243	577	556	3.79	575	0.36

CALIBRATION FACTOR DATA
FORM 9C

Initial Calibration Date: 10/22/97
Continuing Calibration #: 9
Continuing Cal Date : 10/25/97
Continuing Cal Level : 3
Instrument ID : IN13
Column ID : CARB-05
Injection File Name : 5EX1021E,12
Calculation Mode : Area

Component Name	Response Cont Std	Cont CF	Init CF Mean	%D	Init CF Slope	%D
HMX	231926	351	341	2.92	352	0.22
RDX	122232	443	424	4.56	457	3.09
TNB	190518	967	936	3.28	966	0.16
DNB	134474	1273	1238	2.83	1258	1.19
TETRYL	169742	701	678	3.42	696	0.73
NB	187961	878	846	3.77	874	0.50
3,4DNT	188028	570	557	2.25	572	0.33
TNT	162657	924	911	1.43	935	1.14
4ADNT	98120	637	623	2.34	648	1.70
2ADNT	139272	904	896	0.97	925	2.23
26DNT	154581	609	608	0.07	614	0.83
24DNT	194755	1265	1256	0.69	1274	0.76
2NT	117474	534	519	2.88	529	0.94
4NT	99515	452	421	7.41	449	0.65
	121220	577	556	3.77	575	0.34

CALIBRATION FACTOR DATA
FORM 9D

EXPLOSIVE CALIBRATION - METHOD SW846-8330

ection volume = 200uL

Amount Units = ng

COMPONENT NAME	AMT ON COL Level 1	AMT ON COL Level 2	AMT ON COL Level 3	AMT ON COL Level 4	AMT ON COL Level 5
HMX	10.140	254.000	660.000	990.000	1320.000
RDX	4.220	105.600	276.000	412.000	550.000
TNB	3.020	75.600	197.000	296.000	394.000
DNB	1.620	40.600	105.600	158.400	212.000
TETRYL	3.720	93.000	242.000	364.000	484.000
NB	3.300	82.400	214.000	322.000	430.000
3,4DNT	5.080	126.800	330.000	496.000	660.000
TNT	2.700	67.600	176.000	264.000	352.000
4ADNT	2.360	59.200	154.000	232.000	308.000
2ADNT	2.360	59.200	154.000	232.000	308.000
26DNT	3.880	97.200	254.000	380.000	506.000
24DNT	2.360	59.200	154.000	232.000	308.000
2NT	3.380	84.600	220.000	330.000	440.000
4NT	3.380	84.600	220.000	330.000	440.000
3NT	3.220	80.400	210.000	314.000	418.000

CALIBRATION FACTOR DAT
FORM 9E

EXPLOSIVE CALIBRATION - METHOD SW846-8330

Instrument ID : IN13
Column ID : CARB-05
Calibration Date : 10/22/97
Number of Calibration Levels: 5

COMPONENT NAME	RT Level 1	RT Level 2	RT Level 3	RT Level 4	RT Level 5
HMX	4.84	4.85	4.85	4.84	4.84
RDX	7.37	7.37	7.37	7.34	7.34
TNB	10.01	10.00	10.00	9.97	9.97
DNB	12.25	12.27	12.26	12.23	12.23
TETRYL	13.03	13.04	13.02	12.99	12.98
NB	13.95	13.96	13.95	13.92	13.92
3,4DNT	15.15	15.15	15.14	15.10	15.10
TNT	15.79	15.78	15.77	15.74	15.74
4ADNT	16.49	16.50	16.49	16.44	16.44
2ADNT	17.39	17.38	17.38	17.33	17.33
26DNT	18.52	18.49	18.49	18.44	18.44
24DNT	19.16	19.15	19.15	19.10	19.10
2NT	22.56	22.51	22.53	22.49	22.49
2NT					
4NT	24.14	24.08	24.10	24.05	24.04
3NT	25.94	25.94	25.97	25.92	25.92

COMPONENT NAME	RT Cont CA6	RT Cont CA7	RT Cont CA8	RT Cont CA9	RT Cont CA10
HMX	4.86	4.85	4.86	4.85	4.85
RDX	7.39	7.37	7.36	7.37	7.35
TNB	10.01	9.98	9.98	9.98	9.97
DNB	12.27	12.24	12.22	12.24	12.21
TETRYL	13.05	12.99	12.99	12.99	12.93
NB	13.94	13.90	13.89	13.92	13.89
3,4DNT	15.12	15.06	15.04	15.08	15.03
TNT	15.78	15.72	15.71	15.73	15.69
4ADNT	16.51	16.41	16.40	16.44	16.36
2ADNT	17.40	17.30	17.30	17.33	17.24
26DNT	18.47	18.40	18.38	18.43	18.36
24DNT	19.13	19.06	19.05	19.09	19.02
2NT	22.48	22.41	22.37	22.43	22.37
4NT	24.03	23.96	23.92	23.98	23.92
3NT	25.90	25.82	25.78	25.85	25.78

COMPONENT NAME	AVE RT	STD_DEV	3X SD	AVE+-3X SD
HMX	4.85	0.011	0.033	4.81 - 4.88
RDX	7.36	0.022	0.065	7.29 - 7.42
TNB	9.98	0.019	0.056	9.93 - 10.04
DNB	12.24	0.025	0.075	12.16 - 12.31
TETRYL	12.99	0.050	0.149	12.84 - 13.14
NB	13.92	0.026	0.078	13.84 - 14.00
3,4DNT	15.09	0.042	0.125	14.96 - 15.21
TNT	15.74	0.036	0.108	15.63 - 15.84

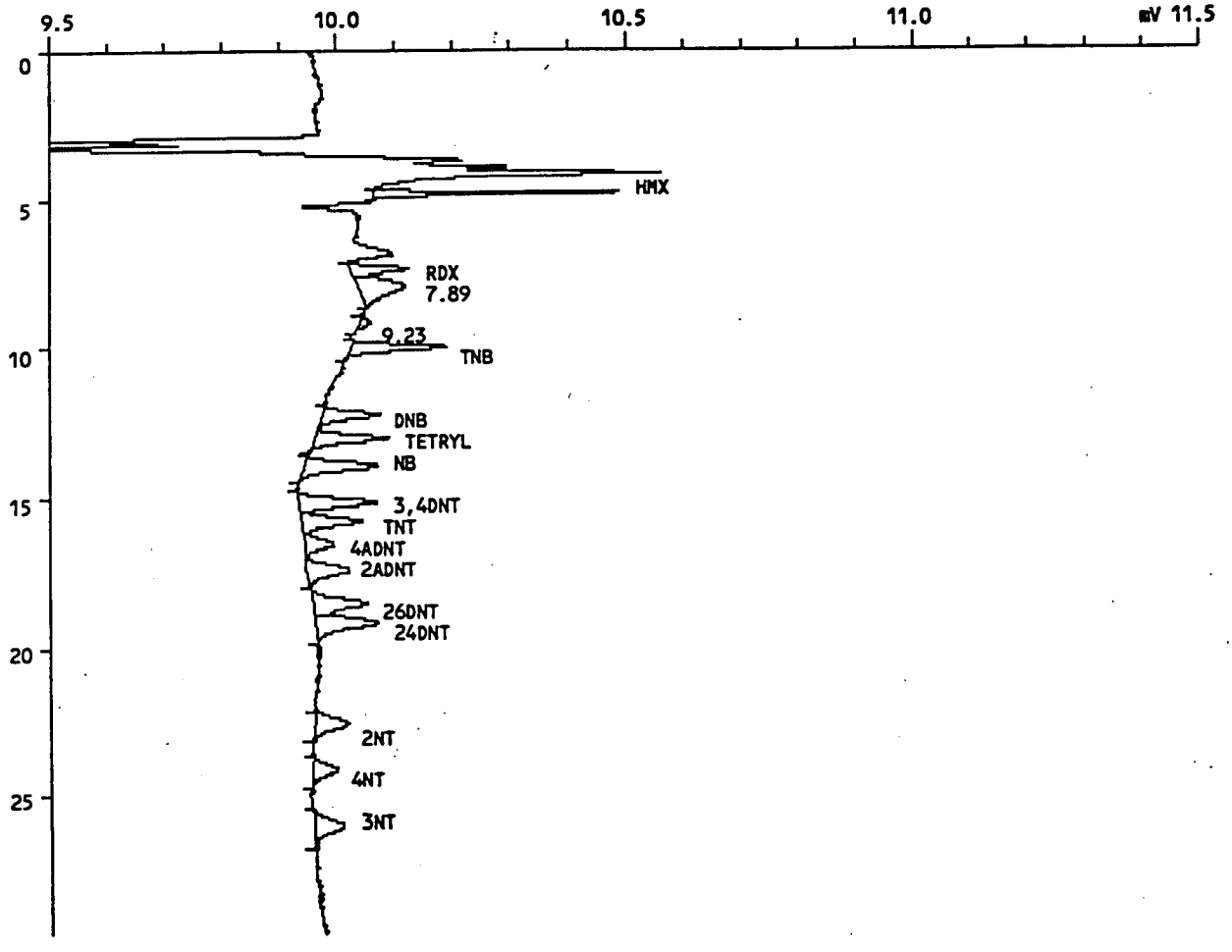
4ADNT	16.44	0.056	0.169	16.27 - 16.61
2ADNT	17.32	0.065	0.196	17.13 - 17.52
26DNT	18.43	0.048	0.143	18.29 - 18.57
24DNT	19.09	0.048	0.144	18.95 - 19.24
2NT	22.45	0.058	0.174	22.28 - 22.63
4NT	24.01	0.066	0.198	23.81 - 24.21
	25.87	0.064	0.193	25.68 - 26.06

LONG PLOT

Injection F: <MC3> 5 5EX1021,4,1

Sample name.....: EXP L1 SOIL STD
Sample ID.....: 1-129:IA1
INST ID: IN13, COL. TYPE: ODS-II, ID: CARB-05, INJ:200,COL#1

Acquired on 22-Oct-97 at 05:44:19
Reported on 30-Oct-97 at 15:45:44



INJECTION REPORT

Injection F: <MC3> 5 5EX1021,4,1

Acquired on 22-Oct-97 at 05:44:19
Modified on 30-Oct-81 at 15:37:32
Reported on 30-Oct-97 at 15:38:06

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN13, COL. TYPE: ODS-II, ID: CARB-05, INJ:200,COL#1
Number of samples.: 8
Calibration file..: 5EX1021 Last modified on 30-Oct-81 at 15:37:30
Method file.....: EXPLOS Last modified on 30-Oct-81 at 15:37:50
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: EXP L1 SOIL STD
Sample ID.....: 1-129:IA1
Sample type.....: Standard
Sample amount.....: 1.0000
Number of injections.....: 1
Bottle Number.....: 1

Retention time update.....: No update
Calibration level identifier.....: 1
Response factor update.....: Initialise

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 1.000
Dilution.....: 1.000
Amount (g or mL).....: 1.000

PEAK INFORMATION

Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
1	4.837	427	3066	0.044	HMX
2	7.371	98	1355	0.015	RDX
5	10.005	168	2537	0.013	TNB
6	12.251	103	1895	7.528E-003	DNB
7	13.029	128	2288	0.016	TETRYL
8	13.952	128	2496	0.014	NB
9	15.152	134	2602	0.023	3,4DNT
10	15.787	106	2265	0.012	TNT
11	16.491	51	1262	0.011	4ADNT
12	17.392	74	1906	0.010	2ADNT

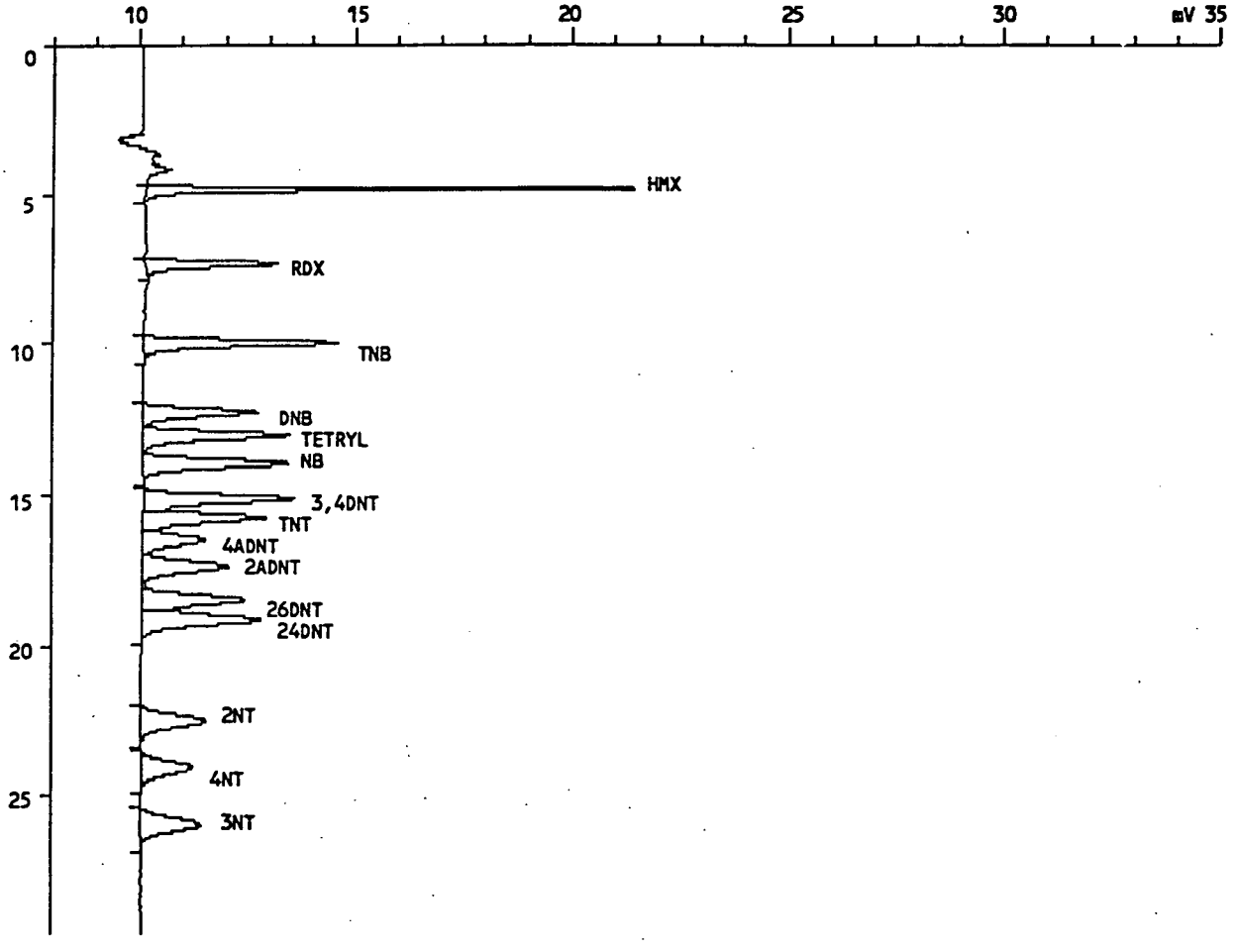
Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
13	18.523	97	2320	0.019	26DNT
14	19.163	111	2868	0.011	24DNT
15	22.560	59	1666	0.016	2NT
16	24.139	42	1099	0.012	4NT
17	25.941	52	1597	0.014	3NT
Total		1777	31224	0.238	
Residual		106	3181	0.035	

LONG PLOT

Injection F: <MC3> 5 5EX1021,5,1

Sample name.....: EXP L2 SOIL STD
Sample ID.....: 1-128:IA2
INST ID: IN13, COL. TYPE: ODS-II, ID: CARB-05, INJ:200,COL#1

Acquired on 22-Oct-97 at 06:29:03
Reported on 30-Oct-97 at 15:44:50



INJECTION REPORT

Injection F: <MC3> 5 5EX1021,5,1

Acquired on 22-Oct-97 at 06:29:03
Modified on 30-Oct-81 at 15:36:18
Reported on 30-Oct-97 at 15:38:28

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN13, COL. TYPE: ODS-II, ID: CARB-05, INJ:200,COL#1
Number of samples.: 8
Calibration file..: 5EX1021 Last modified on 30-Oct-81 at 15:37:30
Method file.....: EXPLOS Last modified on 30-Oct-81 at 15:37:50
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: EXP L2 SOIL STD
Sample ID.....: 1-128:IA2
Sample type.....: Standard
Sample amount.....: 1.0000
Number of injections.....: 1
Bottle Number.....: 1

Retention time update.....: No update
Calibration level identifier.....: 2
Response factor update.....: Initialise

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 1.000
Dilution.....: 1.000
Amount (g or mL).....: 1.000

PEAK INFORMATION

Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
1	4.853	11344	88544	1.256	HMX
2	7.371	3066	44475	0.489	RDX
3	10.000	4497	71239	0.369	TNB
4	12.272	2637	50409	0.200	DNB
5	13.035	3363	64075	0.449	TETRYL
6	13.957	3365	70775	0.406	NB
7	15.147	3482	71147	0.623	3,4DNT
8	15.781	2823	61822	0.331	TNT
9	16.496	1459	37415	0.330	4ADNT
10	17.381	1992	52901	0.285	2ADNT

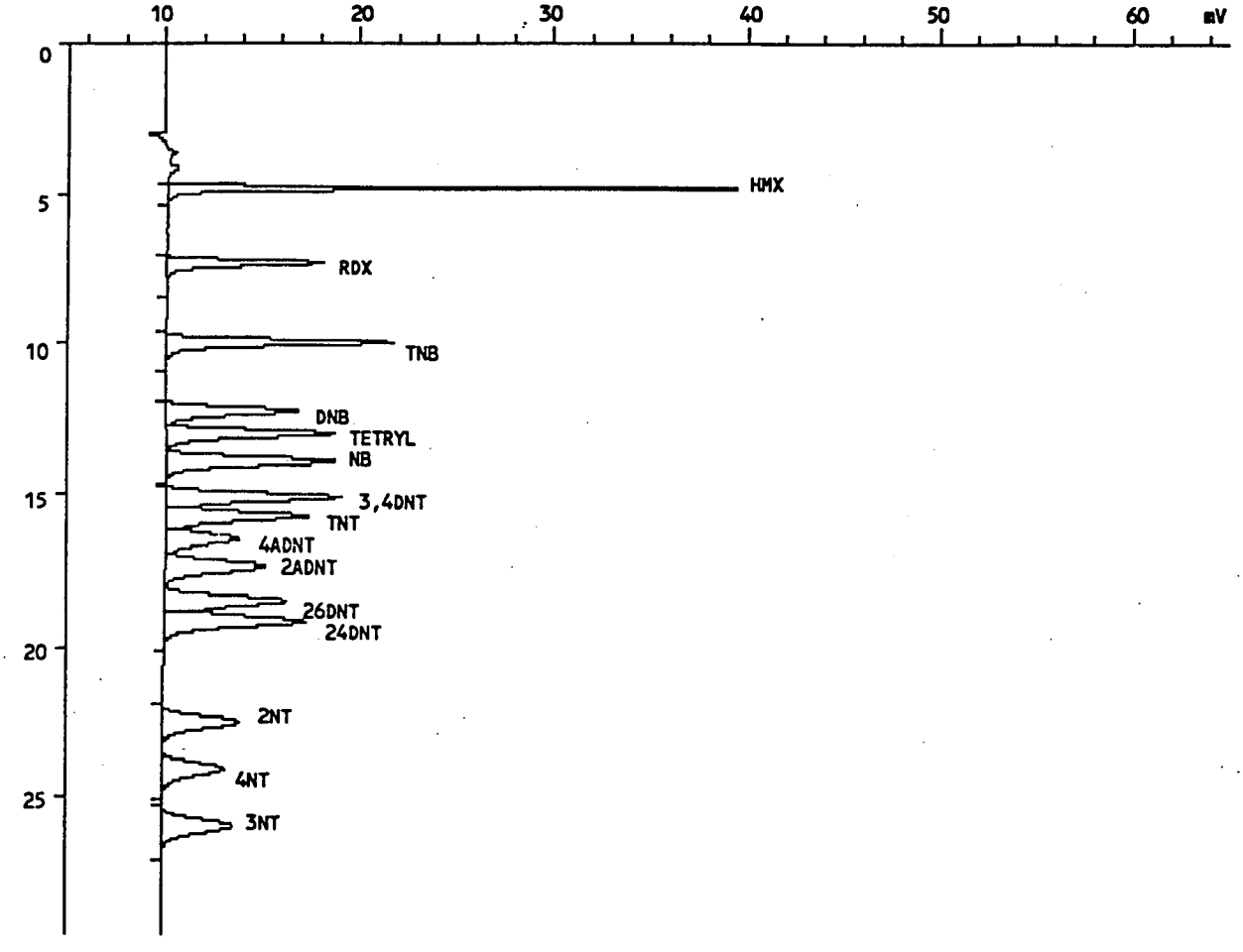
Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
11	18.485	2402	58139	0.473	26DNT
12	19.152	2779	73431	0.289	24DNT
13	22.512	1490	43507	0.412	2NT
14	24.080	1205	36566	0.407	4NT
15	25.936	1383	44953	0.391	3NT
Total		47287	869398	6.712	
Residual		0	0	0.000	

LONG PLOT

Injection F: <MC3> 5 5EX1021,6,1

Sample name.....: EXP L3 SOIL STD
Sample ID.....: 1-127:IA3
INST ID: IN13, COL. TYPE: ODS-II, ID: CARB-05, INJ:200,COL#1

Acquired on 22-Oct-97 at 07:13:48
Reported on 30-Oct-97 at 15:44:09



INJECTION REPORT

Injection F: <MC3> 5 5EX1021,6,1

Acquired on 22-Oct-97 at 07:13:48
Modified on 30-Oct-81 at 15:36:22
Reported on 30-Oct-97 at 15:38:52

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN13, COL. TYPE: ODS-II, ID: CARB-05, INJ:200,COL#1
Number of samples.: 8
Calibration file..: 5EX1021 Last modified on 30-Oct-81 at 15:37:30
Method file.....: EXPLOS Last modified on 30-Oct-81 at 15:37:50
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: EXP L3 SOIL STD
Sample ID.....: 1-127:IA3
Sample type.....: Standard
Sample amount.....: 1.0000
Number of injections.....: 1
Bottle Number.....: 1

Retention time update.....: No update
Calibration level identifier.....: 3
Response factor update.....: Initialise

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 1.000
Dilution.....: 1.000
Amount (g or mL).....: 1.000

PEAK INFORMATION

Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
1	4.848	29402	231457	3.286	HMX
2	7.365	8031	126587	1.383	RDX
3	9.995	11655	190291	0.985	TNB
4	12.261	6831	133184	0.529	DNB
5	13.024	8688	168132	1.186	TETRYL
6	13.947	8685	186081	1.066	NB
7	15.136	9012	188169	1.647	3,4DNT
8	15.771	7355	164153	0.878	TNT
9	16.485	3824	100532	0.886	4ADNT
10	17.376	5221	143077	0.771	2ADNT

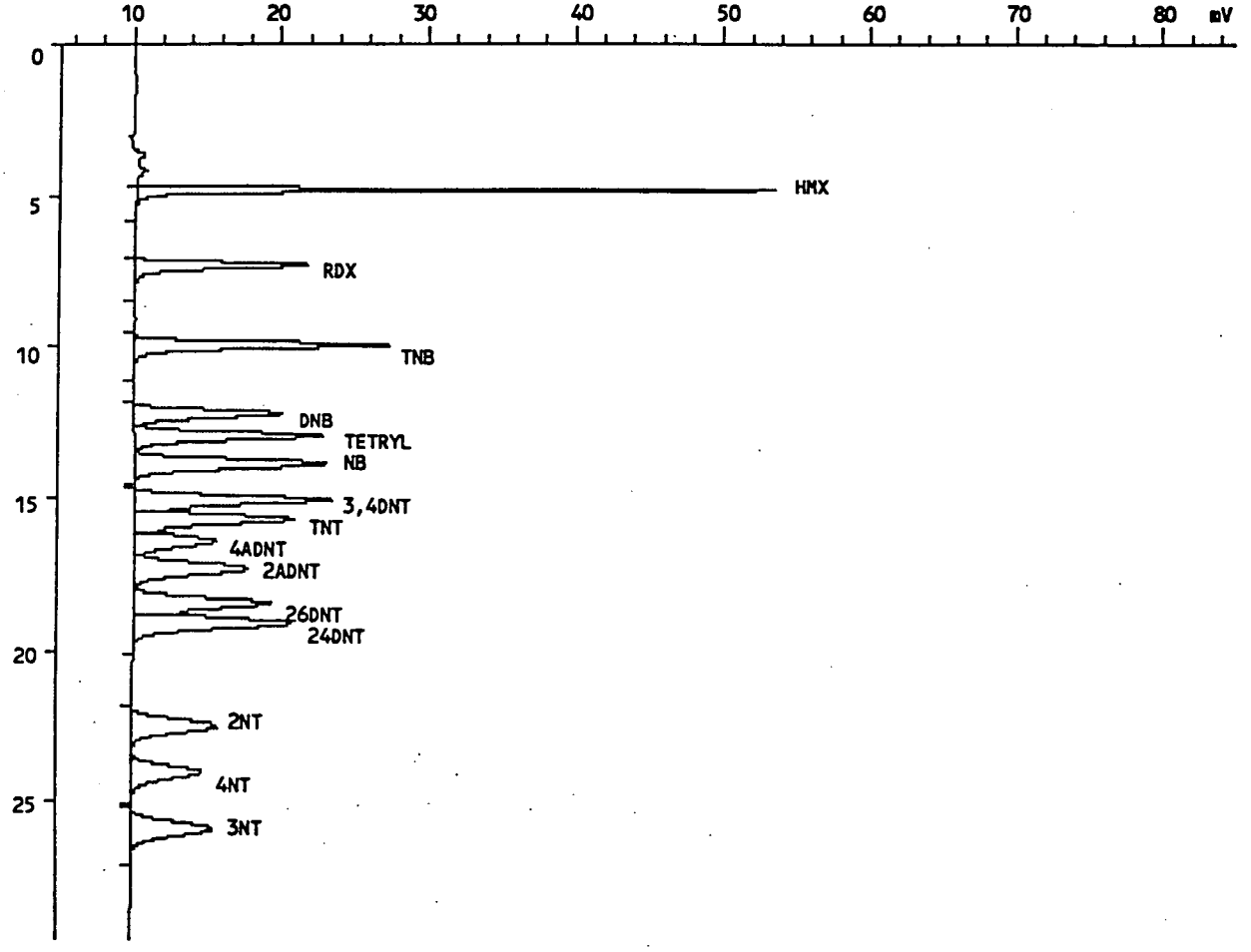
Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
11	18.485	6295	156842	1.275	26DNT
12	19.147	7241	196189	0.771	24DNT
13	22.533	3889	116034	1.098	2NT
14	24.101	3151	98127	1.093	4NT
15	25.973	3627	120328	1.047	3NT
Total		122909	2319182	17.902	
Residual		0	0	0.000	

LONG PLOT

Injection F: <MC3> 5 5EX1021,7,1

Sample name.....: EXP L4 SOIL STD
Sample ID.....: 1-126:IA4
INST ID: IN13, COL. TYPE: ODS-II, ID: CARB-05, INJ:200,COL#1

Acquired on 22-Oct-97 at 07:58:33
Reported on 30-Oct-97 at 15:42:58



INJECTION REPORT

Injection F: <MC3> 5 5EX1021,7,1

Acquired on 22-Oct-97 at 07:58:33
Modified on 30-Oct-81 at 15:36:30
Reported on 30-Oct-97 at 15:39:16

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN13, COL. TYPE: ODS-II, ID: CARB-05, INJ:200,COL#1
Number of samples.: 8
Calibration file..: 5EX1021 Last modified on 30-Oct-81 at 15:37:30
Method file.....: EXPLOS Last modified on 30-Oct-81 at 15:37:50
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: EXP L4 SOIL STD
Sample ID.....: 1-126:IA4
Sample type.....: Standard
Sample amount.....: 1.0000
Number of injections.....: 1
Bottle Number.....: 1

Retention time update.....: No update
Calibration level identifier.....: 4
Response factor update.....: Initialise

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 1.000
Dilution.....: 1.000
Amount (g or mL).....: 1.000

PEAK INFORMATION

Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
1	4.837	43426	350142	4.971	HMX
2	7.344	11806	190203	2.081	RDX
3	9.968	17208	287092	1.487	TNB
4	12.229	10099	200295	0.796	DNB
5	12.987	12832	252759	1.815	TETRYL
6	13.920	12858	279712	1.600	NB
7	15.099	13359	281948	2.466	3,4DNT
8	15.739	10881	246131	1.316	TNT
9	16.443	5634	149044	1.316	4ADNT
10	17.328	7723	213551	1.154	2ADNT

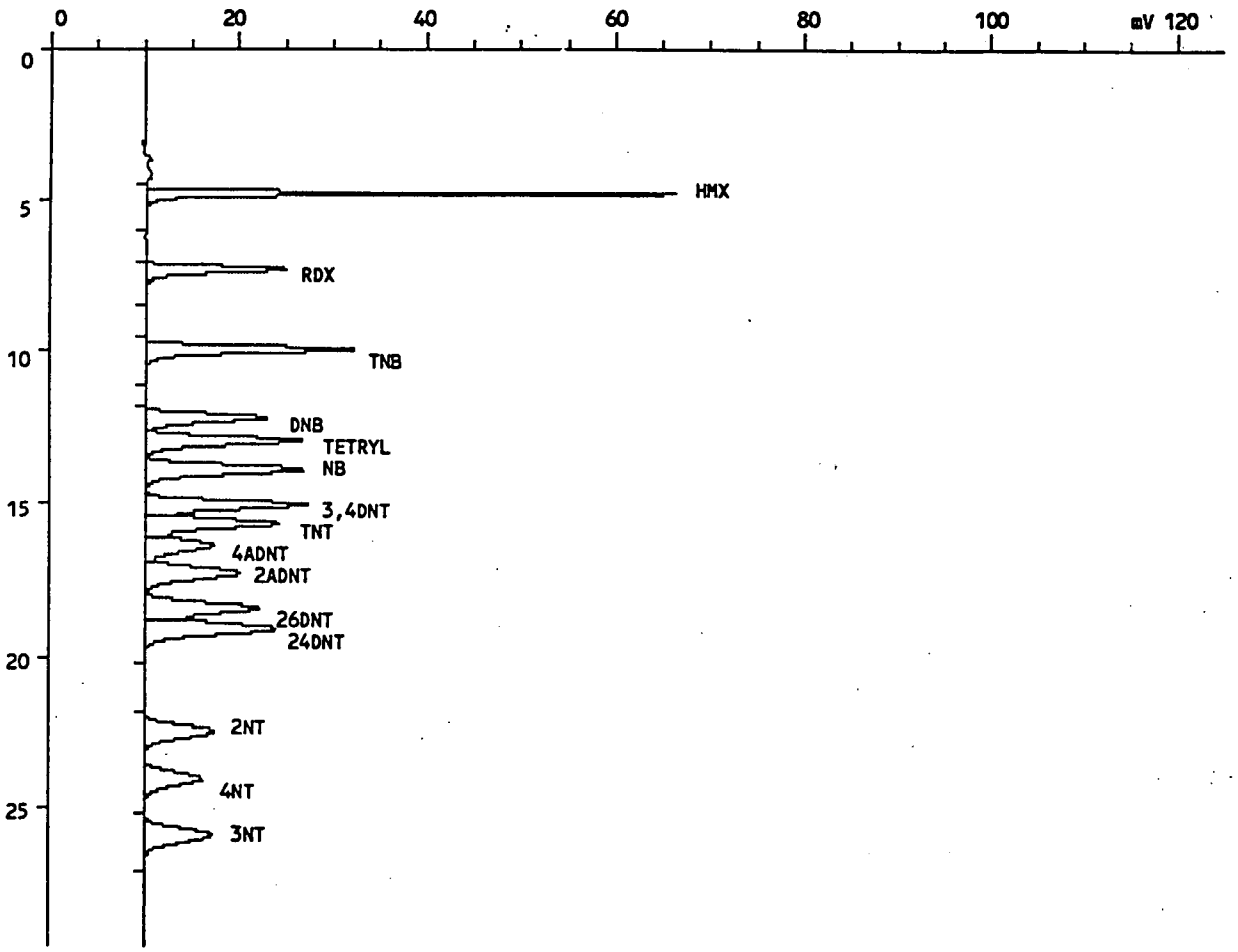
Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
11	18.443	9328	233509	1.903	26DNT
12	19.104	10782	295983	1.161	24DNT
13	22.485	5820	175851	1.662	2NT
14	24.048	4735	149796	1.667	4NT
15	25.915	5422	181820	1.580	3NT
Total		181914	3487837	26.977	
Residual		0	0	0.000	

LONG PLOT

Injection F: <MC3> 5 5EX1021,8,1

Sample name.....: EXP L5 SOIL STD
Sample ID.....: 1-125:IA5
INST ID: IN13, COL. TYPE: ODS-II, ID: CARB-05, INJ:200,COL#1

Acquired on 22-Oct-97 at 08:43:19
Reported on 30-Oct-97 at 15:40:50



INJECTION REPORT

Injection F: <MC3> 5 5EX1021,8,1

Acquired on 22-Oct-97 at 08:43:19
Modified on 30-Oct-81 at 15:36:36
Reported on 30-Oct-97 at 15:39:39

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN13, COL. TYPE: ODS-II, ID: CARB-05, INJ:200,COL#1
Number of samples.: 8
Calibration file..: 5EX1021 Last modified on 30-Oct-81 at 15:37:30
Method file.....: EXPLOS Last modified on 30-Oct-81 at 15:37:50
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: EXP L5 SOIL STD
Sample ID.....: 1-125:IA5
Sample type.....: Standard
Sample amount.....: 1.0000
Number of injections.....: 1
Bottle Number.....: 1

Retention time update.....: No update
Calibration level identifier.....: 5
Response factor update.....: Initialise

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 1.000
Dilution.....: 1.000
Amount (g or mL).....: 1.000

PEAK INFORMATION

Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
1	4.837	56297	464379	6.593	HMX
2	7.344	15157	250351	2.739	RDX
3	9.968	22250	379725	1.966	TNB
4	12.229	13091	266051	1.057	DNB
5	12.981	16661	337865	2.426	TETRYL
6	13.920	16780	377789	2.161	NB
7	15.099	17470	378994	3.315	3,4DNT
8	15.739	14265	329992	1.765	TNT
9	16.443	7375	200444	1.770	4ADNT
10	17.328	10057	285686	1.544	2ADNT

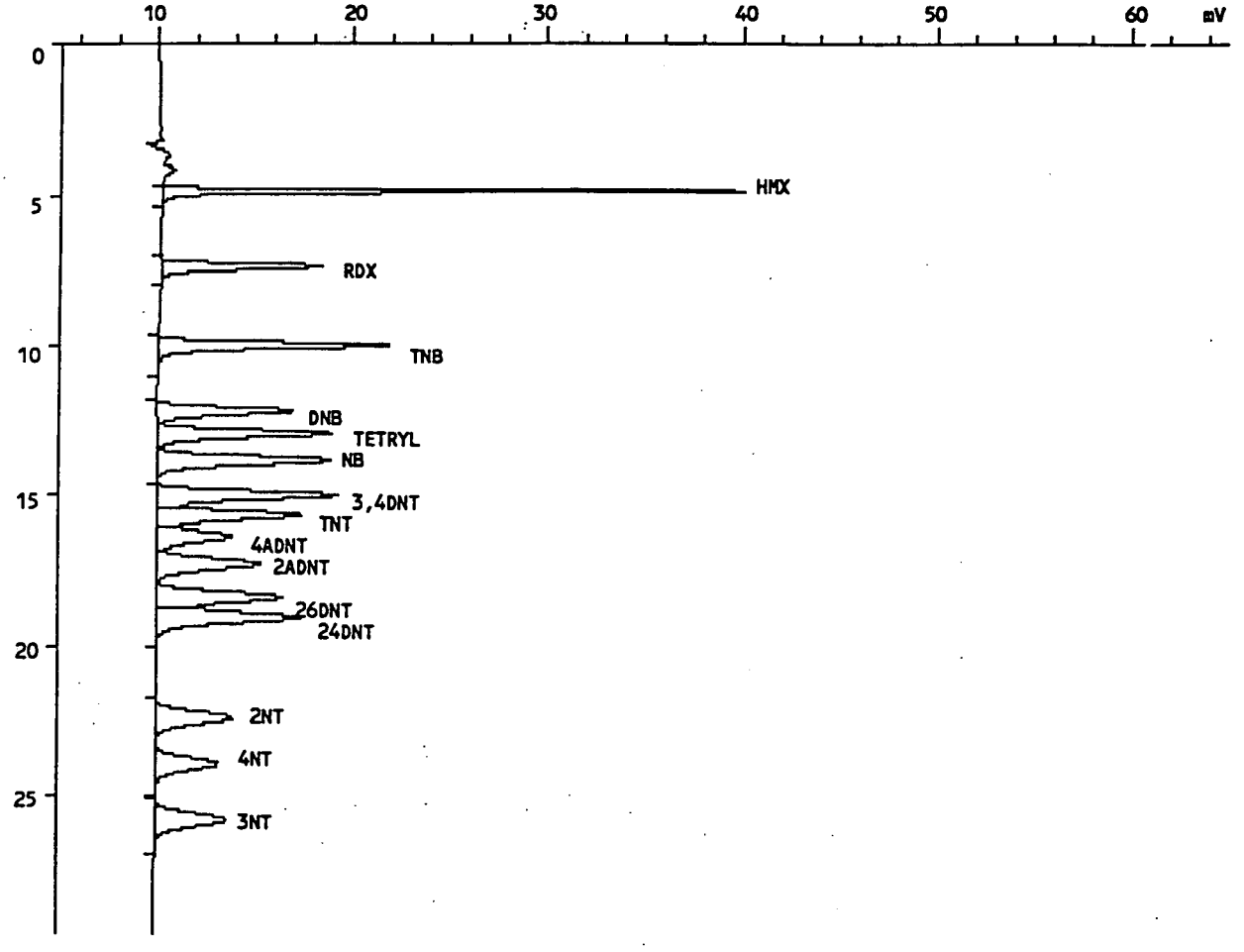
Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
11	18.443	12177	310082	2.526	26DNT
12	19.104	14078	392609	1.541	24DNT
13	22.485	7599	232201	2.195	2NT
14	24.043	6193	197242	2.195	4NT
15	25.915	7122	240073	2.087	3NT
Total		236570	4643483	35.881	
Residual		0	0	0.000	

LONG PLOT

Injection F: <MC3> 5 5EX1021E,1,1

Sample name.....: EXP L3 SOIL STD
Sample ID.....: 1-127:CA8
INST ID: IN13, COL. TYPE: ODS-II, ID: CARB-05, INJ:200,COL#1

Acquired on 24-Oct-97 at 18:07:50
Reported on 30-Oct-97 at 17:14:57



INJECTION REPORT

Injection F: <MC3> 5 5EX1021E,1,1

Acquired on 24-Oct-97 at 18:07:50
Modified on 30-Oct-81 at 17:14:30
Reported on 30-Oct-97 at 17:14:33

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN13, COL. TYPE: ODS-II, ID: CARB-05, INJ:200,COL#1
Number of samples.: 34
Calibration file..: 5EX1021 Last modified on 30-Oct-81 at 16:00:08
Method file.....: EXPLOS Last modified on 30-Oct-81 at 16:03:44
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: EXP L3 SOIL STD
Sample ID.....: 1-127:CA8
Sample type.....: Standard
Sample amount.....: 1.0000
Number of injections.....: 1
Bottle Number.....: 1

Retention time update.....: No update
Calibration level identifier.....: 3
Response factor update.....: No update

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 1.000
Dilution.....: 1.000
Amount (g or mL).....: 1000.000

PEAK INFORMATION

Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
1	4.859	29859	233946	3.430	HMX
2	7.360	8167	120238	1.418	RDX
3	9.979	11860	192094	1.026	TNB
4	12.224	6980	134333	0.543	DNB
5	12.987	8923	171228	1.263	TETRYL
6	13.888	8834	186596	1.103	NB
7	15.040	9175	189233	1.699	3,4DNT
8	15.712	7413	163355	0.897	TNT
9	16.400	3885	100148	0.804	4ADNT
10	17.296	5343	142674	0.796	2ADNT

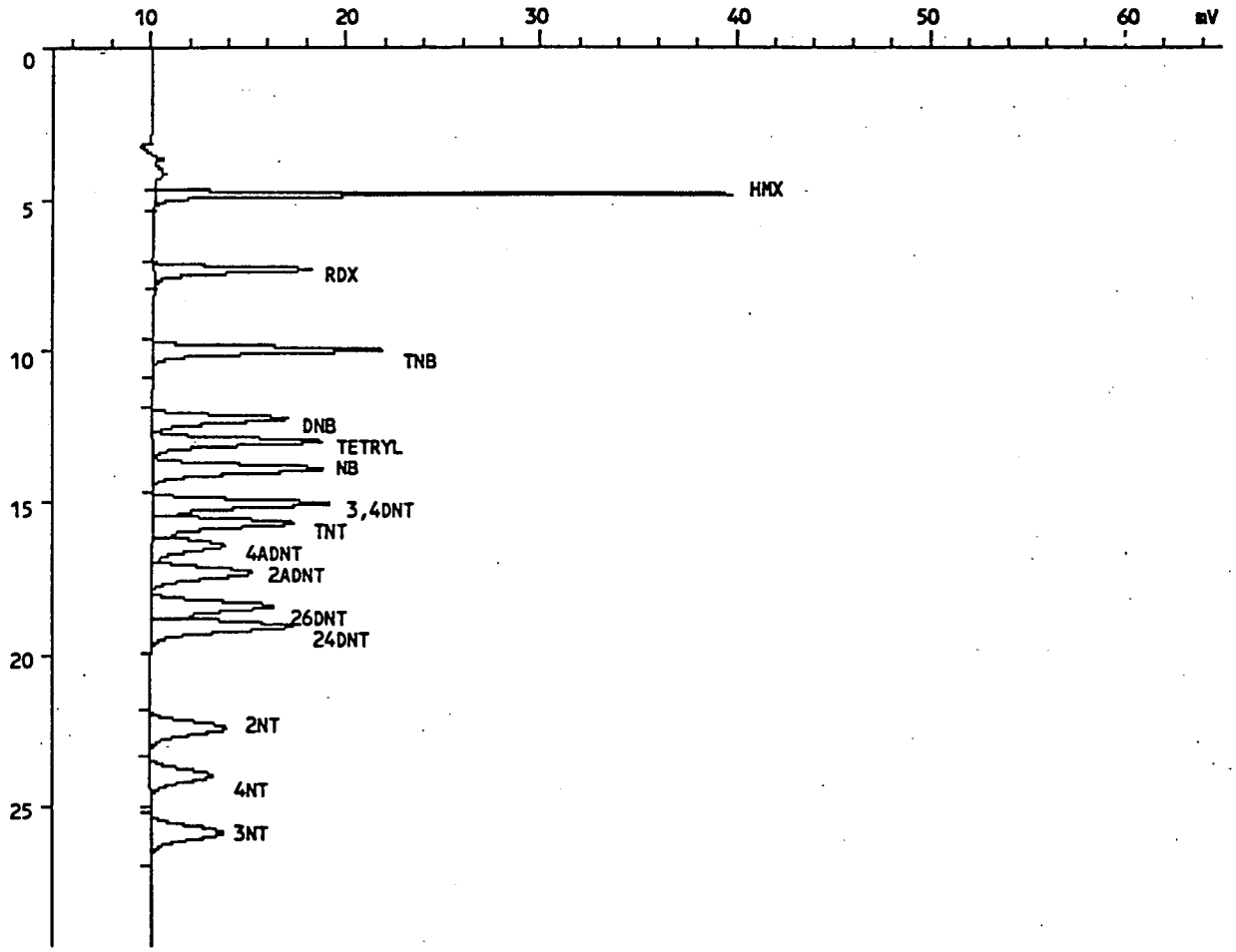
Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
11	18.384	6406	156959	1.291	26DNT
12	19.045	7375	197328	0.786	24DNT
13	22.373	3955	116837	1.126	2NT
14	23.920	3233	100123	1.189	4NT
15	25.781	3682	121243	1.090	3NT
Total		125092	2326337	18.459	
Residual		0	0	0.000	

LONG PLOT

Injection F: <MC3> 5 5EX1021E,12,1

Sample name.....: EXP L3 SOIL STD
Sample ID.....: 1-127:CA9
INST ID: IN13, COL. TYPE: ODS-II, ID: CARB-05, INJ:200,COL#1

Acquired on 25-Oct-97 at 02:19:19
Reported on 30-Oct-97 at 17:15:52



INJECTION REPORT

Injection F: <MC3> 5 5EX1021E,12,1

Acquired on 25-Oct-97 at 02:19:19
Modified on 30-Oct-81 at 17:15:24
Reported on 30-Oct-97 at 17:15:26

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN13, COL. TYPE: ODS-II, ID: CARB-05, INJ:200,COL#1
Number of samples.: 34
Calibration file..: 5EX1021 Last modified on 30-Oct-81 at 16:00:08
Method file.....: EXPLOS Last modified on 30-Oct-81 at 16:03:44
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: EXP L3 SOIL STD
Sample ID.....: 1-127:CA9
Sample type.....: Standard
Sample amount.....: 1.0000
Number of injections.....: 1
Bottle Number.....: 1

Retention time update.....: No update
Calibration level identifier.....: 3
Response factor update.....: No update

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 1.000
Dilution.....: 1.000
Amount (g or mL).....: 1000.000

PEAK INFORMATION

Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
1	4.853	29552	231926	3.401	HMX
2	7.365	8103	122232	1.441	RDX
3	9.984	11767	190518	1.018	TNB
4	12.240	6907	134474	0.543	DNB
5	12.992	8761	169742	1.252	TETRYL
6	13.915	8799	187961	1.111	NB
7	15.077	9050	188028	1.688	3,4DNT
8	15.733	7332	162657	0.893	TNT
9	16.437	3806	98120	0.787	4ADNT
10	17.333	5213	139272	0.777	2ADNT

Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
11	18.427	6304	154581	1.271	26DNT
12	19.088	7276	194755	0.775	24DNT
13	22.432	3949	117474	1.132	2NT
14	23.984	3209	99515	1.182	4NT
15	25.845	3678	121220	1.090	3NT
Total		123704	2312477	18.361	
Residual		0	0	0.000	

CALIBRATION FACTOR DATA
FORM 9A-1

EXPLOSIVE CALIBRATION - METHOD SW846-8330
Instrument ID: IN12
umn ID : CARB-04

Inj 1

Injection File Name Level 1 : 6EX1104,4
Injection File Name Level 2 : 6EX1104,5
Injection File Name Level 3 : 6EX1104,6
Injection File Name Level 4 : 6EX1104,7
Injection File Name Level 5 : 6EX1104,8

Calibration Date : 11/04/97
Number of Calibration Levels: 5
Line forced thru zero : Yes
Calculation Method : Area

COMPONENT NAME	Area Level 1	CAL FACTOR Level 1	Area Level 2	CAL FACTOR Level 2	Area Level 3	CAL FACTOR Level 3	Area Level 4	CAL FACTOR Level 4	Area Level 5	CAL FACTOR Level 5
HMX	9542	941	276353	1088	675071	1023	1089259	1100	1439765	1091
RDX	6149	1457	153730	1456	381602	1383	625447	1518	822226	1495
TNB	8501	2815	244465	3234	602202	3057	984023	3324	1276549	3240
DNB	7080	4370	183368	4516	451306	4274	738895	4665	968980	4571
TETRYL	8285	2227	217102	2334	532097	2199	856941	2354	1111653	2297
NB	11753	3562	303364	3682	736467	3441	1210409	3759	1601142	3724
3,4DNT	9541	1878	262266	2068	652380	1977	1058405	2134	1384670	2098
TNT	8487	3143	233707	3457	570915	3244	947417	3589	1242940	3531
4ADNT	5331	2259	138036	2332	339091	2202	557130	2401	726597	2359
2ADNT	7682	3255	199147	3364	491398	3191	809499	3489	1056377	3430
26DNT	7648	1971	202267	2081	502357	1978	815958	2147	1061298	2097
24DNT	10143	4298	289248	4886	708249	4599	1191897	5137	1555708	5051
2NT	6538	1934	176048	2081	431514	1961	698570	2117	932176	2119
[illegible]	5656	1673	162625	1922	403595	1835	648164	1964	874169	1987
	6495	2017	193258	2404	475185	2263	768000	2446	1030247	2465

CALIBRATION FACTOR DATA
FORM 9B

EXPLOSIVE CALIBRATION - METHOD SW846-8330
Instrument ID: IN12
Column ID : CARB-04

Inj 1

Injection File Name Level 1 : 6EX1104,4
Injection File Name Level 2 : 6EX1104,5
Injection File Name Level 3 : 6EX1104,6
Injection File Name Level 4 : 6EX1104,7
Injection File Name Level 5 : 6EX1104,8

Calibration Date : 11/04/97
Number of Calibration Levels: 5
Line forced thru zero : Yes
Calculation Method : Area

Component Name	Mean Cal Factor	% RSD	Corr. Coeff.	Slope area/amt	Intercept
HMX	1049	6.44	0.99936	1085	0.000
RDX	1462	3.52	0.99901	1487	0.000
TNB	3134	6.48	0.99919	3243	0.000
DNB	4479	3.50	0.99914	4561	0.000
TETRYL	2282	2.95	0.99942	2303	0.000
NB	3633	3.60	0.99908	3698	0.000
3,4DNT	2031	5.09	0.99934	2093	0.000
TNT	3393	5.63	0.99886	3512	0.000
[illegible]NT	2311	3.46	0.99915	2352	0.000
[illegible]DNT	3346	3.67	0.99910	3417	0.000
26DNT	2055	3.77	0.99923	2097	0.000
24DNT	4794	7.20	0.99865	5019	0.000
2NT	2042	4.32	0.99923	2097	0.000
4NT	1876	6.79	0.99926	1959	0.000
3NT	2319	8.03	0.99912	2432	0.000

CALIBRATION FACTOR DATA
FORM 9C

Initial Calibration Date: 11/04/97
Continuing Calibration #: 4
Continuing Cal Date : 11/06/97
Continuing Cal Level : 3
Instrument ID : IN12
Column ID : CARB-04
Injection File Name : 6EX1104A,4
Calculation Mode : Area

Component Name	Response Cont Std	Cont CF	Init CF Mean	%D	Init CF Slope	%D
HMX	653917	991	1049	5.51	1085	8.68
RDX	371565	1346	1462	7.90	1487	9.47
TNB	584944	2969	3134	5.26	3243	8.44
DNB	438162	4149	4479	7.37	4561	9.03
TETRYL	504927	2086	2282	8.58	2303	9.40
NB	707056	3304	3633	9.07	3698	10.67
3,4DNT	634922	1924	2031	5.27	2093	8.08
TNT	553754	3146	3393	7.27	3512	10.40
4ADNT	329783	2141	2311	7.32	2352	8.95
2ADNT	478337	3106	3346	7.16	3417	9.10
26DNT	485033	1910	2055	7.07	2097	8.95
24DNT	687523	4464	4794	6.88	5019	11.05
2NT	415304	1888	2042	7.57	2097	10.00
4NT	390030	1773	1876	5.51	1959	9.52
[illegible]T	458297	2182	2319	5.88	2432	10.27

CALIBRATION FACTOR DATA
FORM 9C

Initial Calibration Date: 11/04/97
Continuing Calibration #: 5
Continuing Cal Date : 11/07/97
Continuing Cal Level : 3
Instrument ID : IN12
Column ID : CARB-04
Injection File Name : 6EX1104B,11
Calculation Mode : Area

Component Name	Response Cont Std	Cont CF	Init CF Mean	%D	Init CF Slope	%D
HMX	715506	1084	1049	3.39	1085	0.08
RDX	408934	1482	1462	1.36	1487	0.37
TNB	635544	3226	3134	2.94	3243	0.52
DNB	485973	4602	4479	2.74	4561	0.89
TETRYL	558094	2306	2282	1.05	2303	0.14
NB	748362	3497	3633	3.75	3698	5.45
3,4DNT	702595	2129	2031	4.83	2093	1.72
TNT	612059	3478	3393	2.50	3512	0.97
4ADNT	363900	2363	2311	2.27	2352	0.47
2ADNT	527311	3424	3346	2.34	3417	0.21
26DNT	541315	2131	2055	3.71	2097	1.62
24DNT	754956	4902	4794	2.25	5019	2.32
2NT	437752	1990	2042	2.58	2097	5.13
4NT	407216	1851	1876	1.34	1959	5.53
[illegible]NT	477343	2273	2319	1.97	2432	6.54

CALIBRATION FACTOR DATA
FORM 9D

EXPLOSIVE CALIBRATION - METHOD SW846-8330

ection volume = 200uL

Amount Units = ng

COMPONENT NAME	AMT ON COL Level 1	AMT ON COL Level 2	AMT ON COL Level 3	AMT ON COL Level 4	AMT ON COL Level 5
HMX	10.140	254.000	660.000	990.000	1320.000
RDX	4.220	105.600	276.000	412.000	550.000
TNB	3.020	75.600	197.000	296.000	394.000
DNB	1.620	40.600	105.600	158.400	212.000
TETRYL	3.720	93.000	242.000	364.000	484.000
NB	3.300	82.400	214.000	322.000	430.000
3,4DNT	5.080	126.800	330.000	496.000	660.000
TNT	2.700	67.600	176.000	264.000	352.000
4ADNT	2.360	59.200	154.000	232.000	308.000
2ADNT	2.360	59.200	154.000	232.000	308.000
26DNT	3.880	97.200	254.000	380.000	506.000
24DNT	2.360	59.200	154.000	232.000	308.000
2NT	3.380	84.600	220.000	330.000	440.000
4NT	3.380	84.600	220.000	330.000	440.000
3NT	3.220	80.400	210.000	314.000	418.000

CALIBRATION FACTOR DATA
FORM 9E

EXPLOSIVE CALIBRATION - METHOD SW846-8330

Instrument ID : IN12
Column ID : CARB-04
Calibration Date : 11/04/97
Number of Calibration Levels: 5

COMPONENT NAME	RT Level 1	RT Level 2	RT Level 3	RT Level 4	RT Level 5
HMX	5.44	5.44	5.41	5.41	5.40
RDX	8.30	8.28	8.26	8.23	8.23
TNB	11.12	11.10	11.08	11.06	11.05
DNB	13.65	13.62	13.60	13.58	13.57
TETRYL	14.64	14.61	14.58	14.57	14.56
NB	15.46	15.42	15.40	15.38	15.37
3,4DNT	16.87	16.83	16.80	16.79	16.77
TNT	17.50	17.48	17.46	17.45	17.44
4ADNT	18.45	18.42	18.40	18.40	18.37
2ADNT	19.52	19.46	19.44	19.44	19.41
26DNT	20.57	20.51	20.49	20.49	20.46
24DNT	21.25	21.25	21.23	21.22	21.20
2NT	24.89	24.88	24.86	24.85	24.83
4NT	26.59	26.60	26.58	26.58	26.54
3NT	28.67	28.69	28.66	28.66	28.62

COMPONENT NAME	RT Cont CA1	RT Cont CA2	RT Cont CA3	RT Cont CA4	RT Cont CA5
HMX	5.41	5.40	5.41	5.40	5.41
RDX	8.25	8.23	8.23	8.23	8.26
TNB	11.07	11.05	11.05	11.05	11.08
DNB	13.58	13.55	13.55	13.56	13.60
TETRYL	14.57	14.52	14.52	14.52	14.58
NB	15.38	15.35	15.34	15.35	15.41
3,4DNT	16.78	16.73	16.73	16.73	16.80
TNT	17.45	17.40	17.40	17.40	17.47
4ADNT	18.38	18.33	18.31	18.31	18.43
2ADNT	19.41	19.36	19.34	19.35	19.46
26DNT	20.46	20.41	20.40	20.41	20.50
24DNT	21.20	21.15	21.13	21.13	21.24
2NT	24.82	24.77	24.75	24.75	24.87
4NT	26.54	26.49	26.45	26.47	26.60
3NT	28.61	28.56	28.53	28.52	28.67

COMPONENT NAME	AVE RT	STD_DEV	3X SD	AVE+-3X SD
HMX	5.41	0.014	0.041	5.37 - 5.45
RDX	8.25	0.025	0.074	8.18 - 8.32
TNB	11.07	0.024	0.072	11.00 - 11.14
DNB	13.58	0.033	0.099	13.48 - 13.68
TETRYL	14.56	0.039	0.118	14.44 - 14.68
NB	15.38	0.036	0.108	15.27 - 15.49
3,4DNT	16.78	0.047	0.142	16.63 - 16.92
TNT	17.44	0.037	0.110	17.33 - 17.55

4ADNT	18.37	0.052	0.155	18.22 - 18.53
2ADNT	19.41	0.059	0.177	19.24 - 19.59
26DNT	20.46	0.054	0.163	20.30 - 20.63
24DNT	21.19	0.050	0.150	21.04 - 21.34
2NT	24.82	0.053	0.158	24.66 - 24.98
4NT	26.54	0.057	0.172	26.37 - 26.71
[illegible]	28.61	0.064	0.192	28.42 - 28.80

CALIBRATION FACTOR DAT
FORM 9E

EXPLOSIVE CALIBRATION - METHOD SW846-8330

Instrument ID : IN12
Column ID : CARB-04
Calibration Date : 11/04/97
Number of Calibration Levels: 5

COMPONENT NAME	RT Level 1	RT Level 2	RT Level 3	RT Level 4	RT Level 5
HMX	5.44	5.44	5.41	5.41	5.40
RDX	8.30	8.28	8.26	8.23	8.23
TNB	11.12	11.10	11.08	11.06	11.05
DNB	13.65	13.62	13.60	13.58	13.57
TETRYL	14.64	14.61	14.58	14.57	14.56
NB	15.46	15.42	15.40	15.38	15.37
3,4DNT	16.87	16.83	16.80	16.79	16.77
TNT	17.50	17.48	17.46	17.45	17.44
4ADNT	18.45	18.42	18.40	18.40	18.37
2ADNT	19.52	19.46	19.44	19.44	19.41
26DNT	20.57	20.51	20.49	20.49	20.46
24DNT	21.25	21.25	21.23	21.22	21.20
2NT	24.89	24.88	24.86	24.85	24.83
4NT	26.59	26.60	26.58	26.58	26.54
3NT	28.67	28.69	28.66	28.66	28.62

COMPONENT NAME	RT Cont CA6
HMX	5.40
RDX	8.22
TNB	11.05
DNB	13.55
TETRYL	14.52
NB	15.34
3,4DNT	16.73
TNT	17.40
4ADNT	18.31
2ADNT	19.34
26DNT	20.41
24DNT	21.14
2NT	24.76
4NT	26.48
3NT	28.54

COMPONENT NAME	AVE RT	STD_DEV	3X SD	AVE+-3X SD
HMX	5.41	0.014	0.041	5.37 - 5.45
RDX	8.25	0.025	0.074	8.18 - 8.32
TNB	11.07	0.024	0.072	11.00 - 11.14
DNB	13.58	0.033	0.099	13.48 - 13.68
TETRYL	14.56	0.039	0.118	14.44 - 14.68
NB	15.38	0.036	0.108	15.27 - 15.49
3,4DNT	16.78	0.047	0.142	16.63 - 16.92
TNT	17.44	0.037	0.110	17.33 - 17.55
4ADNT	18.37	0.052	0.155	18.22 - 18.53

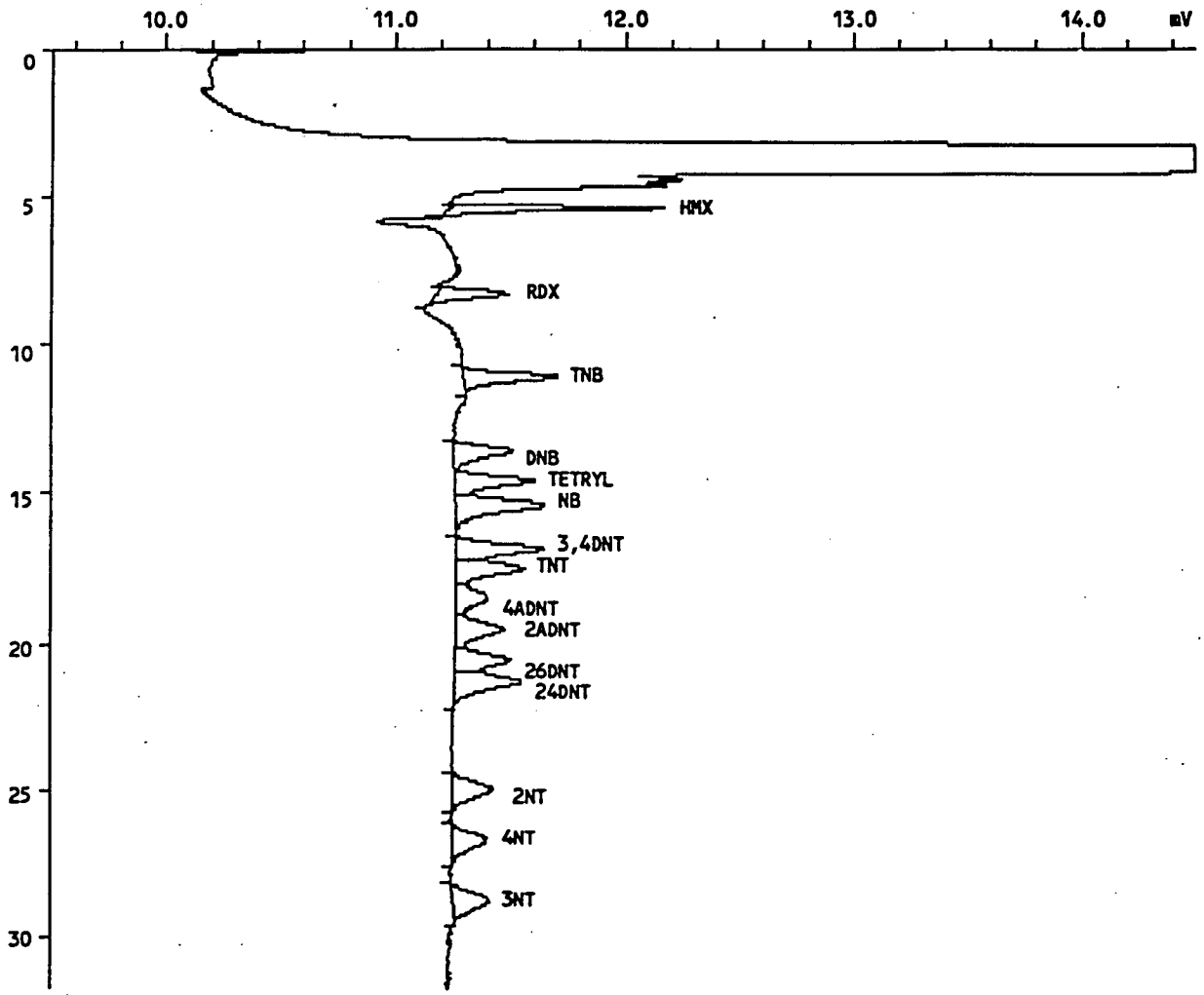
2ADNT	19.41	0.059	0.177	19.24 - 19.59
26DNT	20.46	0.054	0.163	20.30 - 20.63
24DNT	21.19	0.050	0.150	21.04 - 21.34
2NT	24.82	0.053	0.158	24.66 - 24.98
4NT	26.54	0.057	0.172	26.37 - 26.71
3NT	28.61	0.064	0.192	28.42 - 28.80

LONG PLOT

Injection F: <MC3> 6 6EX1104,4,1

Sample name.....: EXP L1 SOIL STD
Sample ID.....: 1-129:IA1
INST ID: IN12, COL. TYPE: ODS-II, ID: CARB-04, INJ:200,COL#1

Acquired on 04-Nov-97 at 20:55:55
Reported on 18-Nov-97 at 11:48:31



INJECTION REPORT

Injection F: <MC3> 6 6EX1104,4,1

Acquired on 04-Nov-97 at 20:55:55
Modified on 18-Nov-81 at 11:42:30
Reported on 18-Nov-97 at 11:43:01

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN12, COL. TYPE: ODS-II, ID: CARB-04, INJ:200,COL#1
Number of samples.: 55
Calibration file..: 6EX1104 Last modified on 18-Nov-81 at 11:42:30
Method file.....: EXPLOS Last modified on 18-Nov-81 at 11:42:48
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: EXP L1 SOIL STD
Sample ID.....: 1-129:IA1
Sample type.....: Standard
Sample amount.....: 1.0000
Number of injections.....: 1
Bottle Number.....: 1

Retention time update.....: No update
Calibration level identifier.....: 1
Response factor update.....: Initialise

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 1.000
Dilution.....: 1.000
Amount (g or mL).....: 1.000

PEAK INFORMATION

Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
1	5.440	946	9542	0.044	HMX
2	8.304	321	6149	0.021	RDX
3	11.120	419	8501	0.013	TNB
4	13.653	257	7080	7.767E-003	DNB
5	14.635	347	8285	0.018	TETRYL
6	15.456	395	11753	0.016	NB
7	16.869	382	9541	0.023	3,4DNT
8	17.499	304	8487	0.012	TNT
9	18.448	141	5331	0.013	4ADNT
10	19.520	214	7682	0.011	2ADNT

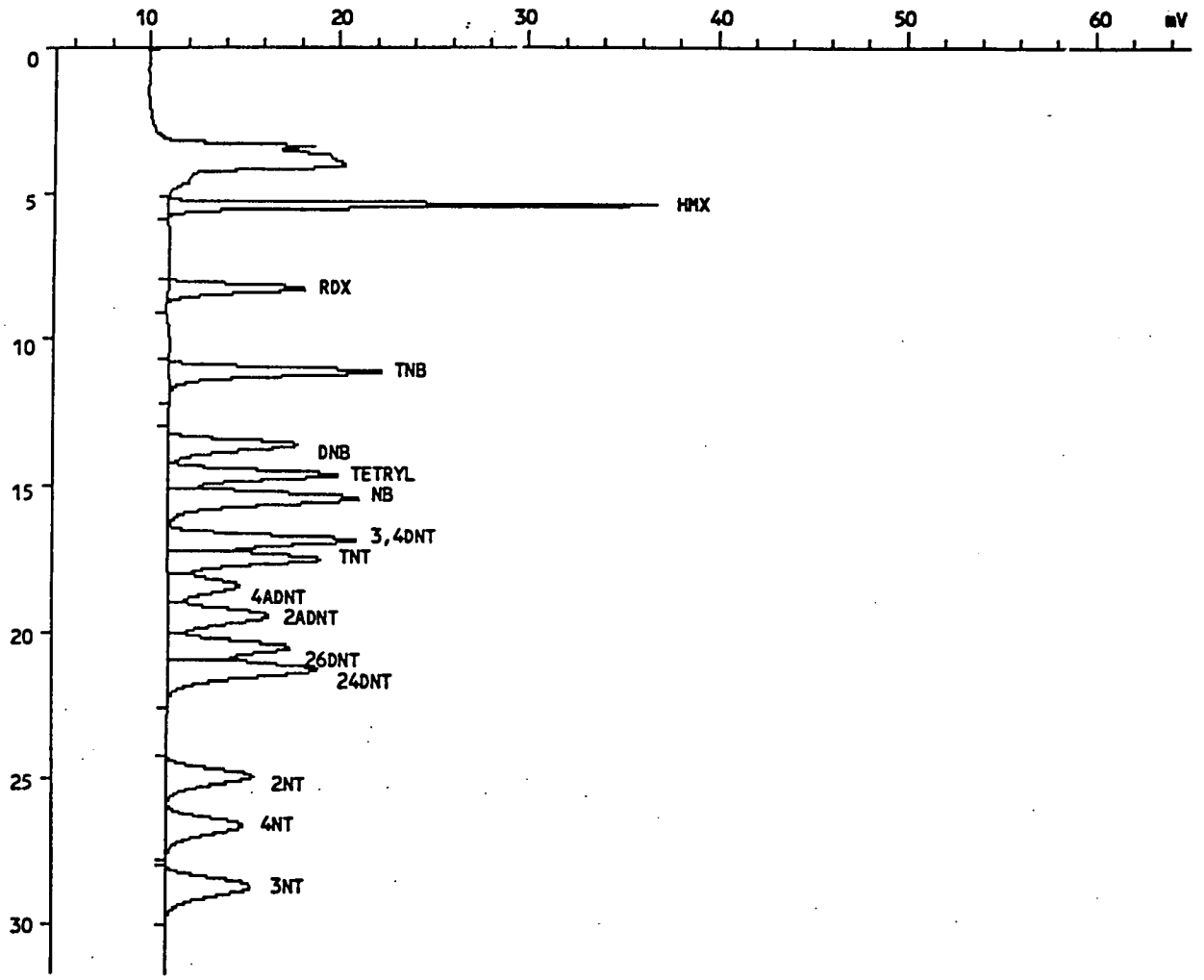
Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
11	20.565	244	7648	0.018	26DNT
12	21.253	296	10143	0.010	24DNT
13	24.885	176	6538	0.016	2NT
14	26.592	148	5656	0.014	4NT
15	28.672	163	6495	0.013	3NT
Total		4753	118830	0.250	
Residual		0	0	0.000	

LONG PLOT

Injection F: <MC3> 6 6EX1104,5,1

Sample name.....: EXP L2 SOIL STD
Sample ID.....: 1-128:IA2
INST ID: IN12, COL. TYPE: ODS-II, ID: CARB-04, INJ:200,COL#1

Acquired on 04-Nov-97 at 21:40:33
Reported on 18-Nov-97 at 11:47:39



INJECTION REPORT

Injection F: <MC3> 6 6EX1104,5,1

Acquired on 04-Nov-97 at 21:40:33
Modified on 18-Nov-81 at 11:42:26
Reported on 18-Nov-97 at 11:43:24

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN12, COL. TYPE: ODS-II, ID: CARB-04, INJ:200,COL#1
Number of samples.: 55
Calibration file..: 6EX1104 Last modified on 18-Nov-81 at 11:42:30
Method file.....: EXPLOS Last modified on 18-Nov-81 at 11:42:48
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: EXP L2 SOIL STD
Sample ID.....: 1-128:IA2
Sample type.....: Standard
Sample amount.....: 1.0000
Number of injections.....: 1
Bottle Number.....: 1

Retention time update.....: No update
Calibration level identifier.....: 2
Response factor update.....: Initialise

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 1.000
Dilution.....: 1.000
Amount (g or mL).....: 1.000

PEAK INFORMATION

Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
1	5.440	25833	276353	1.274	HMX
2	8.277	7190	153730	0.517	RDX
3	11.099	11270	244465	0.377	TNB
4	13.621	6824	183368	0.201	DNB
5	14.608	8926	217102	0.472	TETRYL
6	15.419	10021	303364	0.410	NB
7	16.827	9964	262266	0.627	3,4DNT
8	17.483	8076	233707	0.333	TNT
9	18.421	3805	138036	0.335	4ADNT
10	19.461	5320	199147	0.292	2ADNT

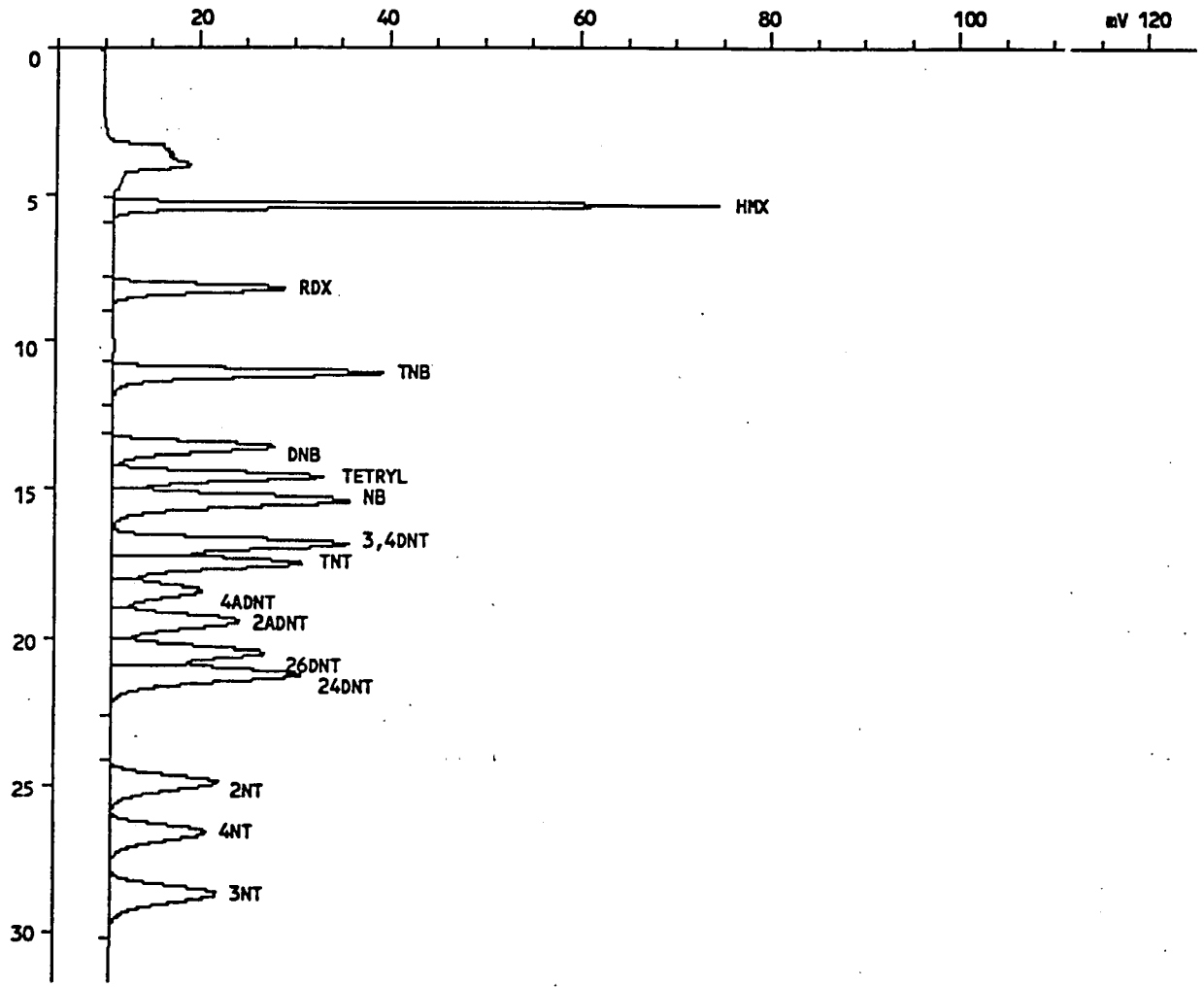
Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
11	20.512	6443	202267	0.483	26DNT
12	21.253	7911	289248	0.288	24DNT
13	24.875	4552	176048	0.420	2NT
14	26.603	4049	162625	0.415	4NT
15	28.688	4497	193258	0.398	3NT
Total		124682	3234985	6.842	
Residual		0	0	0.000	

LONG PLOT

Injection F: <MC3> 6 6EX1104,6,1

Sample name.....: EXP L3 SOIL STD
Sample ID.....: 1-127:IA3
INST ID: IN12, COL. TYPE: ODS-II, ID: CARB-04, INJ:200,COL#1

Acquired on 04-Nov-97 at 22:25:11
Reported on 18-Nov-97 at 11:47:00



INJECTION REPORT

Injection F: <MC3> 6 6EX1104,6,1

Acquired on 04-Nov-97 at 22:25:11
Modified on 18-Nov-81 at 11:42:20
Reported on 18-Nov-97 at 11:43:47

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN12, COL. TYPE: ODS-II, ID: CARB-04, INJ:200,COL#1
Number of samples.: 55
Calibration file..: 6EX1104 Last modified on 18-Nov-81 at 11:42:30
Method file.....: EXPLOS Last modified on 18-Nov-81 at 11:42:48
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: EXP L3 SOIL STD
Sample ID.....: 1-127:IA3
Sample type.....: Standard
Sample amount.....: 1.0000
Number of injections.....: 1
Bottle Number.....: 1

Retention time update.....: No update
Calibration level identifier.....: 3
Response factor update.....: Initialise

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 1.000
Dilution.....: 1.000
Amount (g or mL).....: 1.000

PEAK INFORMATION

Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
1	5.413	63721	675071	3.156	HMX
2	8.256	18111	381602	1.302	RDX
3	11.077	28260	602202	0.942	TNB
4	13.600	17248	451306	0.502	DNB
5	14.581	22056	532097	1.173	TETRYL
6	15.397	24971	736467	1.012	NB
7	16.800	24935	652380	1.582	3,4DNT
8	17.461	20067	570915	0.826	TNT
9	18.400	9572	339091	0.834	4ADNT
10	19.440	13536	491398	0.730	2ADNT

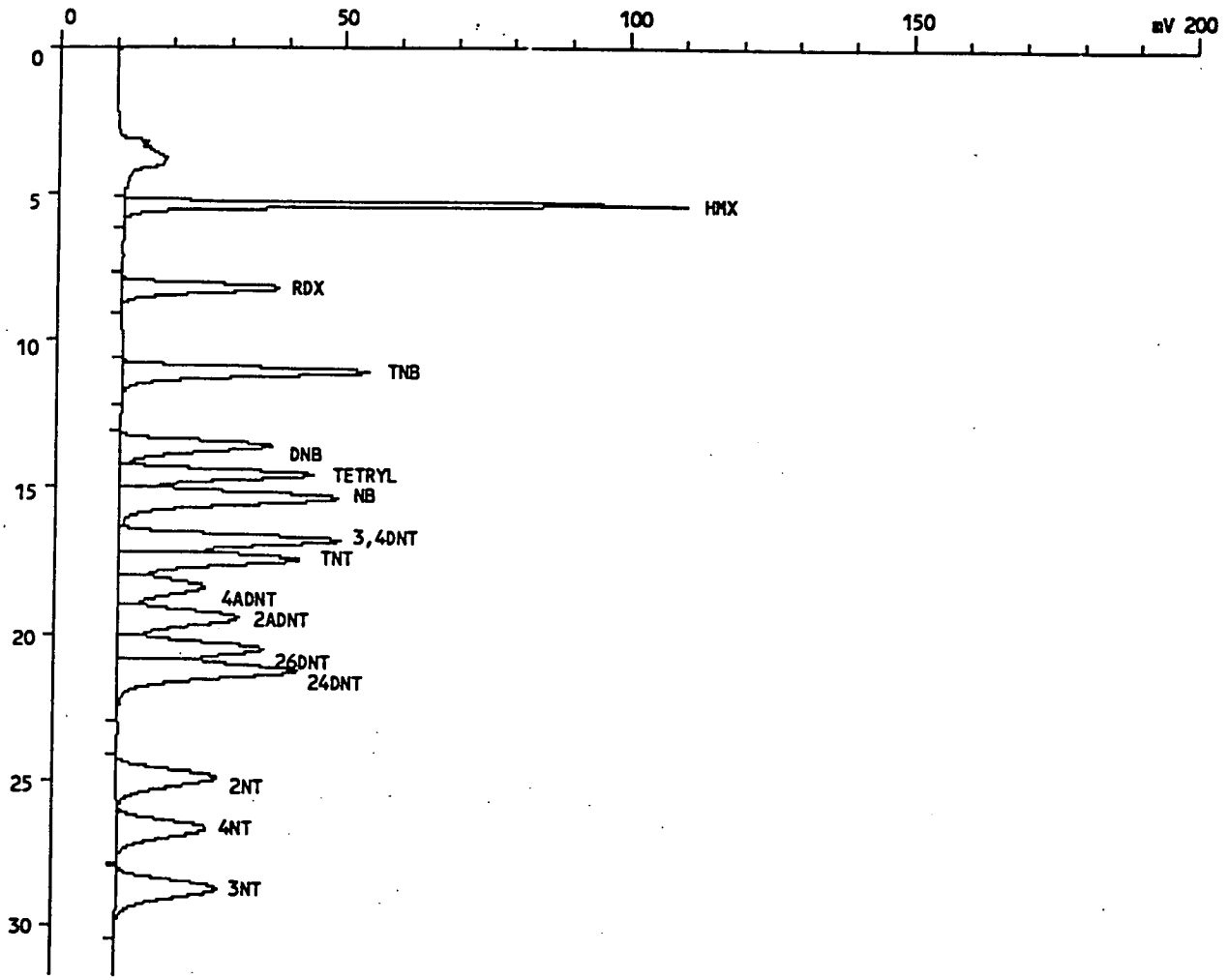
Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
11	20.491	16133	502357	1.216	26DNT
12	21.227	19821	708249	0.717	24DNT
13	24.859	11311	431514	1.045	2NT
14	26.581	10096	403595	1.047	4NT
15	28.656	11205	475185	0.993	3NT
Total		311043	7953429	17.076	
Residual		0	0	0.000	

LONG PLOT

Injection F: <MC3> 6 6EX1104,7,1

Sample name.....: EXP L4 SOIL STD
Sample ID.....: 1-126:IA4
INST ID: IN12, COL. TYPE: ODS-II, ID: CARB-04, INJ:200,COL#1

Acquired on 04-Nov-97 at 23:09:48
Reported on 18-Nov-97 at 11:46:13



INJECTION REPORT

Injection F: <MC3> 6 6EX1104,7,1

Acquired on 04-Nov-97 at 23:09:48
Modified on 18-Nov-81 at 11:42:16
Reported on 18-Nov-97 at 11:44:11

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN12, COL. TYPE: ODS-II, ID: CARB-04, INJ:200,COL#1
Number of samples.: 55
Calibration file..: 6EX1104 Last modified on 18-Nov-81 at 11:42:30
Method file.....: EXPLOS Last modified on 18-Nov-81 at 11:42:48
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: EXP L4 SOIL STD
Sample ID.....: 1-126:IA4
Sample type.....: Standard
Sample amount.....: 1.0000
Number of injections.....: 1
Bottle Number.....: 1

Retention time update.....: No update
Calibration level identifier.....: 4
Response factor update.....: Initialise

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 1.000
Dilution.....: 1.000
Amount (g or mL).....: 1.000

PEAK INFORMATION

Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
1	5.408	98946	1089259	5.554	HMX
2	8.235	27624	625447	2.334	RDX
3	11.061	43387	984023	1.686	TNB
4	13.579	26471	738895	0.902	DNB
5	14.571	33839	856941	2.065	TETRYL
6	15.381	38285	1210409	1.835	NB
7	16.789	38644	1058405	2.822	3,4DNT
8	17.451	31567	947417	1.502	TNT
9	18.395	14847	557130	1.485	4ADNT
10	19.440	20881	809499	1.318	2ADNT

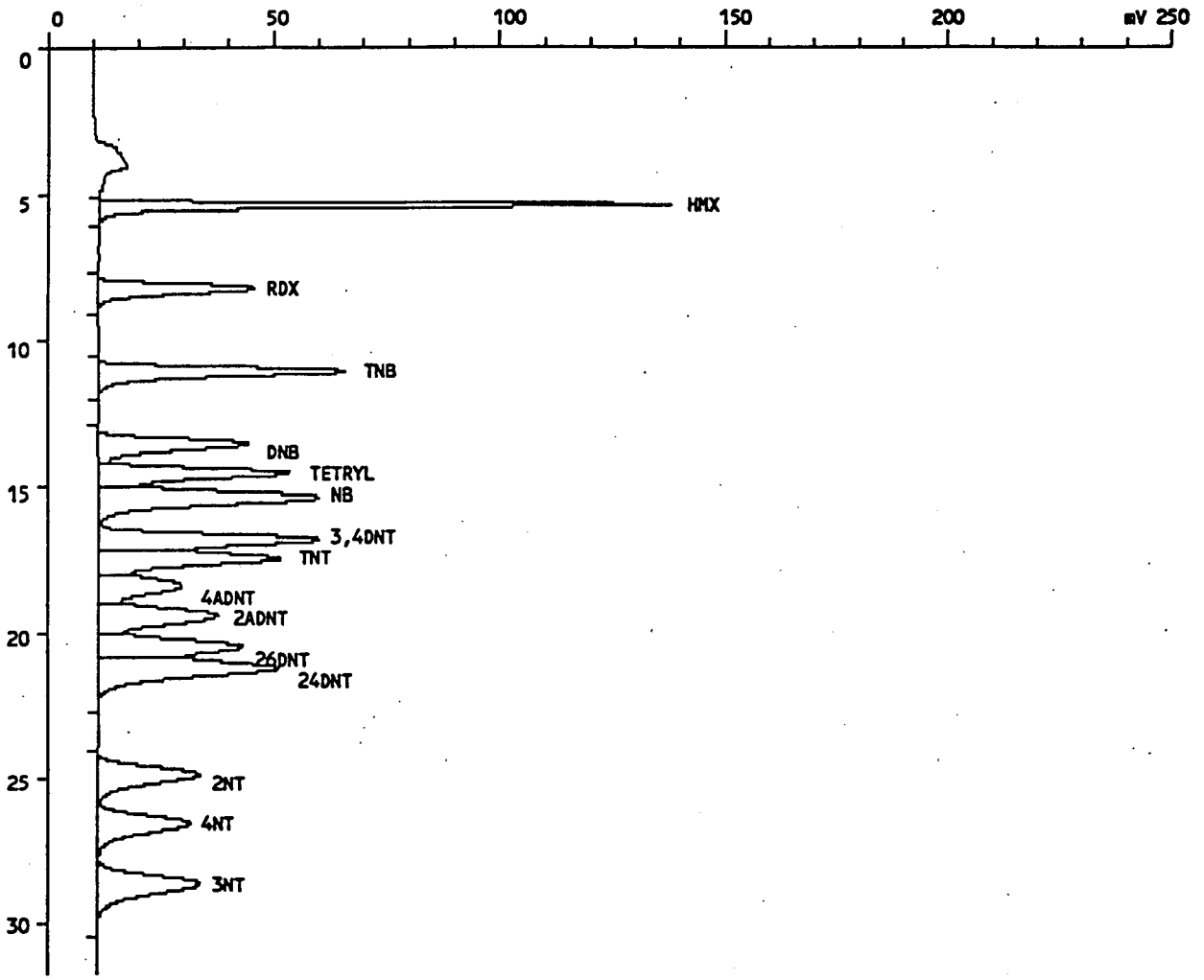
Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
11	20.485	25156	815958	2.165	26DNT
12	21.221	31135	1191897	1.324	24DNT
13	24.853	17532	698570	1.866	2NT
14	26.576	15724	648164	1.861	4NT
15	28.661	17468	768000	1.774	3NT
Total		481504	13000012	30.495	
Residual		0	0	0.000	

LONG PLOT

Injection F: <MC3> 6 6EX1104,8,1

Sample name.....: EXP L5 SOIL STD
Sample ID.....: 1-125:IA5
INST ID: IN12, COL. TYPE: ODS-II, ID: CARB-04, INJ:200,COL#1

Acquired on 04-Nov-97 at 23:54:26
Reported on 18-Nov-97 at 11:45:34



INJECTION REPORT

Injection F: <MC3> 6 6EX1104,8,1

Acquired on 04-Nov-97 at 23:54:26
Modified on 18-Nov-81 at 11:42:12
Reported on 18-Nov-97 at 11:44:35

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN12, COL. TYPE: ODS-II, ID: CARB-04, INJ:200,COL#1
Number of samples.: 55
Calibration file..: 6EX1104 Last modified on 18-Nov-81 at 11:42:30
Method file.....: EXPLOS Last modified on 18-Nov-81 at 11:42:48
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: EXP L5 SOIL STD
Sample ID.....: 1-125:IA5
Sample type.....: Standard
Sample amount.....: 1.0000
Number of injections.....: 1
Bottle Number.....: 1

Retention time update.....: No update
Calibration level identifier.....: 5
Response factor update.....: Initialise

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 1.000
Dilution.....: 1.000
Amount (g or mL).....: 1.000

PEAK INFORMATION

Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
1	5.403	126902	1439765	9.510	HMX
2	8.229	34708	822226	4.022	RDX
3	11.051	54886	1276549	2.889	TNB
4	13.573	33645	968980	1.573	DNB
5	14.555	42610	1111653	3.503	TETRYL
6	15.371	49252	1601142	3.298	NB
7	16.773	49382	1384670	4.925	3,4DNT
8	17.435	40508	1242940	2.631	TNT
9	18.373	18844	726597	2.774	4ADNT
10	19.413	26531	1056377	2.293	2ADNT

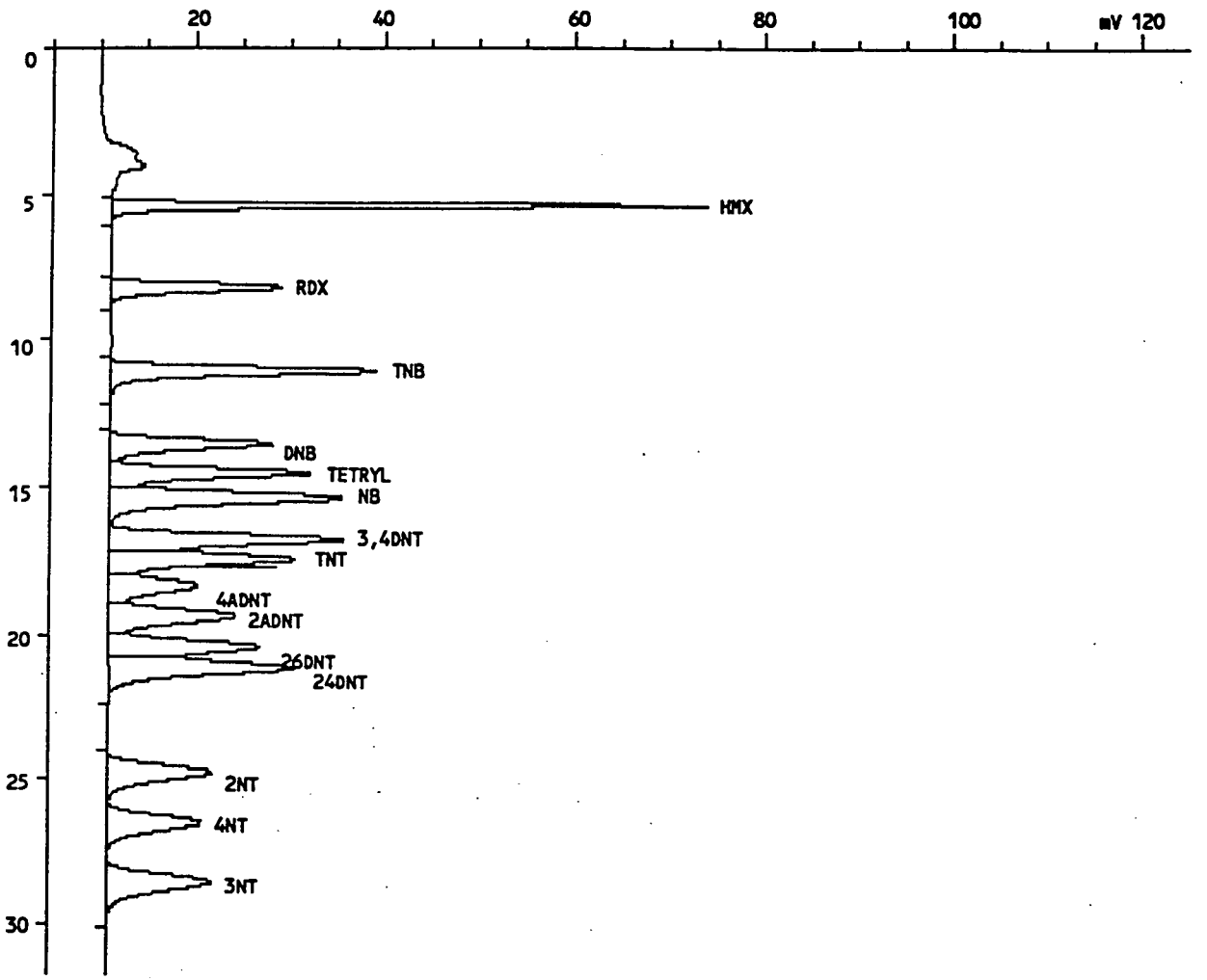
Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
11	20.464	32129	1061298	3.724	26DNT
12	21.195	39983	1555708	2.336	24DNT
13	24.827	22812	932176	3.349	2NT
14	26.544	20546	874169	3.404	4NT
15	28.624	22829	1030247	3.224	3NT
Total		615567	17084498	53.456	
Residual		0	0	0.000	

LONG PLOT

Injection F: <MC3> 6 6EX1104A,4,1

Sample name.....: EXP L3 SOIL STD
Sample ID.....: 1-127:CA4
INST ID: IN12, COL. TYPE: ODS-II, ID: CARB-04, INJ:200,COL#1

Acquired on 06-Nov-97 at 14:40:52
Reported on 18-Nov-97 at 12:17:22



INJECTION REPORT

Injection F: <MC3> 6 6EX1104A,4,1

Acquired on 06-Nov-97 at 14:40:52
Modified on 18-Nov-81 at 12:16:38
Reported on 18-Nov-97 at 12:16:42

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN12, COL. TYPE: ODS-II, ID: CARB-04, INJ:200,COL#1
Number of samples.: 5
Calibration file..: 6EX1104 Last modified on 18-Nov-81 at 11:55:16
Method file.....: EXPLOS Last modified on 18-Nov-81 at 11:57:46
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: EXP L3 SOIL STD
Sample ID.....: 1-127:CA4
Sample type.....: Standard
Sample amount.....: 1.0000
Number of injections.....: 1
Bottle Number.....: 1

Retention time update.....: No update
Calibration level identifier.....: 3
Response factor update.....: No update

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 1.000
Dilution.....: 1.000
Amount (g or mL).....: 1000.000

PEAK INFORMATION

Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
1	5.403	62712	653917	3.117	HMX
2	8.229	18018	371565	1.271	RDX
3	11.051	27918	584944	0.933	TNB
4	13.557	17087	438162	0.489	DNB
5	14.517	21172	504927	1.106	TETRYL
6	15.349	24512	707056	0.973	NB
7	16.725	24603	634922	1.563	3,4DNT
8	17.403	19622	553754	0.816	TNT
9	18.309	9490	329783	0.714	4ADNT
10	19.349	13420	478337	0.715	2ADNT

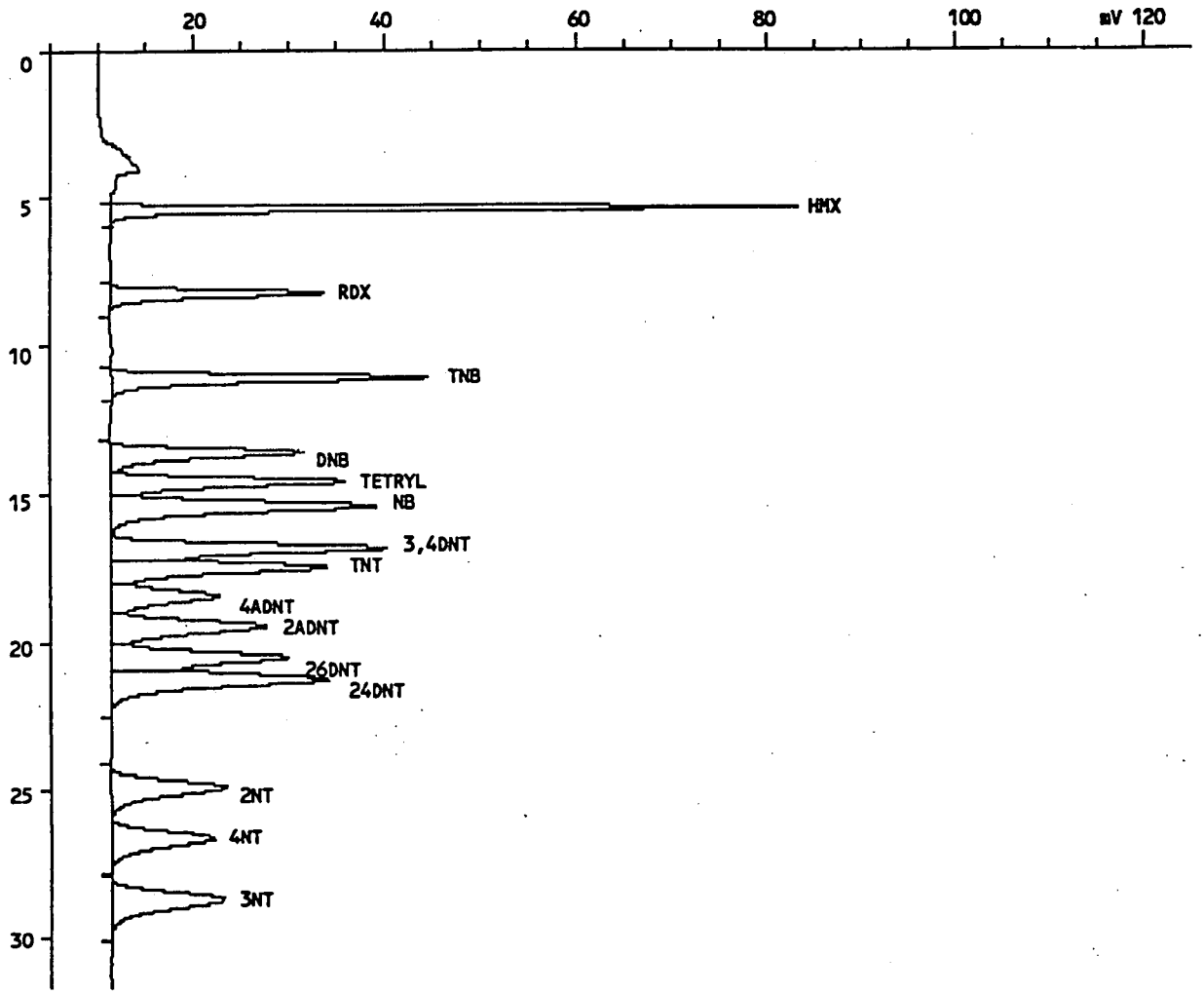
Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
11	20.405	15906	485033	1.180	26DNT
12	21.131	19574	687523	0.717	24DNT
13	24.752	11052	415304	1.017	2NT
14	26.469	9883	390030	1.040	4NT
15	28.523	10930	458297	0.988	3NT
Total		305898	7693551	16.639	
Residual		0	0	0.000	

LONG PLOT

Injection F: <MC3> 6 6EX1104B,11,1

Sample name.....: EXP L3 SOIL STD
Sample ID.....: 1-127:CA5
INST ID: IN12, COL. TYPE: ODS-II, ID: CARB-04, INJ:200,COL#1

Acquired on 07-Nov-97 at 01:49:47
Reported on 18-Nov-97 at 12:18:40



INJECTION REPORT

Injection F: <MC3> 6 6EX1104B,11,1

Acquired on 07-Nov-97 at 01:49:47
Modified on 18-Nov-81 at 12:18:02
Reported on 18-Nov-97 at 12:18:14

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN12, COL. TYPE: ODS-II, ID: CARB-04, INJ:200,COL#1
Number of samples.: 40
Calibration file..: 6EX1104 Last modified on 18-Nov-81 at 11:55:16
Method file.....: EXPLOS Last modified on 18-Nov-81 at 11:57:46
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: EXP L3 SOIL STD
Sample ID.....: 1-127:CA5
Sample type.....: Standard
Sample amount.....: 1.0000
Number of injections.....: 1
Bottle Number.....: 1

Retention time update.....: No update
Calibration level identifier.....: 3
Response factor update.....: No update

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 1.000
Dilution.....: 1.000
Amount (g or mL).....: 1000.000

PEAK INFORMATION

Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
1	5.413	71853	715506	3.410	HMX
2	8.261	22581	408934	1.399	RDX
3	11.083	32982	635544	1.014	TNB
4	13.600	20428	485973	0.543	DNB
5	14.581	24641	558094	1.223	TETRYL
6	15.408	27727	748362	1.030	NB
7	16.800	28813	702595	1.730	3,4DNT
8	17.472	22718	612059	0.902	TNT
9	18.427	11398	363900	0.787	4ADNT
10	19.461	16234	527311	0.788	2ADNT

Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
11	20.501	18654	541315	1.317	26DNT
12	21.237	22795	754956	0.787	24DNT
13	24.869	12191	437752	1.072	2NT
14	26.597	10827	407216	1.085	4NT
15	28.672	11871	477343	1.029	3NT
Total		355714	8376858	18.116	
Residual		0	0	0.000	

CALIBRATION FACTOR DATA
FORM 9A-1

EXPLOSIVE CALIBRATION - METHOD SW846-8330
Instrument ID: IN12
lumn ID : LC-CN-05

Inj 1

Injection File Name Level 1 : 2CN1024,4
Injection File Name Level 2 : 2CN1024,5
Injection File Name Level 3 : 2CN1024,6
Injection File Name Level 4 : 2CN1024,7
Injection File Name Level 5 : 2CN1024,8

Calibration Date : 10/24/97
Number of Calibration Levels: 5
Line forced thru zero : Yes
Calculation Method : Area

COMPONENT NAME	Area Level 1	CAL FACTOR Level 1	Area Level 2	CAL FACTOR Level 2	Area Level 3	CAL FACTOR Level 3	Area Level 4	CAL FACTOR Level 4	Area Level 5	CAL FACTOR Level 5
TNB	9616	2090	53170	2312	134596	2341	267447	2326	547189	2379
26DNT	8132	1626	42884	1715	108907	1743	214372	1715	436380	1746
TNT	10923	2375	59697	2596	152104	2645	301079	2618	619068	2692
3,4DNT	11396	1628	59281	1694	152184	1845	297288	1802	610956	1851
TETRYL	15151	1683	82281	1828	207087	1841	410955	1826	829280	1843
HMX	4949	707	25975	742	68047	778	134580	769	272134	778

CALIBRATION FACTOR DATA
FORM 9A-1

EXPLOSIVE CALIBRATION - METHOD SW846-8330
Instrument ID: IN12
Column ID : LC-CN-05

Inj 1

Injection File Name Level 1 : 2CN1024,9
Injection File Name Level 2 : 2CN1024,10
Injection File Name Level 3 : 2CN1024,11
Injection File Name Level 4 : 2CN1024,12
Injection File Name Level 5 : 2CN1024,13

Calibration Date : 10/24/97
Number of Calibration Levels: 5
Line forced thru zero : Yes
Calculation Method : Area

COMPONENT NAME	Area Level 1	CAL FACTOR Level 1	Area Level 2	CAL FACTOR Level 2	Area Level 3	CAL FACTOR Level 3	Area Level 4	CAL FACTOR Level 4	Area Level 5	CAL FACTOR Level 5
NB	12662	2532	59601	2384	170369	2726	341307	2730	666417	2666
DNB	14230	3558	65678	3284	192199	3844	388641	3886	762869	3814
24DNT	16899	3380	79418	3177	229940	3679	464892	3719	910618	3642
4ADNT	14145	1538	65542	1425	186226	1619	375137	1631	715160	1555
2ADNT	25109	2511	119869	2397	350542	2804	707856	2831	1396915	2794
RDX	5840	1168	25900	1036	76466	1223	154508	1236	308080	1232

CALIBRATION FACTOR DATA
FORM 9B

EXPLOSIVE CALIBRATION - METHOD SW846-8330
Instrument ID: IN12
lumn ID : LC-CN-05

Inj 1

Injection File Name Level 1 : 2CN1024,4
Injection File Name Level 2 : 2CN1024,5
Injection File Name Level 3 : 2CN1024,6
Injection File Name Level 4 : 2CN1024,7
Injection File Name Level 5 : 2CN1024,8

Calibration Date : 10/24/97
Number of Calibration Levels: 5
Line forced thru zero : Yes
Calculation Method : Area

Component Name	Mean Cal Factor	% RSD	Corr. Coeff.	Slope area/amt	Intercept
TNB	2290	4.98	0.99994	2367	0.000
26DNT	1709	2.83	0.99996	1739	0.000
TNT	2585	4.76	0.99991	2675	0.000
3,4DNT	1764	5.60	0.99990	1841	0.000
TETRYL	1804	3.77	0.99999	1840	0.000
HMX	755	4.02	0.99998	776	0.000

CALIBRATION FACTOR DATA
FORM 9B

EXPLOSIVE CALIBRATION - METHOD SW846-8330
Instrument ID: IN12
Column ID : LC-CN-05

Inj 1

Injection File Name Level 1 : 2CN1024,9
Injection File Name Level 2 : 2CN1024,10
Injection File Name Level 3 : 2CN1024,11
Injection File Name Level 4 : 2CN1024,12
Injection File Name Level 5 : 2CN1024,13

Calibration Date : 10/24/97
Number of Calibration Levels: 5
Line forced thru zero : Yes
Calculation Method : Area

Component Name	Mean Cal Factor	% RSD	Corr. Coeff.	Slope area/amt	Intercept
NB	2608	5.69	0.99980	2679	0.000
DNB	3677	6.93	0.99981	3826	0.000
24DNT	3519	6.62	0.99981	3656	0.000
4ADNT	1553	5.30	0.99953	1572	0.000
2ADNT	2668	7.47	0.99985	2799	0.000
RDX	1179	7.18	0.99987	1231	0.000

CALIBRATION FACTOR DATA
FORM 9C

Initial Calibration Date: 10/24/97
Continuing Calibration #: 1
Continuing Cal Date : 10/25/97
Continuing Cal Level : 3
Instrument ID : IN12
Column ID : LC-CN-05
Injection File Name : 2CN1024,23
Calculation Mode : Area

Component Name	Response Cont Std	Cont CF	Init CF Mean	%D	Init CF Slope	%D
TNB	132179	2299	2290	0.40	2367	2.87
26DNT	106820	1709	1709	0.01	1739	1.74
TNT	149348	2597	2585	0.48	2675	2.90
3,4DNT	148994	1806	1764	2.39	1841	1.88
TETRYL	198935	1768	1804	2.00	1840	3.87
HMX	64931	742	755	1.67	776	4.33

CALIBRATION FACTOR DATA
FORM 9C

Initial Calibration Date: 10/24/97
Continuing Calibration #: 1
Continuing Cal Date : 10/25/97
Continuing Cal Level : 3
Instrument ID : IN12
Column ID : LC-CN-05
Injection File Name : 2CN1024,24
Calculation Mode : Area

Component Name	Response Cont Std	Cont CF	Init CF Mean	%D	Init CF Slope	%D
NB	169046	2705	2608	3.72	2679	0.96
DNB	192085	3842	3677	4.47	3826	0.41
24DNT	229116	3666	3519	4.16	3656	0.27
4ADNT	185942	1617	1553	4.08	1572	2.86
2ADNT	348977	2792	2668	4.66	2799	0.25
RDX	78258	1252	1179	6.19	1231	1.69

CALIBRATION FACTOR DATA
FORM 9D

EXPLOSIVE CALIBRATION - METHOD SW846-8330

jection volume = 100uL

Amount Units = ng

COMPONENT NAME	AMT ON COL Level 1	AMT ON COL Level 2	AMT ON COL Level 3	AMT ON COL Level 4	AMT ON COL Level 5
TNB	4.600	23.000	57.500	115.000	230.000
26DNT	5.000	25.000	62.500	125.000	250.000
TNT	4.600	23.000	57.500	115.000	230.000
3,4DNT	7.000	35.000	82.500	165.000	330.000
TETRYL	9.000	45.000	112.500	225.000	450.000
HMX	7.000	35.000	87.500	175.000	350.000

CALIBRATION FACTOR DATA
FORM 9D

EXPLOSIVE CALIBRATION - METHOD SW846-8330

jection volume = 100uL

Amount Units = ng

COMPONENT NAME	AMT ON COL Level 1	AMT ON COL Level 2	AMT ON COL Level 3	AMT ON COL Level 4	AMT ON COL Level 5
NB	5.000	25.000	62.500	125.000	250.000
DNB	4.000	20.000	50.000	100.000	200.000
24DNT	5.000	25.000	62.500	125.000	250.000
4ADNT	9.200	46.000	115.000	230.000	460.000
2ADNT	10.000	50.000	125.000	250.000	500.000
RDX	5.000	25.000	62.500	125.000	250.000

CALIBRATION FACTOR DATA
FORM 9E

EXPLOSIVE CALIBRATION - METHOD SW846-8330

strument ID : IN12
olumn ID : LC-CN-05
Calibration Date : 10/24/97
Number of Calibration Levels: 5

COMPONENT NAME	RT Level 1	RT Level 2	RT Level 3	RT Level 4	RT Level 5
TNB	5.98	5.97	5.98	5.96	5.96
26DNT	6.57	6.57	6.57	6.55	6.55
TNT	7.06	7.06	7.06	7.05	7.04
3,4DNT	8.06	8.06	8.06	8.04	8.04
TETRYL	10.71	10.72	10.72	10.69	10.69
HMX	12.24	12.27	12.28	12.25	12.28

COMPONENT NAME	RT Cont CA1
TNB	5.96
26DNT	6.54
TNT	7.04
3,4DNT	8.03
TETRYL	10.65
HMX	12.16

COMPONENT NAME	AVE RT	STD_DEV	3X SD	AVE+-3X SD
TNB	5.97	0.012	0.035	5.93 - 6.00
26DNT	6.56	0.012	0.035	6.52 - 6.59
TNT	7.05	0.010	0.031	7.02 - 7.08
3,4DNT	8.05	0.016	0.047	8.00 - 8.10
TETRYL	10.70	0.026	0.079	10.62 - 10.78
HMX	12.25	0.047	0.140	12.11 - 12.39

CALIBRATION FACTOR DATA
FORM 9E

EXPLOSIVE CALIBRATION - METHOD SW846-8330

strument ID : IN12
olumn ID : LC-CN-05
Calibration Date : 10/24/97
Number of Calibration Levels: 5

COMPONENT NAME	RT Level 1	RT Level 2	RT Level 3	RT Level 4	RT Level 5
NB	5.40	5.39	5.37	5.38	5.38
DNB	5.89	5.88	5.86	5.87	5.87
24DNT	6.83	6.82	6.80	6.80	6.80
4ADNT	7.48	7.47	7.45	7.45	7.45
2ADNT	7.96	7.95	7.92	7.93	7.92
RDX	8.66	8.65	8.62	8.64	8.64

COMPONENT NAME	RT Cont CA1
NB	5.37
DNB	5.85
24DNT	6.78
4ADNT	7.42
2ADNT	7.90
RDX	8.62

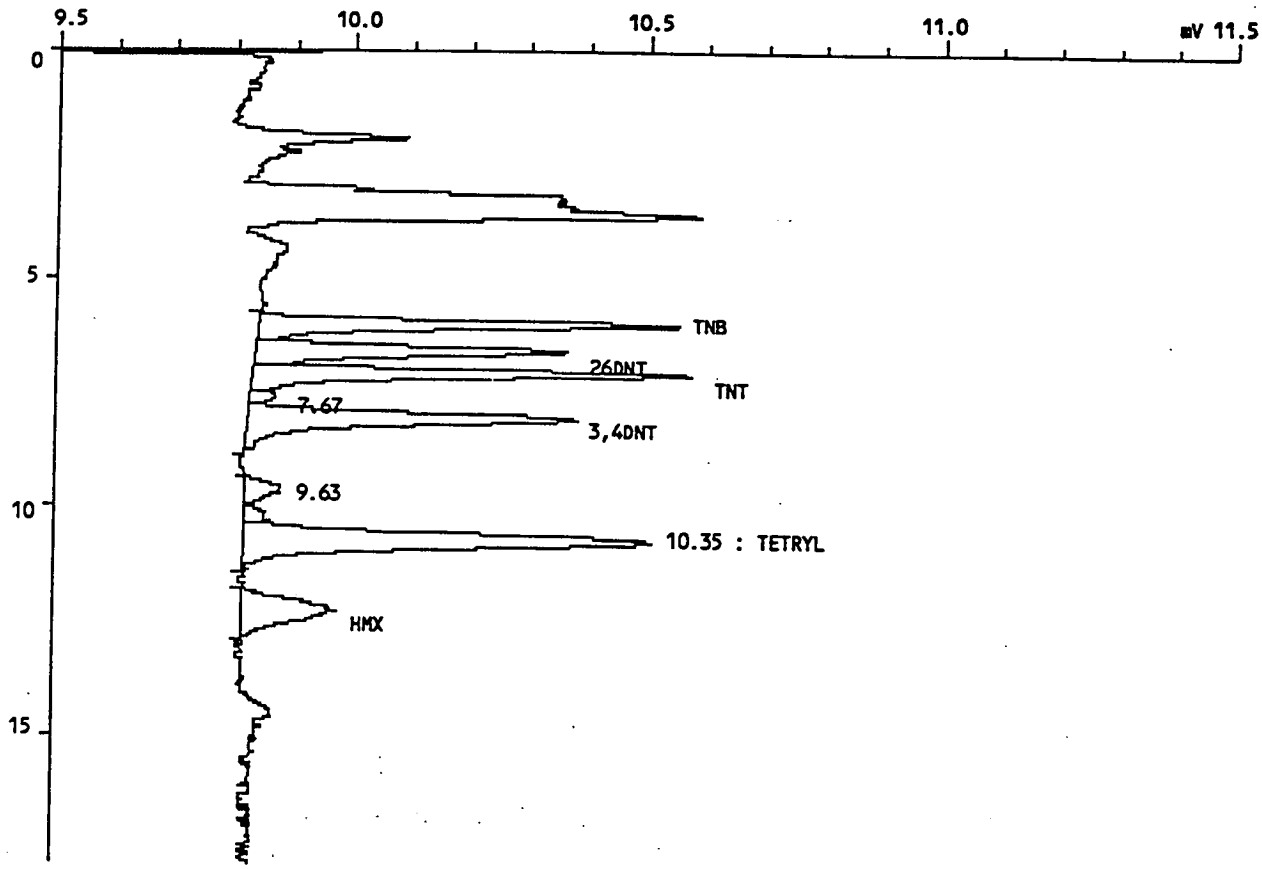
COMPONENT NAME	AVE RT	STD_DEV	3X SD	AVE+-3X SD
NB	5.38	0.014	0.041	5.34 - 5.42
DNB	5.87	0.015	0.045	5.83 - 5.92
24DNT	6.80	0.019	0.057	6.75 - 6.86
4ADNT	7.45	0.020	0.059	7.39 - 7.51
2ADNT	7.93	0.022	0.066	7.87 - 8.00
RDX	8.64	0.017	0.051	8.59 - 8.69

LONG PLOT

Injection F: <MC3> 2 2CN1024,4,1

Sample name.....: EXP L1A STD
Sample ID.....: 1-118:IA1
INST ID: IN12, COL. TYPE: LC-CN, ID: LC-CN-05, INJ:100, UV

Acquired on 24-Oct-97 at 21:11:06
Reported on 05-Nov-97 at 11:29:54



INJECTION REPORT

Injection F: <MC3> 2 2CN1024,4,1

Acquired on 24-Oct-97 at 21:11:06
Modified on 05-Nov-81 at 11:12:06
Reported on 05-Nov-97 at 11:13:49

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN12, COL. TYPE: LC-CN, ID: LC-CN-05, INJ:100, UV
Number of samples.: 67
Calibration file..: 2CN1024B Last modified on 05-Nov-81 at 11:12:56
Method file.....: LCCN Last modified on 05-Nov-81 at 11:13:30
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: EXP L1A STD
Sample ID.....: 1-118:IA1
Sample type.....: Standard
Sample amount.....: 1.0000
Number of injections.....: 1
Bottle Number.....: 1

Retention time update.....: No update
Calibration level identifier.....: 1
Response factor update.....: Initialise

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 1.000
Dilution.....: 1.000
Amount (g or mL).....: 1.000

PEAK INFORMATION

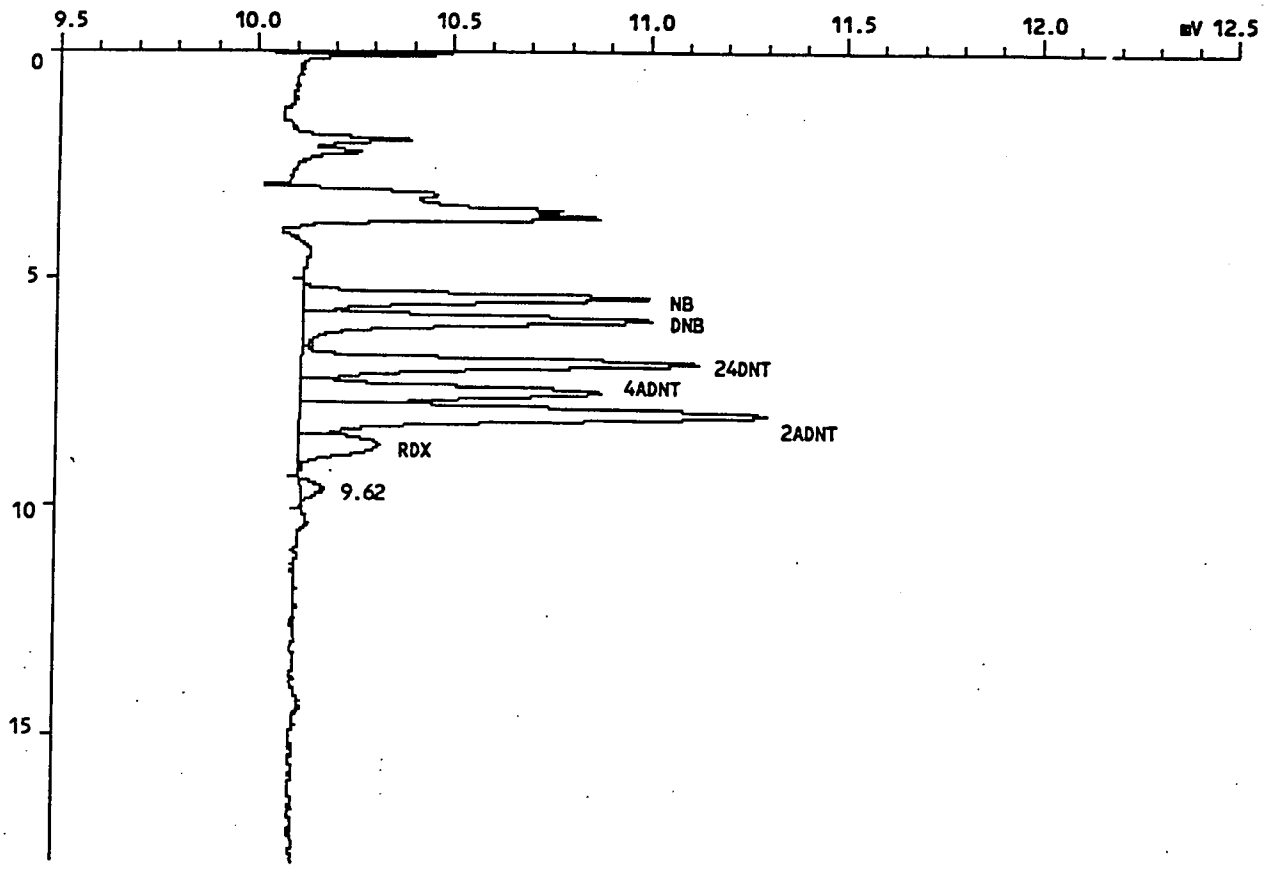
Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
1	5.984	717	9616	0.041	TNB
2	6.571	532	8132	0.047	26DNT
3	7.061	748	10923	0.041	TNT
5	8.064	561	11396	0.062	3,4DNT
8	10.709	694	15151	0.082	TETRYL
9	12.235	162	4949	0.064	HMX
Total		3415	60167	0.336	
Residual		145	2585	0.000	

LONG PLOT

Injection F: <MC3> 2 2CN1024,9,1

Sample name.....: EXP L1B STD
Sample ID.....: 1-122:IB1
INST ID: IN12, COL. TYPE: LC-CN, ID: LC-CN-05, INJ:100, UV

Acquired on 24-Oct-97 at 23:52:24
Reported on 05-Nov-97 at 11:33:43



INJECTION REPORT

Injection F: <MC3> 2 2CN1024,9,1

Acquired on 24-Oct-97 at 23:52:24
Modified on 05-Nov-81 at 11:12:38
Reported on 05-Nov-97 at 11:15:12

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN12, COL. TYPE: LC-CN, ID: LC-CN-05, INJ:100, UV
Number of samples.: 67
Calibration file..: 2CN1024B Last modified on 05-Nov-81 at 11:12:56
Method file.....: LCCN Last modified on 05-Nov-81 at 11:13:30
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: EXP L1B STD
Sample ID.....: 1-122:IB1
Sample type.....: Standard
Sample amount.....: 1.0000
Number of injections.....: 1
Bottle Number.....: 1

Retention time update.....: No update
Calibration level identifier.....: 1
Response factor update.....: Initialise

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 1.000
Dilution.....: 1.000
Amount (g or mL).....: 1.000

PEAK INFORMATION

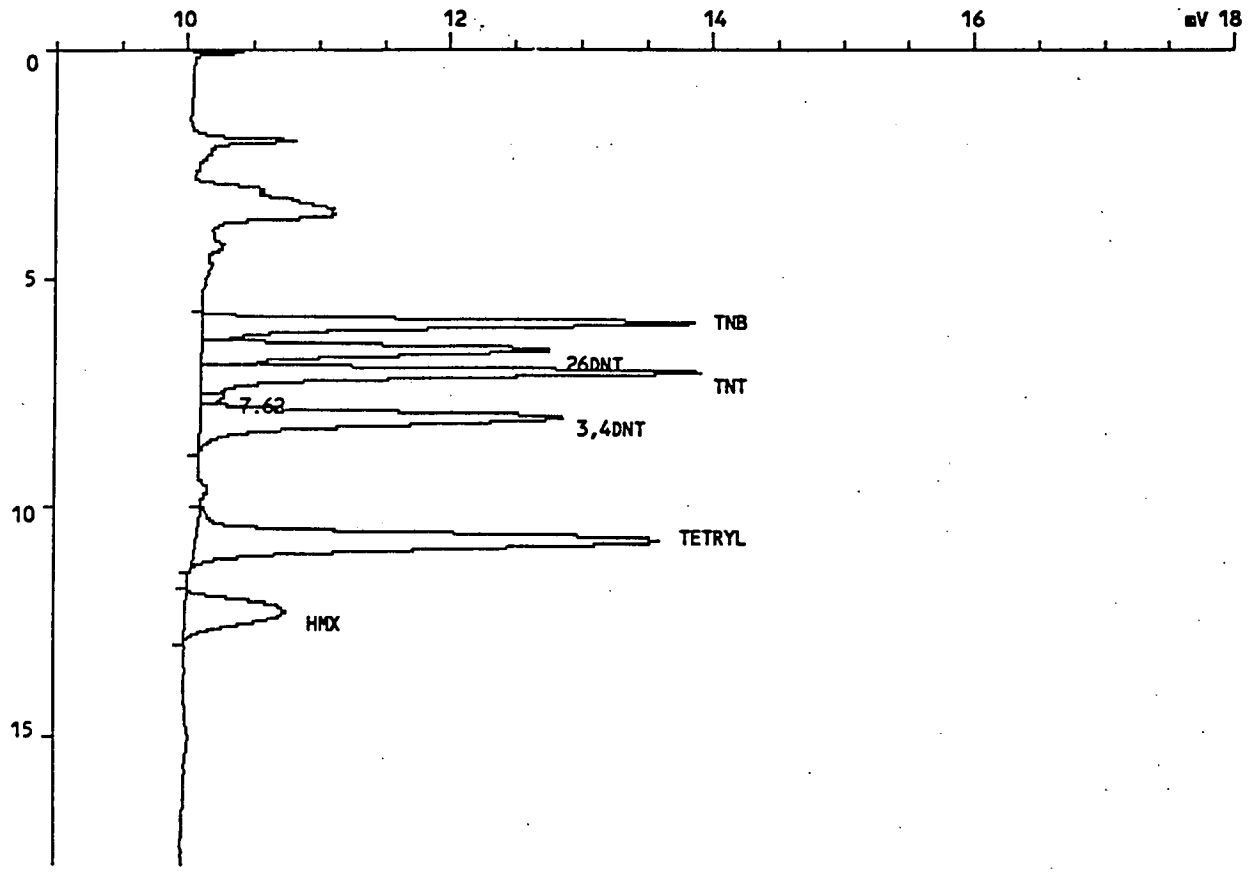
Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
1	5.403	878	12662	0.046	NB
2	5.893	895	14230	0.037	DNB
3	6.832	1009	16899	0.046	24DNT
4	7.477	770	14145	0.087	4ADNT
5	7.963	1189	25109	0.088	2ADNT
6	8.661	208	5840	0.046	RDX
Total		4949	88885	0.350	
Residual		63	1050	0.000	

LONG PLOT

Injection F: <MC3> 2 2CN1024,5,1

Sample name.....: EXP L2A STD
Sample ID.....: 1-117:IA2
INST ID: IN12, COL. TYPE: LC-CN, ID: LC-CN-05, INJ:100, UV

Acquired on 24-Oct-97 at 21:43:21
Reported on 05-Nov-97 at 11:30:56



INJECTION REPORT

Injection F: <MC3> 2 2CN1024,5,1

Acquired on 24-Oct-97 at 21:43:21
Modified on 05-Nov-81 at 11:12:02
Reported on 05-Nov-97 at 11:14:05

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN12, COL. TYPE: LC-CN, ID: LC-CN-05, INJ:100, UV
Number of samples.: 67
Calibration file..: 2CN1024B Last modified on 05-Nov-81 at 11:12:56
Method file.....: LCCN Last modified on 05-Nov-81 at 11:13:30
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: EXP L2A STD
Sample ID.....: 1-117:IA2
Sample type.....: Standard
Sample amount.....: 1.0000
Number of injections.....: 1
Bottle Number.....: 1

Retention time update.....: No update
Calibration level identifier.....: 2
Response factor update.....: Initialise

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 1.000
Dilution.....: 1.000
Amount (g or mL).....: 1.000

PEAK INFORMATION

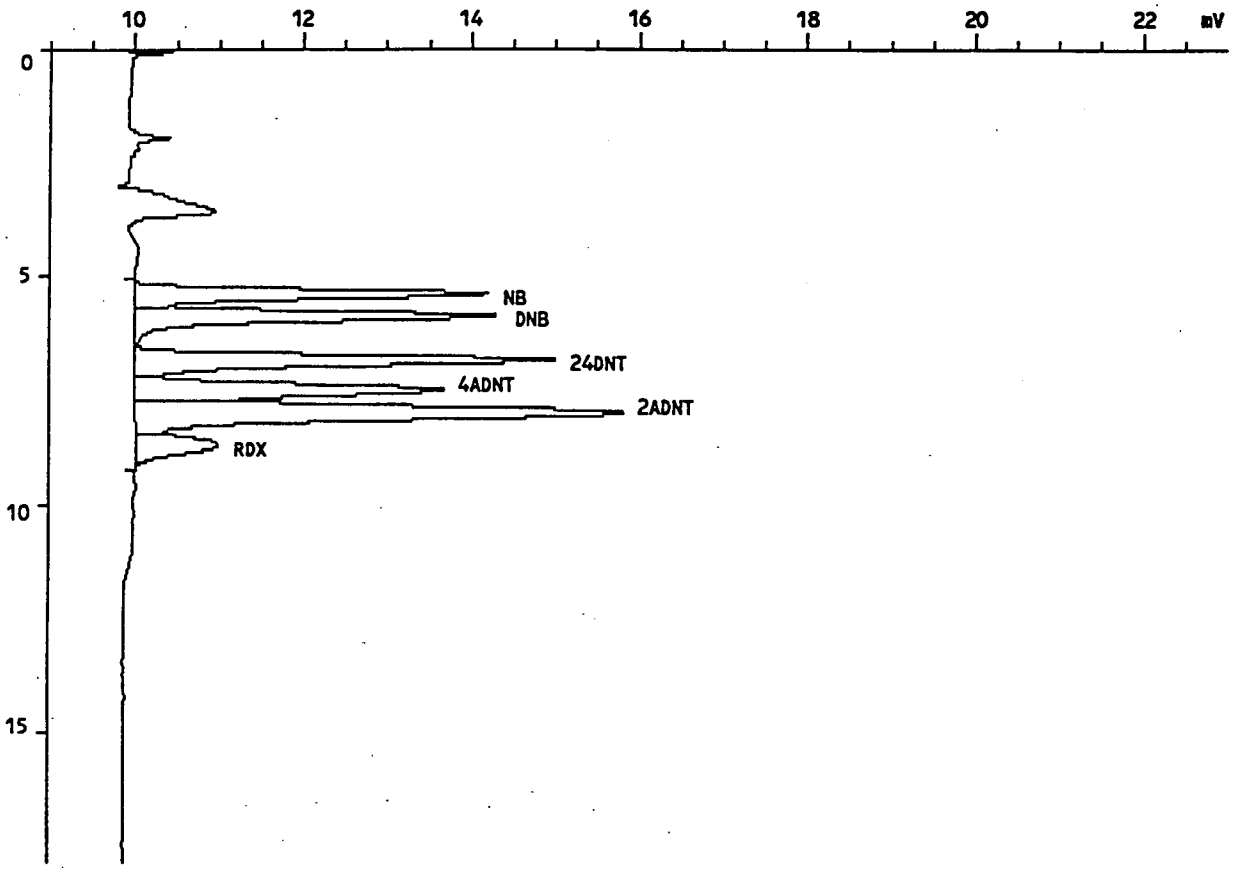
Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
1	5.973	3742	53170	0.225	TNB
2	6.565	2654	42884	0.247	26DNT
3	7.061	3809	59697	0.223	TNT
5	8.059	2766	59281	0.322	3,4DNT
6	10.720	3536	82281	0.447	TETRYL
7	12.272	754	25975	0.335	HMX
Total		17262	323288	1.799	
Residual		186	2480	0.000	

LONG PLOT

Injection F: <MC3> 2 2CN1024,10,1

Sample name.....: EXP L2B STD
Sample ID.....: 1-122:IB2
INST ID: IN12, COL. TYPE: LC-CN, ID: LC-CN-05, INJ:100, UV

Acquired on 25-Oct-97 at 00:24:39
Reported on 05-Nov-97 at 11:34:26



INJECTION REPORT

Injection F: <MC3> 2 2CN1024,10,1

Acquired on 25-Oct-97 at 00:24:39
Modified on 05-Nov-81 at 11:12:42
Reported on 05-Nov-97 at 11:15:29

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN12, COL. TYPE: LC-CN, ID: LC-CN-05, INJ:100, UV
Number of samples.: 67
Calibration file..: 2CN1024B Last modified on 05-Nov-81 at 11:12:56
Method file.....: LCCN Last modified on 05-Nov-81 at 11:13:30
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: EXP L2B STD
Sample ID.....: 1-122:IB2
Sample type.....: Standard
Sample amount.....: 1.0000
Number of injections.....: 1
Bottle Number.....: 1

Retention time update.....: No update
Calibration level identifier.....: 2
Response factor update.....: Initialise

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 1.000
Dilution.....: 1.000
Amount (g or mL).....: 1.000

PEAK INFORMATION

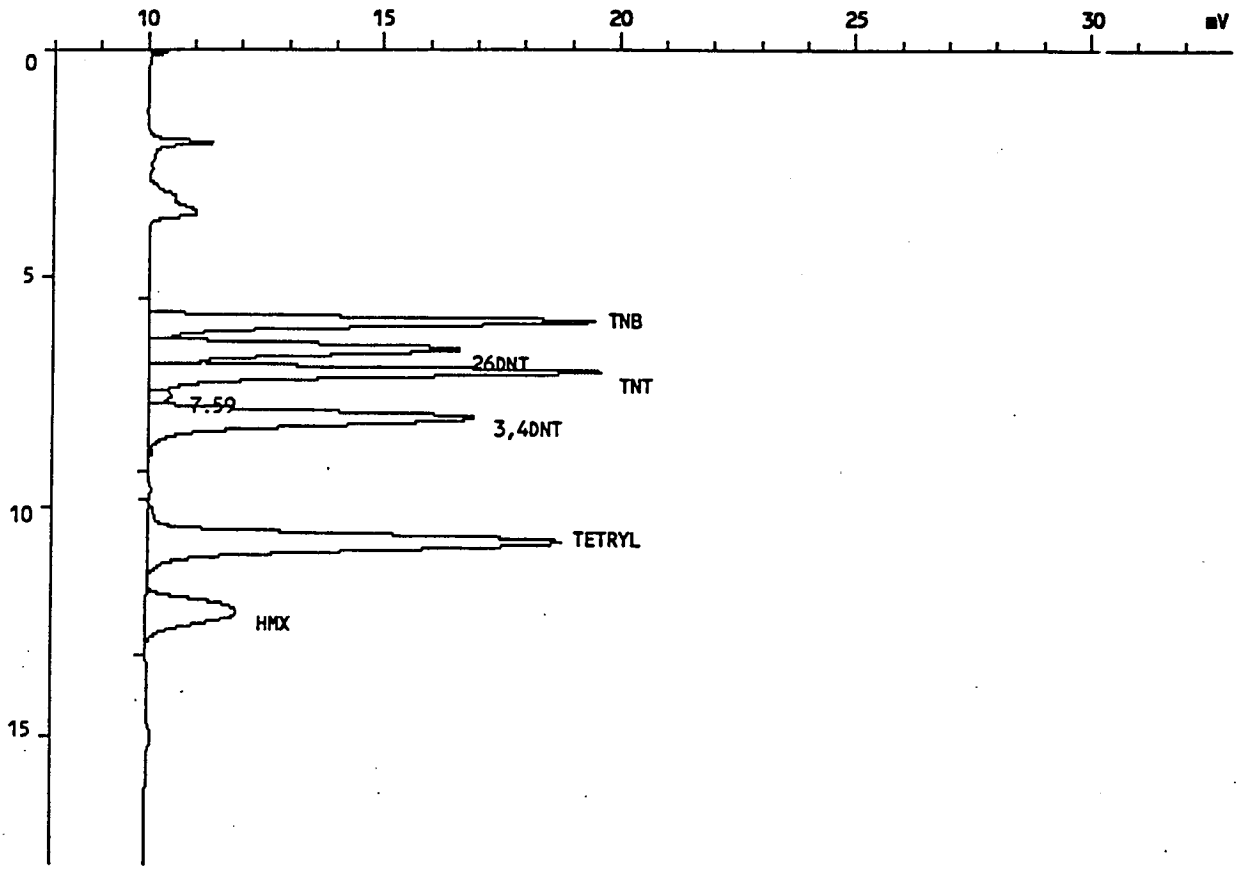
Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
1	5.392	4181	59601	0.219	NB
2	5.883	4288	65678	0.170	DNB
3	6.821	4972	79418	0.215	24DNT
4	7.472	3681	65542	0.404	4ADNT
5	7.952	5816	119869	0.419	2ADNT
6	8.651	980	25900	0.205	RDX
Total		23918	416007	1.632	
Residual		0	0	0.000	

LONG PLOT

Injection F: <MC3> 2 2CN1024,6,1

Sample name.....: EXP L3A STD
Sample ID.....: 1-116:IA3
INST ID: IN12, COL. TYPE: LC-CN, ID: LC-CN-05, INJ:100, UV

Acquired on 24-Oct-97 at 22:15:37
Reported on 05-Nov-97 at 11:31:46



INJECTION REPORT

Injection F: <MC3> 2 2CN1024,6,1

Acquired on 24-Oct-97 at 22:15:37
Modified on 05-Nov-81 at 11:11:58
Reported on 05-Nov-97 at 11:14:22

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN12, COL. TYPE: LC-CN, ID: LC-CN-05, INJ:100, UV
Number of samples.: 67
Calibration file..: 2CN1024B Last modified on 05-Nov-81 at 11:12:56
Method file.....: LCCN Last modified on 05-Nov-81 at 11:13:30
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: EXP L3A STD
Sample ID.....: 1-116:IA3
Sample type.....: Standard
Sample amount.....: 1.0000
Number of injections.....: 1
Bottle Number.....: 1

Retention time update.....: No update
Calibration level identifier.....: 3
Response factor update.....: Initialise

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 1.000
Dilution.....: 1.000
Amount (g or mL).....: 1.000

PEAK INFORMATION

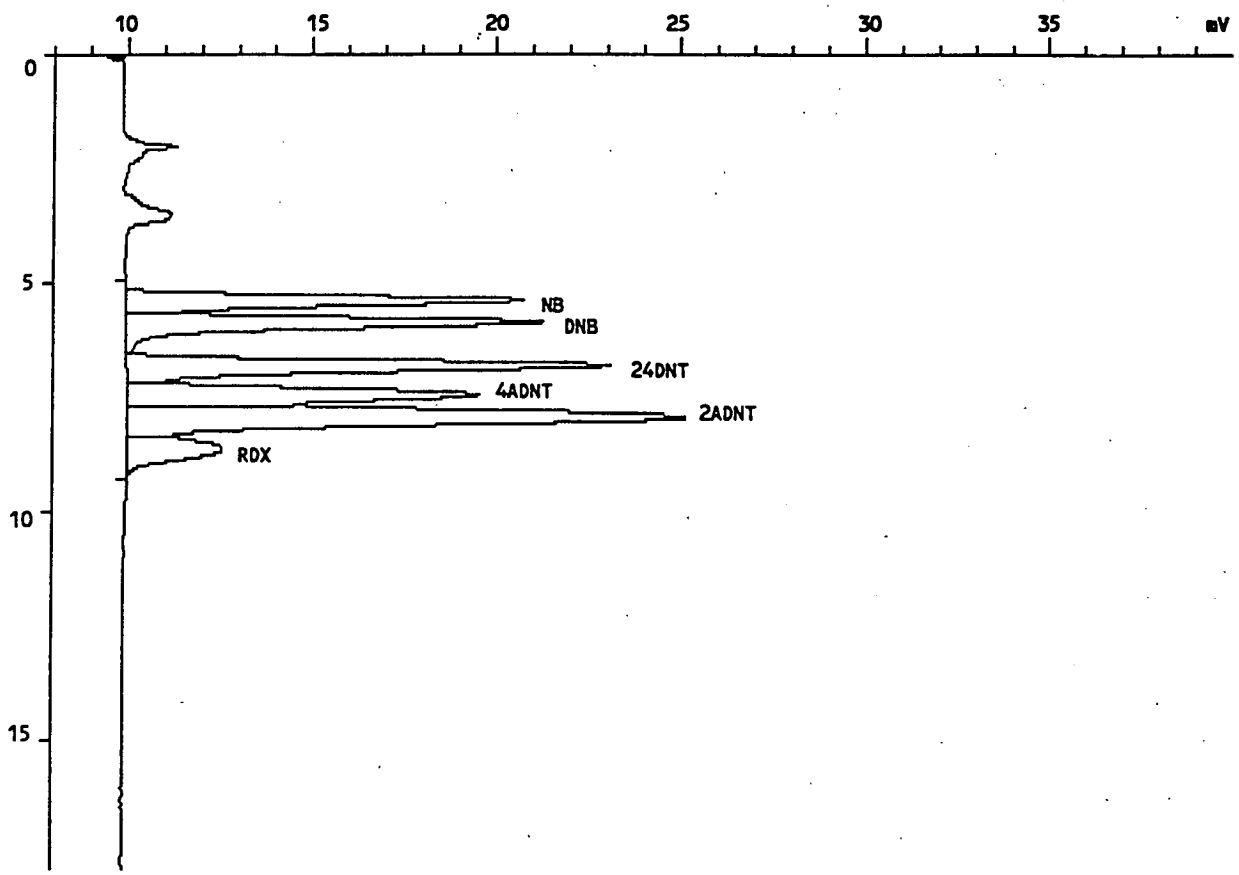
Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
1	5.979	9476	134596	0.569	TNB
2	6.571	6599	108907	0.626	26DNT
3	7.061	9590	152104	0.568	TNT
5	8.064	6935	152184	0.827	3,4DNT
6	10.720	8755	207087	1.126	TETRYL
7	12.283	1916	68047	0.877	HMX
Total		43271	822924	4.593	
Residual		492	6828	0.000	

LONG PLOT

Injection F: <MC3> 2 2CN1024,11,1

Sample name.....: EXP L3B STD
Sample ID.....: 1-121:IB3
INST ID: IN12, COL. TYPE: LC-CN, ID: LC-CN-05, INJ:100, UV

Acquired on 25-Oct-97 at 00:56:54
Reported on 05-Nov-97 at 11:35:05



INJECTION REPORT

Injection F: <MC3> 2 2CN1024,11,1

Acquired on 25-Oct-97 at 00:56:54
Modified on 05-Nov-81 at 11:12:46
Reported on 05-Nov-97 at 11:15:46

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN12, COL. TYPE: LC-CN, ID: LC-CN-05, INJ:100, UV
Number of samples.: 67
Calibration file..: 2CN1024B Last modified on 05-Nov-81 at 11:12:56
Method file.....: LCCN Last modified on 05-Nov-81 at 11:13:30
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: EXP L3B STD
Sample ID.....: 1-121:IB3
Sample type.....: Standard
Sample amount.....: 1.0000
Number of injections.....: 1
Bottle Number.....: 1

Retention time update.....: No update
Calibration level identifier.....: 3
Response factor update.....: Initialise

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 1.000
Dilution.....: 1.000
Amount (g or mL).....: 1.000

PEAK INFORMATION

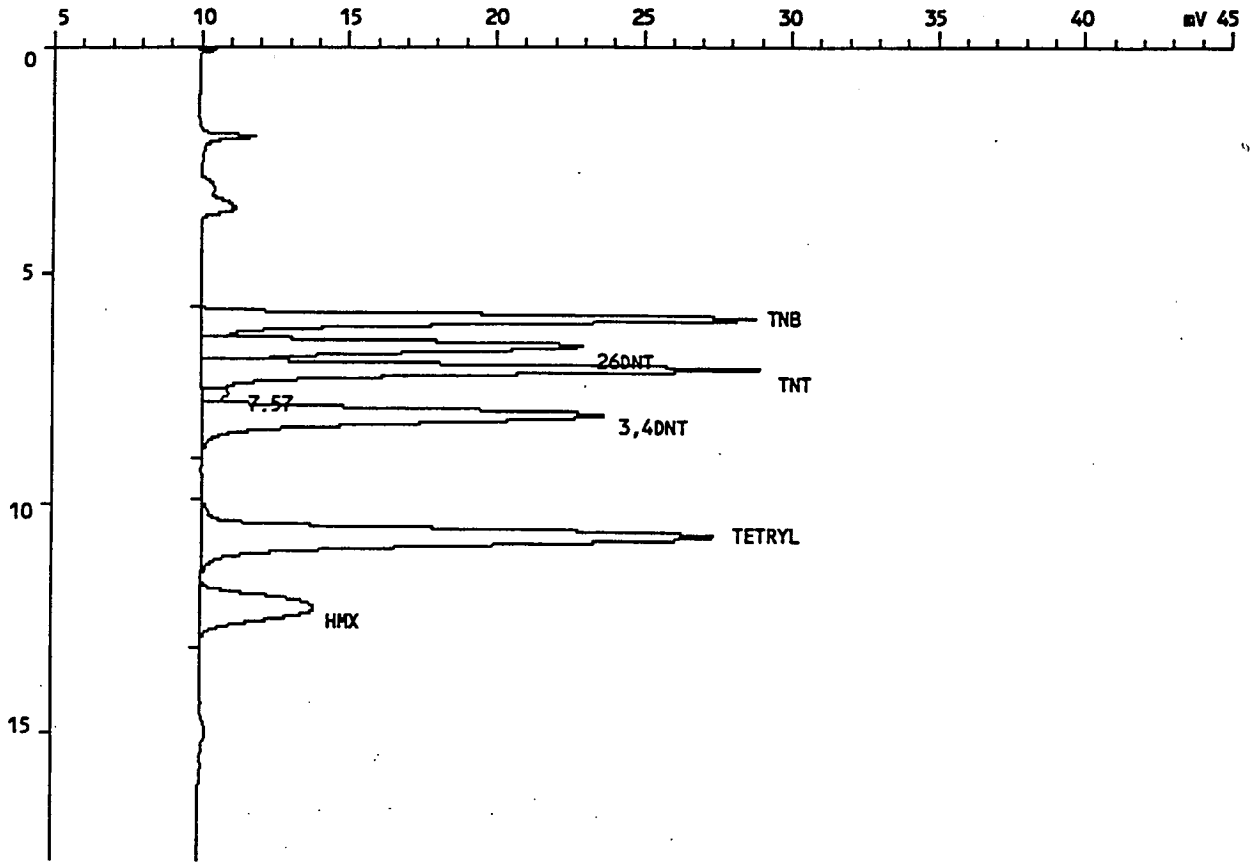
Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
1	5.371	10833	170369	0.625	NB
2	5.861	11308	192199	0.498	DNB
3	6.795	13159	229940	0.623	24DNT
4	7.445	9573	186226	1.149	4ADNT
5	7.920	15180	350542	1.227	2ADNT
6	8.619	2592	76466	0.606	RDX
Total		62645	1205741	4.727	
Residual		0	0	0.000	

LONG PLOT

Injection F: <MC3> 2 2CN1024,7,1

Sample name.....: EXP L4A STD
Sample ID.....: 1-115:IA4
INST ID: IN12, COL. TYPE: LC-CN, ID: LC-CN-05, INJ:100, UV

Acquired on 24-Oct-97 at 22:47:53
Reported on 05-Nov-97 at 11:32:24



INJECTION REPORT

Injection F: <MC3> 2 2CN1024,7,1

Acquired on 24-Oct-97 at 22:47:53
Modified on 05-Nov-81 at 11:11:54
Reported on 05-Nov-97 at 11:14:38

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN12, COL. TYPE: LC-CN, ID: LC-CN-05, INJ:100, UV
Number of samples.: 67
Calibration file..: 2CN1024B Last modified on 05-Nov-81 at 11:12:56
Method file.....: LCCN Last modified on 05-Nov-81 at 11:13:30
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: EXP L4A STD
Sample ID.....: 1-115:IA4
Sample type.....: Standard
Sample amount.....: 1.0000
Number of injections.....: 1
Bottle Number.....: 1

Retention time update.....: No update
Calibration level identifier.....: 4
Response factor update.....: Initialise

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 1.000
Dilution.....: 1.000
Amount (g or mL).....: 1.000

PEAK INFORMATION

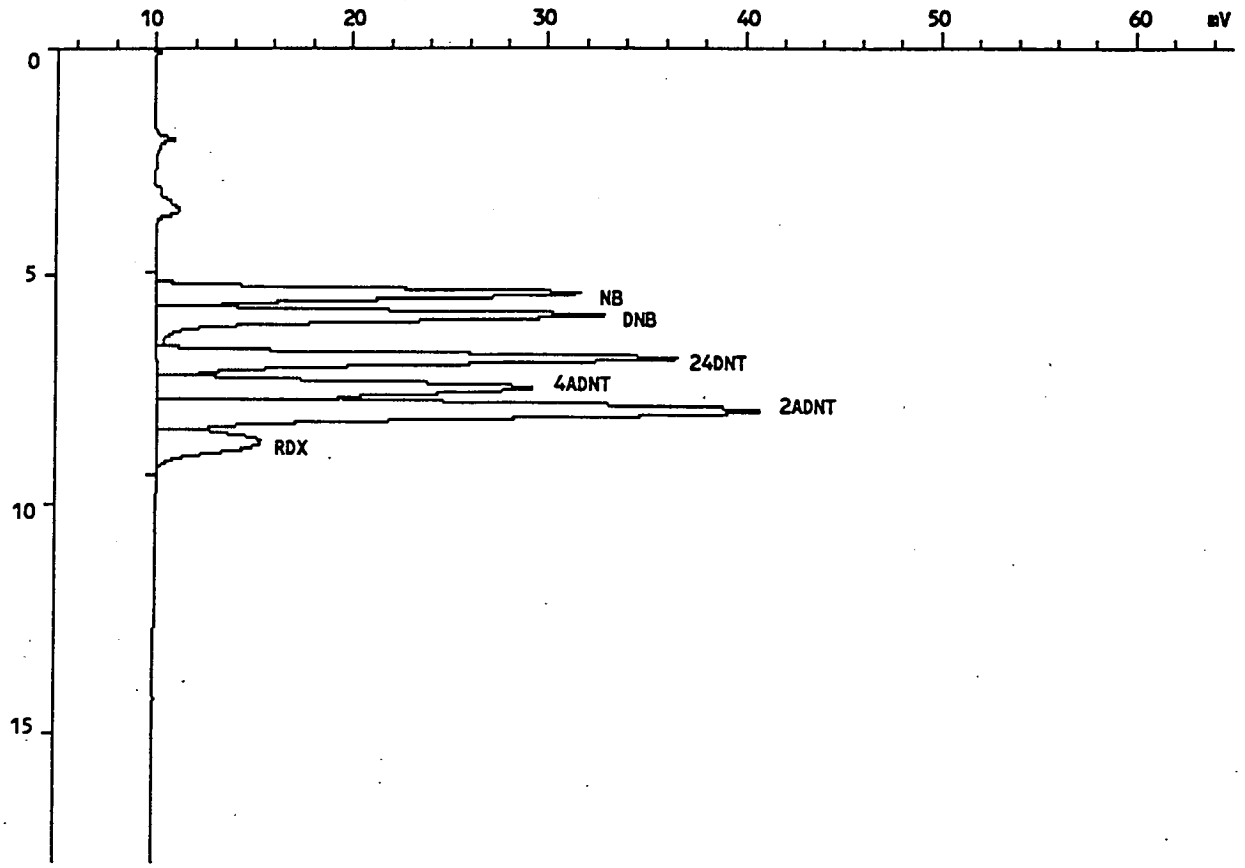
Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
1	5.963	18775	267447	1.130	TNB
2	6.555	12924	214372	1.229	26DNT
3	7.051	18895	301079	1.123	TNT
5	8.043	13625	297288	1.613	3,4DNT
6	10.688	17328	410955	2.231	TETRYL
7	12.251	3834	134580	1.734	HMX
Total		85380	1625720	9.060	
Residual		912	12764	0.000	

LONG PLOT

Injection F: <MC3> 2 2CN1024,12,1

Sample name.....: EXP L4B STD
Sample ID.....: 1-120:IB4
INST ID: IN12, COL. TYPE: LC-CN, ID: LC-CN-05, INJ:100, UV

Acquired on 25-Oct-97 at 01:29:10
Reported on 05-Nov-97 at 11:21:45



INJECTION REPORT

Injection F: <MC3> 2 2CN1024,12,1

Acquired on 25-Oct-97 at 01:29:10
Modified on 05-Nov-81 at 11:12:52
Reported on 05-Nov-97 at 11:16:03

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN12, COL. TYPE: LC-CN, ID: LC-CN-05, INJ:100, UV
Number of samples.: 67
Calibration file..: 2CN1024B Last modified on 05-Nov-81 at 11:12:56
Method file.....: LCCN Last modified on 05-Nov-81 at 11:13:30
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: EXP L4B STD
Sample ID.....: 1-120:IB4
Sample type.....: Standard
Sample amount.....: 1.0000
Number of injections.....: 1
Bottle Number.....: 1

Retention time update.....: No update
Calibration level identifier.....: 4
Response factor update.....: Initialise

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 1.000
Dilution.....: 1.000
Amount (g or mL).....: 1.000

PEAK INFORMATION

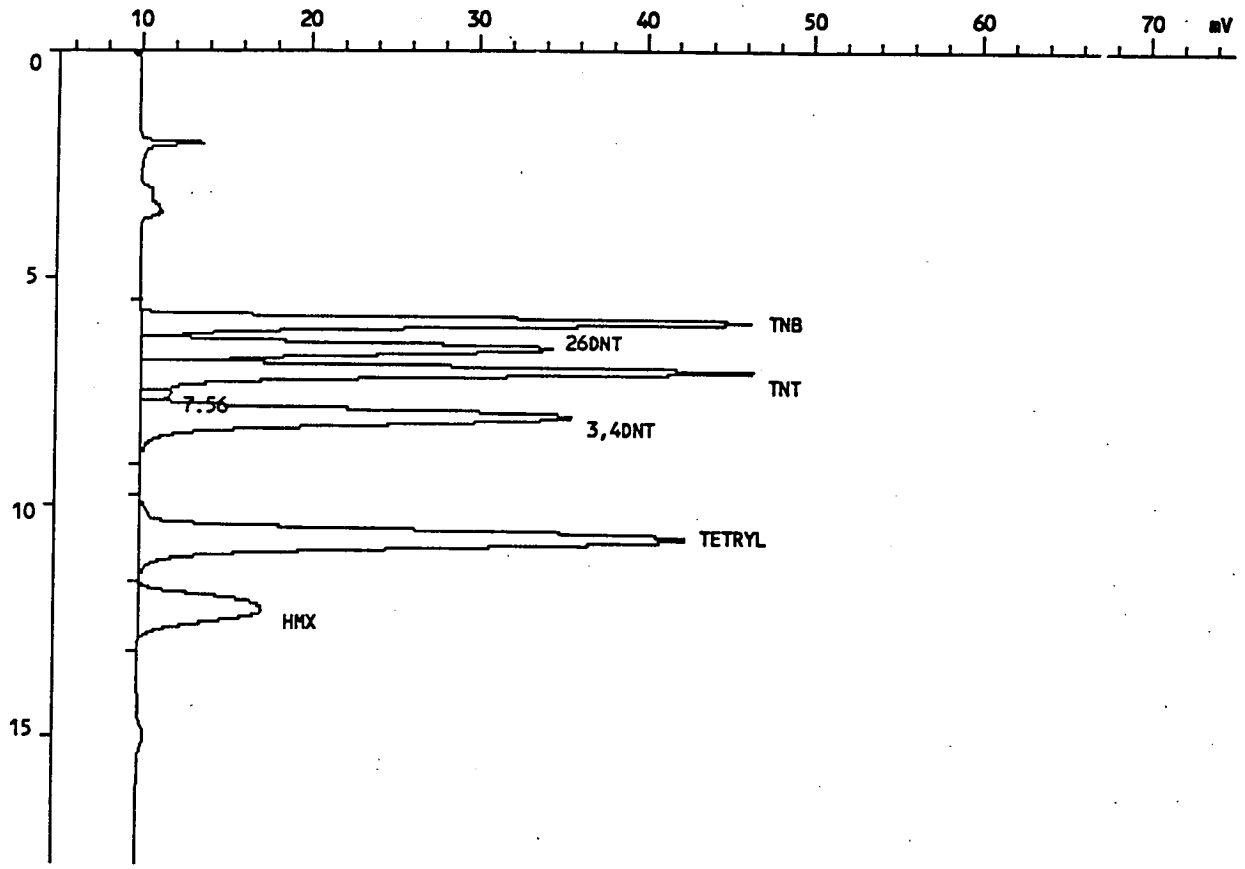
Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
1	5.381	21622	341307	1.248	NB
2	5.872	22789	388641	1.003	DNB
3	6.800	26447	464892	1.253	24DNT
4	7.451	19189	375137	2.310	4ADNT
5	7.931	30608	707856	2.471	2ADNT
6	8.640	5249	154508	1.226	RDX
Total		125904	2432339	9.511	
Residual		0	0	0.000	

LONG PLOT

Injection F: <MC3> 2 2CN1024,8,1

Sample name.....: EXP L5A STD
Sample ID.....: 1-114:IA5
INST ID: IN12, COL. TYPE: LC-CN, ID: LC-CN-05, INJ:100, UV

Acquired on 24-Oct-97 at 23:20:08
Reported on 05-Nov-97 at 11:32:55



INJECTION REPORT

Injection F: <MC3> 2 2CN1024,8,1

Acquired on 24-Oct-97 at 23:20:08
Modified on 05-Nov-81 at 11:11:46
Reported on 05-Nov-97 at 11:14:55

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN12, COL. TYPE: LC-CN, ID: LC-CN-05, INJ:100, UV
Number of samples.: 67
Calibration file..: 2CN1024B Last modified on 05-Nov-81 at 11:12:56
Method file.....: LCCN Last modified on 05-Nov-81 at 11:13:30
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: EXP L5A STD
Sample ID.....: 1-114:IA5
Sample type.....: Standard
Sample amount.....: 1.0000
Number of injections.....: 1
Bottle Number.....: 1

Retention time update.....: No update
Calibration level identifier.....: 5
Response factor update.....: Initialise

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 1.000
Dilution.....: 1.000
Amount (g or mL).....: 1.000

PEAK INFORMATION

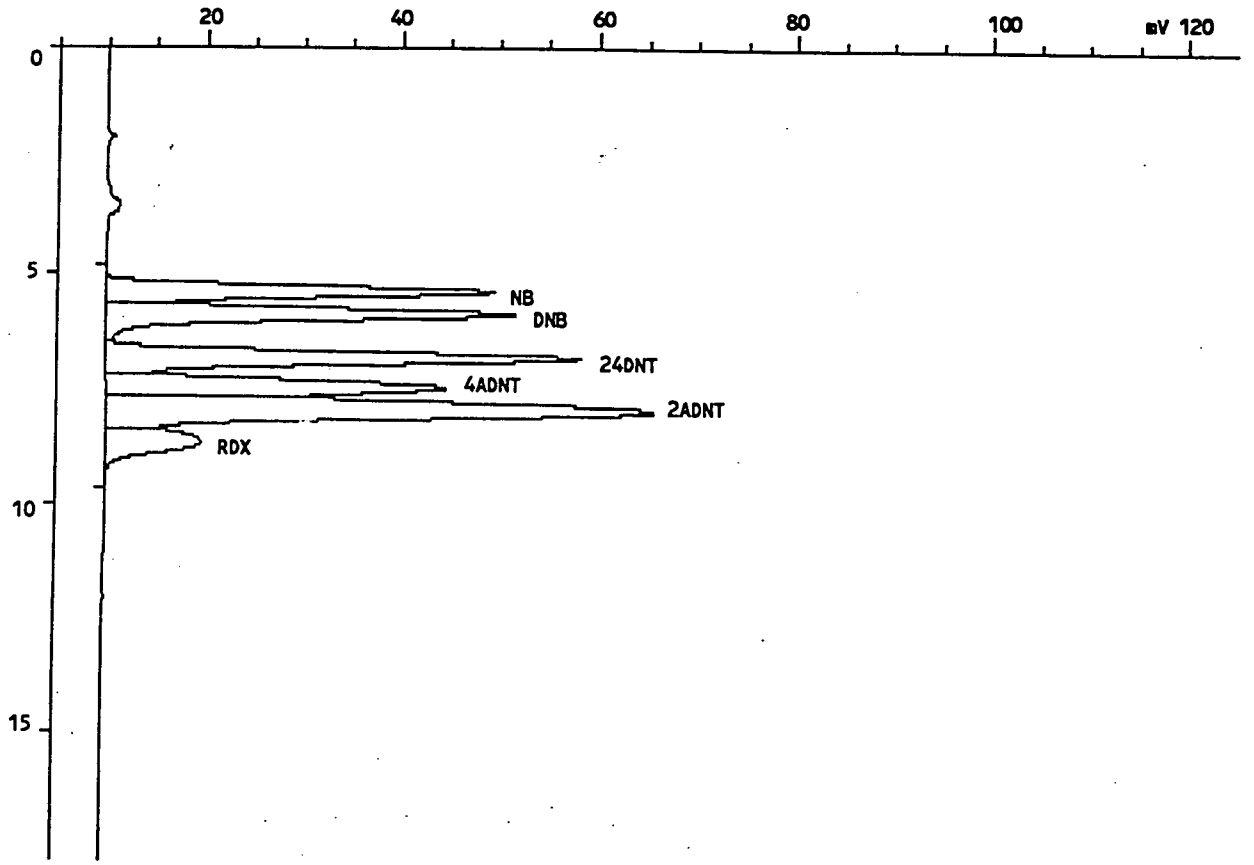
Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
1	5.957	36303	547189	2.306	TNB
2	6.549	24459	436380	2.473	26DNT
3	7.040	36407	619068	2.292	TNT
5	8.037	25609	610956	3.270	3,4DNT
6	10.688	32447	829280	4.472	TETRYL
7	12.283	7264	272134	3.488	HMX
Total		162489	3315007	18.301	
Residual		1860	23856	0.000	

LONG PLOT

Injection F: <MC3> 2 2CN1024,13,1

Sample name.....: EXP L5B STD
Sample ID.....: 1-119:IB5
INST ID: IN12, COL. TYPE: LC-CN, ID: LC-CN-05, INJ:100, UV

Acquired on 25-Oct-97 at 02:01:25
Reported on 05-Nov-97 at 11:21:02



INJECTION REPORT

Injection F: <MC3> 2 2CN1024,13,1

Acquired on 25-Oct-97 at 02:01:25
Modified on 05-Nov-81 at 11:12:56
Reported on 05-Nov-97 at 11:16:20

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN12, COL. TYPE: LC-CN, ID: LC-CN-05, INJ:100, UV
Number of samples.: 67
Calibration file..: 2CN1024B Last modified on 05-Nov-81 at 11:12:56
Method file.....: LCCN Last modified on 05-Nov-81 at 11:13:30
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: EXP L5B STD
Sample ID.....: 1-119:IB5
Sample type.....: Standard
Sample amount.....: 1.0000
Number of injections.....: 1
Bottle Number.....: 1

Retention time update.....: No update
Calibration level identifier.....: 5
Response factor update.....: Initialise

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 1.000
Dilution.....: 1.000
Amount (g or mL).....: 1.000

PEAK INFORMATION

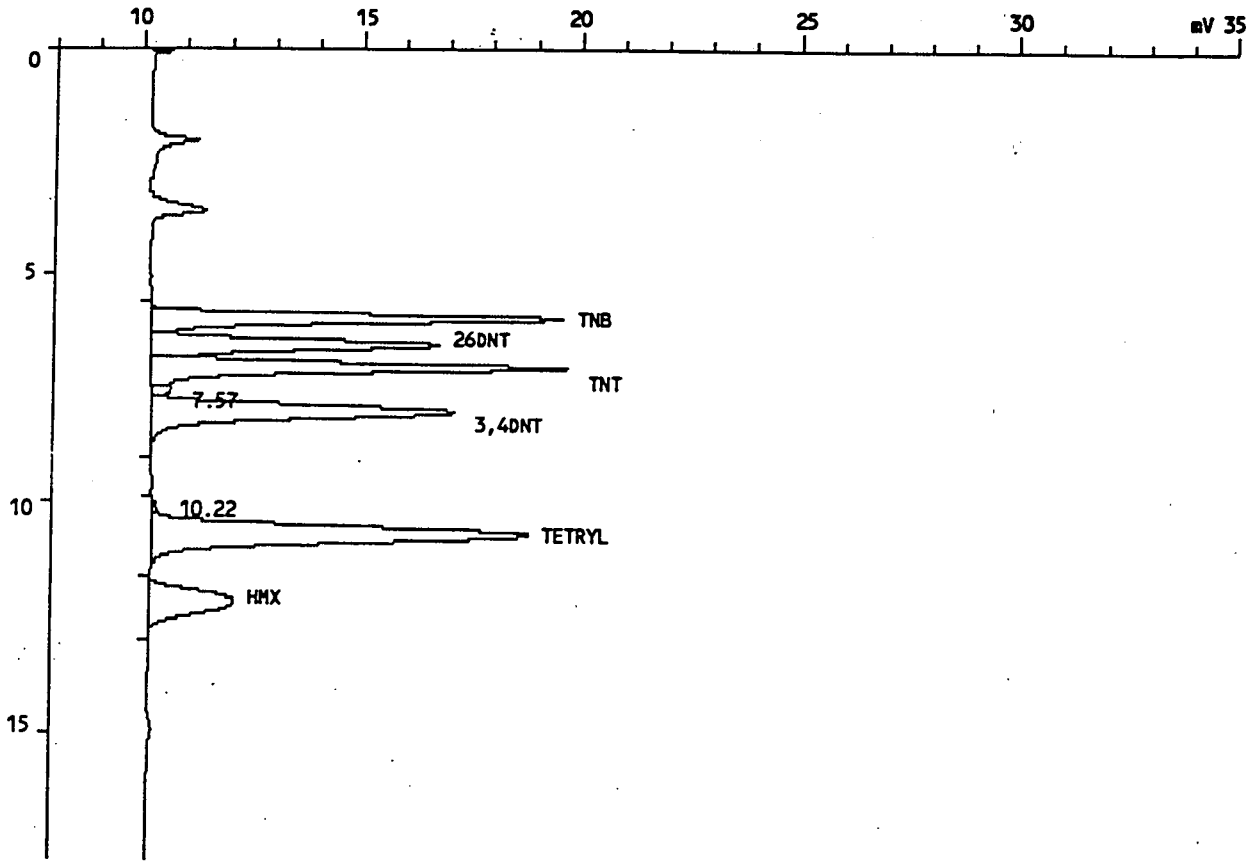
Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
1	5.381	39751	666417	2.488	NB
2	5.872	41947	762869	1.994	DNB
3	6.800	48353	910618	2.491	24DNT
4	7.445	34683	715160	4.552	4ADNT
5	7.920	55756	1396915	4.992	2ADNT
6	8.645	9749	308080	2.502	RDX
Total		230239	4760059	19.020	
Residual		0	0	0.000	

LONG PLOT

Injection F: <MC3> 2 2CN1024,23,1

Sample name.....: EXP L3A STD
Sample ID.....: 1-116:CA1
INST ID: IN12, COL. TYPE: LC-CN, ID: LC-CN-05, INJ:100, UV

Acquired on 25-Oct-97 at 07:23:59
Reported on 05-Nov-97 at 14:49:11



INJECTION REPORT

Injection F: <MC3> 2 2CN1024,23,1

Acquired on 25-Oct-97 at 07:23:59
Modified on 05-Nov-81 at 14:47:12
Reported on 05-Nov-97 at 14:47:44

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN12, COL. TYPE: LC-CN, ID: LC-CN-05, INJ:100, UV
Number of samples.: 67
Calibration file..: 2CN1024B Last modified on 05-Nov-81 at 11:56:18
Method file.....: LCCN Last modified on 05-Nov-81 at 12:04:50
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: EXP L3A STD
Sample ID.....: 1-116:CA1
Sample type.....: Standard
Sample amount.....: 1.0000
Number of injections.....: 1
Bottle Number.....: 1

Retention time update.....: No update
Calibration level identifier.....: 3
Response factor update.....: No update

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 1.000
Dilution.....: 1.000
Amount (g or mL).....: 1000.000

PEAK INFORMATION

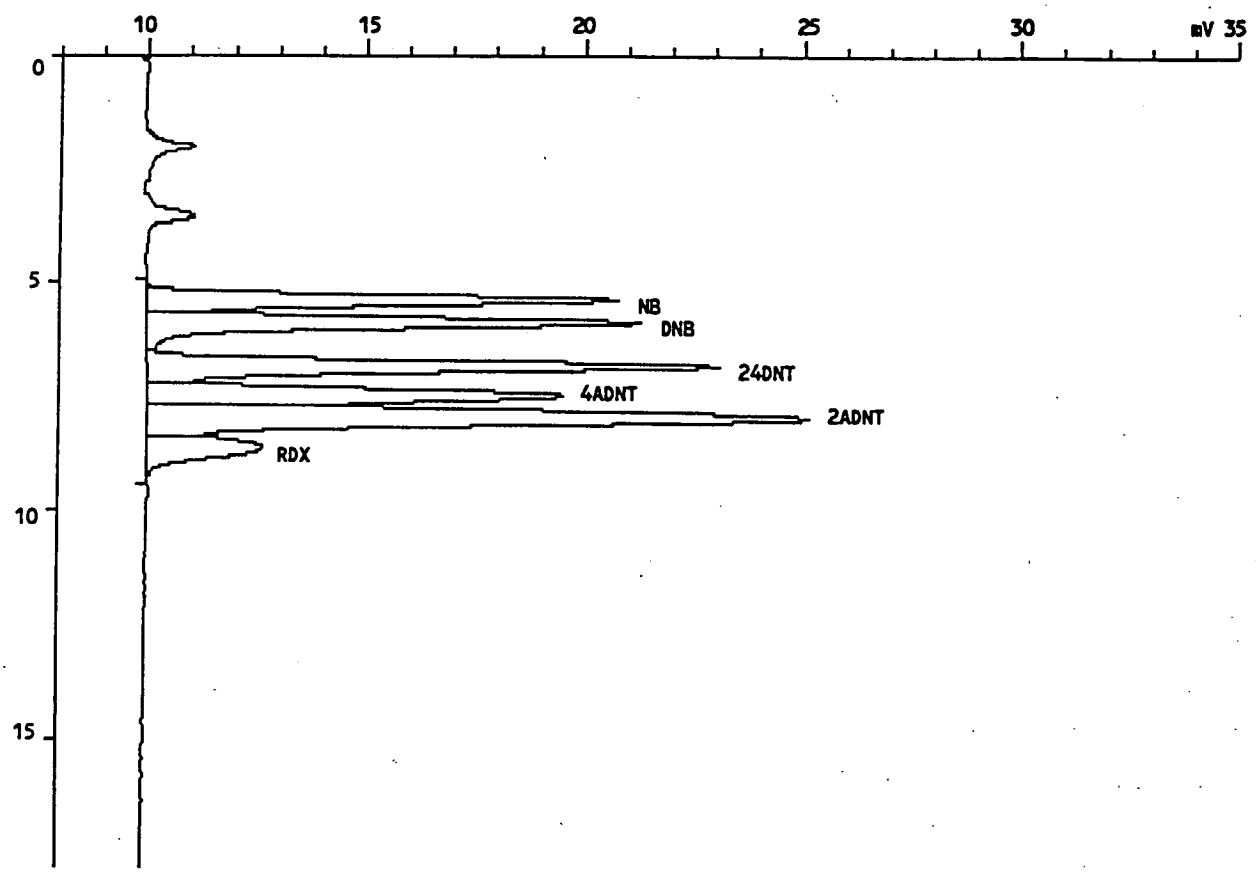
Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
1	5.957	9398	132179	0.577	TNB
2	6.544	6555	106820	0.625	26DNT
3	7.040	9515	149348	0.578	TNT
5	8.027	6928	148994	0.845	3,4DNT
7	10.651	8654	198935	1.103	TETRYL
8	12.160	1897	64931	0.860	HMX
Total		42947	801208	4.587	
Residual		615	7439	0.000	

LONG PLOT

Injection F: <MC3> 2 2CN1024,24,1

Sample name.....: EXP L3B STD
Sample ID.....: 1-121:CB1
INST ID: IN12, COL. TYPE: LC-CN, ID: LC-CN-05, INJ:100, UV

Acquired on 25-Oct-97 at 07:56:15
Reported on 05-Nov-97 at 14:48:51



INJECTION REPORT

Injection F: <MC3> 2 2CN1024,24,1

Acquired on 25-Oct-97 at 07:56:15
Modified on 05-Nov-81 at 14:47:20
Reported on 05-Nov-97 at 14:47:24

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN12, COL. TYPE: LC-CN, ID: LC-CN-05, INJ:100, UV
Number of samples.: 67
Calibration file..: 2CN1024B Last modified on 05-Nov-81 at 11:56:18
Method file.....: LCCN Last modified on 05-Nov-81 at 12:04:50
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: EXP L3B STD
Sample ID.....: 1-121:CB1
Sample type.....: Standard
Sample amount.....: 1.0000
Number of injections.....: 1
Bottle Number.....: 1

Retention time update.....: No update
Calibration level identifier.....: 3
Response factor update.....: No update

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 1.000
Dilution.....: 1.000
Amount (g or mL).....: 1000.000

PEAK INFORMATION

Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
1	5.365	10789	169046	0.648	NB
2	5.851	11297	192085	0.522	DNB
3	6.779	13119	229116	0.651	24DNT
4	7.424	9571	185942	1.197	4ADNT
5	7.904	15166	348977	1.308	2ADNT
6	8.619	2631	78258	0.664	RDX
Total		62574	1203424	4.991	
Residual		0	0	0.000	

CALIBRATION FACTOR DATA
FORM 9A-1

EXPLOSIVE CALIBRATION - METHOD SW846-8330
strument ID: IN10
lumn ID : LC-CN-07

Inj 1

Injection File Name Level 1 : 3CN1027,4
Injection File Name Level 2 : 3CN1027,5
Injection File Name Level 3 : 3CN1027,6
Injection File Name Level 4 : 3CN1027,7
Injection File Name Level 5 : 3CN1027,8

Calibration Date : 10/27/97
Number of Calibration Levels: 5
Line forced thru zero : Yes
Calculation Method : Area

COMPONENT NAME	Area Level 1	CAL FACTOR Level 1	Area Level 2	CAL FACTOR Level 2	Area Level 3	CAL FACTOR Level 3	Area Level 4	CAL FACTOR Level 4	Area Level 5	CAL FACTOR Level 5
TNB	11396	2477	58180	2530	153133	2663	312380	2716	589348	2562
26DNT	9718	1944	48630	1945	128325	2053	259192	2074	515564	2062
TNT	11587	2519	57145	2485	151942	2642	310213	2698	604076	2626
3,4DNT	11271	1610	53552	1530	141258	1712	290701	1762	567503	1720
TETRYL	17029	1892	86380	1920	226027	2009	467032	2076	890076	1978
HMX	6993	999	37758	1079	100405	1147	204417	1168	397348	1135

CALIBRATION FACTOR DATA
FORM 9A-1

EXPLOSIVE CALIBRATION - METHOD SW846-8330
Instrument ID: IN10
Column ID : LC-CN-07

Inj 1

Injection File Name Level 1 : 3CN1027,9
Injection File Name Level 2 : 3CN1027,10
Injection File Name Level 3 : 3CN1027,11
Injection File Name Level 4 : 3CN1027,12
Injection File Name Level 5 : 3CN1027,13

Calibration Date : 10/27/97
Number of Calibration Levels: 5
Line forced thru zero : Yes
Calculation Method : Area

COMPONENT NAME	Area Level 1	CAL FACTOR Level 1	Area Level 2	CAL FACTOR Level 2	Area Level 3	CAL FACTOR Level 3	Area Level 4	CAL FACTOR Level 4	Area Level 5	CAL FACTOR Level 5
NB	9541	1908	47743	1910	126646	2026	257911	2063	498467	1994
DNB	13793	3448	69281	3464	191347	3827	381461	3815	738945	3695
24DNT	15984	3197	80132	3205	221650	3546	438810	3510	847150	3389
4ADNT	16579	1802	83388	1813	229380	1995	452776	1969	867343	1886
2ADNT	24786	2479	124064	2481	337472	2700	673302	2693	1308533	2617
RDX	7609	1522	34218	1369	92737	1484	181518	1452	349323	1397

CALIBRATION FACTOR DATA
FORM 9B

EXPLOSIVE CALIBRATION - METHOD SW846-8330
Instrument ID: IN10
Column ID : LC-CN-07

Inj 1

Injection File Name Level 1 : 3CN1027,4
Injection File Name Level 2 : 3CN1027,5
Injection File Name Level 3 : 3CN1027,6
Injection File Name Level 4 : 3CN1027,7
Injection File Name Level 5 : 3CN1027,8

Calibration Date : 10/27/97
Number of Calibration Levels: 5
Line forced thru zero : Yes
Calculation Method : Area

Component Name	Mean Cal Factor	% RSD	Corr. Coeff.	Slope area/amt	Intercept
TNB	2590	3.78	0.99946	2597	0.000
26DNT	2016	3.24	0.99998	2063	0.000
TNT	2594	3.43	0.99986	2640	0.000
3,4DNT	1667	5.68	0.99981	1726	0.000
TETRYL	1975	3.69	0.99962	1998	0.000
HMX	1106	6.17	0.99984	1142	0.000

CALIBRATION FACTOR DATA
FORM 9B

EXPLOSIVE CALIBRATION - METHOD SW846-8330
Instrument ID: IN10
Column ID : LC-CN-07

Inj 1

Injection File Name Level 1 : 3CN1027,9
Injection File Name Level 2 : 3CN1027,10
Injection File Name Level 3 : 3CN1027,11
Injection File Name Level 4 : 3CN1027,12
Injection File Name Level 5 : 3CN1027,13

Calibration Date : 10/27/97
Number of Calibration Levels: 5
Line forced thru zero : Yes
Calculation Method : Area

Component Name	Mean Cal Factor	% RSD	Corr. Coeff.	Slope area/amt	Intercept
NB	1980	3.51	0.99979	2008	0.000
DNB	3650	5.05	0.99976	3723	0.000
24DNT	3370	4.88	0.99971	3419	0.000
4ADNT	1893	4.64	0.99961	1907	0.000
2ADNT	2594	4.20	0.99982	2635	0.000
RDX	1445	4.32	0.99969	1412	0.000

CALIBRATION FACTOR DATA
FORM 9C

Initial Calibration Date: 10/27/97
Continuing Calibration #: 1
Continuing Cal Date : 10/28/97
Continuing Cal Level : 3
Instrument ID : IN10
Column ID : LC-CN-07
Injection File Name : 3CN1027,28
Calculation Mode : Area

Component Name	Response Cont Std	Cont CF	Init CF Mean	%D	Init CF Slope	%D
TNB	157651	2742	2590	5.87	2597	5.56
26DNT	127429	2039	2016	1.16	2063	1.17
TNT	154256	2683	2594	3.42	2640	1.62
3,4DNT	144860	1756	1667	5.34	1726	1.73
TETRYL	224172	1993	1975	0.90	1998	0.28
HMX	99981	1143	1106	3.34	1142	0.08

12/1/97
BC
2269

CALIBRATION FACTOR DATA
FORM 9C

Initial Calibration Date: 10/27/97
Continuing Calibration #: 1
Continuing Cal Date : 10/28/97
Continuing Cal Level : 3
Instrument ID : IN10
Column ID : LC-CN-07
Injection File Name : 3CN1027,29
Calculation Mode : Area

Component Name	Response Cont Std	Cont CF	Init CF Mean	%D	Init CF Slope	%D
NB	129532	2073	1980	4.66	2008	3.20
DNB	206054	4121	3650	12.92	3723	10.70
24DNT	238847	3822	3370	13.42	3419	11.79
4ADNT	247805	2155	1893	13.85	1907	13.02
2ADNT	361076	2889	2594	11.36	2635	9.63
RDX	100724	1612	1445	11.55	1412	14.13

CALIBRATION FACTOR DATA
FORM 9D

EXPLOSIVE CALIBRATION - METHOD SW846-8330

.ijection volume = 100uL

Amount Units = ng

COMPONENT NAME	AMT ON COL Level 1	AMT ON COL Level 2	AMT ON COL Level 3	AMT ON COL Level 4	AMT ON COL Level 5
TNB	4.600	23.000	57.500	115.000	230.000
26DNT	5.000	25.000	62.500	125.000	250.000
TNT	4.600	23.000	57.500	115.000	230.000
3,4DNT	7.000	35.000	82.500	165.000	330.000
TETRYL	9.000	45.000	112.500	225.000	450.000
HMX	7.000	35.000	87.500	175.000	350.000

CALIBRATION FACTOR DATA
FORM 9D

EXPLOSIVE CALIBRATION - METHOD SW846-8330

.jection volume = 100uL

Amount Units = ng

COMPONENT NAME	AMT ON COL Level 1	AMT ON COL Level 2	AMT ON COL Level 3	AMT ON COL Level 4	AMT ON COL Level 5
NB	5.000	25.000	62.500	125.000	250.000
DNB	4.000	20.000	50.000	100.000	200.000
24DNT	5.000	25.000	62.500	125.000	250.000
4ADNT	9.200	46.000	115.000	230.000	460.000
2ADNT	10.000	50.000	125.000	250.000	500.000
RDX	5.000	25.000	62.500	125.000	250.000

CALIBRATION FACTOR DATA
FORM 9E

EXPLOSIVE CALIBRATION - METHOD SW846-8330

Instrument ID : IN10
Column ID : LC-CN-07
Calibration Date : 10/27/97
Number of Calibration Levels: 5

COMPONENT NAME	RT Level 1	RT Level 2	RT Level 3	RT Level 4	RT Level 5
TNB	6.19	6.19	6.16	6.16	6.15
26DNT	6.61	6.61	6.59	6.59	6.56
TNT	7.39	7.39	7.37	7.37	7.34
3,4DNT	8.40	8.42	8.38	8.38	8.36
TETRYL	12.07	12.08	12.04	12.04	12.00
HMX	15.52	15.57	15.49	15.49	15.44

COMPONENT NAME	RT Cont CA1	RT Cont CA2
TNB	6.11	6.13
26DNT	6.52	6.56
TNT	7.29	7.32
3,4DNT	8.29	8.34
TETRYL	11.88	11.95
HMX	15.22	15.34

COMPONENT NAME	AVE RT	STD_DEV	3X SD	AVE+-3X SD
TNB	6.16	0.032	0.095	6.06 - 6.25
26DNT	6.58	0.031	0.092	6.48 - 6.67
TNT	7.35	0.037	0.111	7.24 - 7.46
3,4DNT	8.37	0.044	0.133	8.23 - 8.50
TETRYL	12.01	0.072	0.217	11.79 - 12.23
HMX	15.44	0.120	0.361	15.08 - 15.80

CALIBRATION FACTOR DATA
FORM 9E

EXPLOSIVE CALIBRATION - METHOD SW846-8330

strument ID : IN10
Column ID : LC-CN-07
Calibration Date : 10/27/97
Number of Calibration Levels: 5

COMPONENT NAME	RT Level 1	RT Level 2	RT Level 3	RT Level 4	RT Level 5
NB	5.25	5.24	5.23	5.22	5.21
DNB	5.96	5.96	5.95	5.94	5.92
24DNT	6.96	6.97	6.95	6.94	6.91
4ADNT	7.68	7.69	7.69	7.68	7.64
2ADNT	8.38	8.39	8.37	8.36	8.33
RDX	9.58	9.65	9.64	9.64	9.61

COMPONENT NAME	RT Cont CA1	RT Cont CA2
NB	5.23	5.24
DNB	5.94	5.94
24DNT	6.92	6.94
4ADNT	7.65	7.68
2ADNT	8.33	8.36
X	9.59	9.64

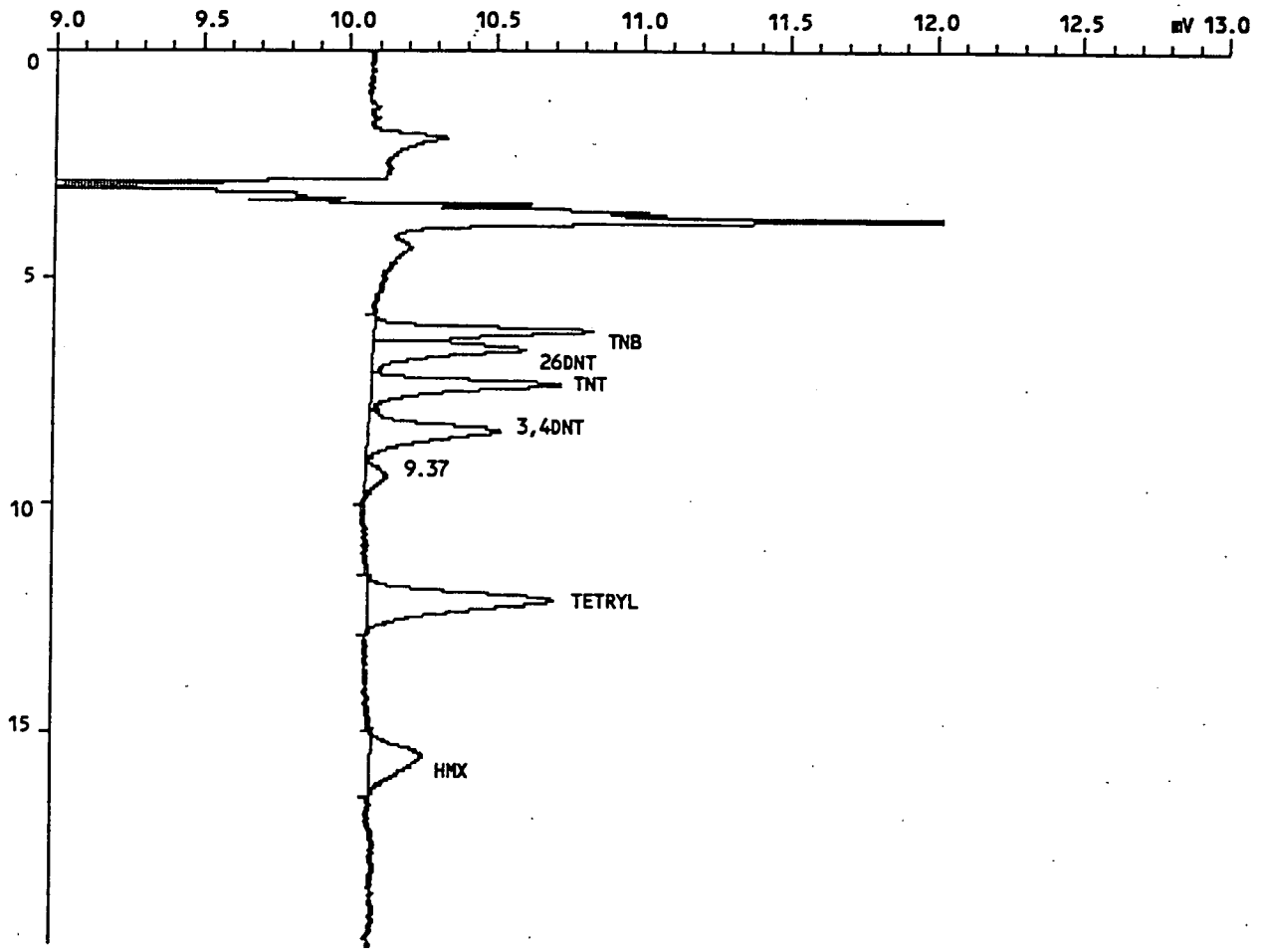
COMPONENT NAME	AVE RT	STD_DEV	3X SD	AVE+-3X SD
NB	5.23	0.014	0.041	5.19 - 5.27
DNB	5.94	0.014	0.041	5.90 - 5.98
24DNT	6.94	0.018	0.055	6.89 - 7.00
4ADNT	7.67	0.017	0.052	7.62 - 7.72
2ADNT	8.36	0.026	0.079	8.28 - 8.44
RDX	9.62	0.029	0.088	9.53 - 9.71

LONG PLOT

Injection F: <MC3> 3 3CN1027,4,1

Sample name.....: EXP L1A STD
Sample ID.....: 1-118:IA1
INST ID: IN10, COL. TYPE: LC-CN, ID: LC-CN-07, INJ:100, UV

Acquired on 27-Oct-97 at 22:50:42
Reported on 05-Nov-97 at 10:34:27



INJECTION REPORT

Injection F: <MC3> 3 3CN1027,4,1

Acquired on 27-Oct-97 at 22:50:42
Modified on 05-Nov-81 at 10:17:42
Reported on 05-Nov-97 at 10:31:59

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN10, COL. TYPE: LC-CN, ID: LC-CN-07, INJ:100, UV
Number of samples.: 56
Calibration file..: 3CN1027B Last modified on 05-Nov-81 at 10:28:52
Method file.....: LCCN Last modified on 05-Nov-81 at 10:29:06
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: EXP L1A STD
Sample ID.....: 1-118:IA1
Sample type.....: Standard
Sample amount.....: 1.0000
Number of injections.....: 1
Bottle Number.....: 1

Retention time update.....: No update
Calibration level identifier.....: 1
Response factor update.....: Initialise

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 1.000
Dilution.....: 1.000
Amount (g or mL).....: 1.000

PEAK INFORMATION

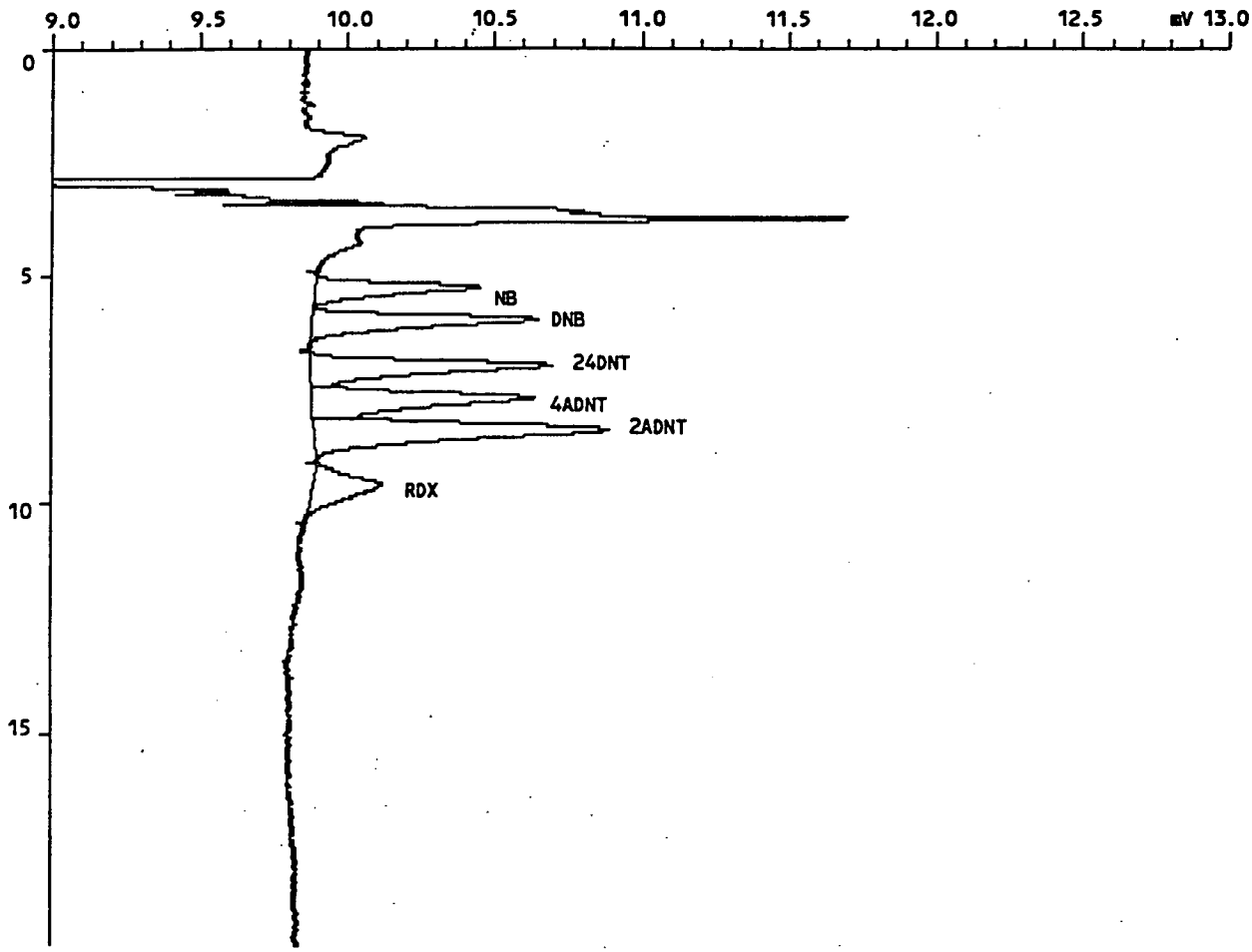
Peak	RT mins	Hght uV	Area uVs		ug/mL	Peak name
1	6.192	748	11396		0.043	TNB
2	6.608	519	9718		0.049	26DNT
3	7.392	654	11587		0.045	TNT
4	8.400	454	11271		0.051	3,4DNT
6	12.069	636	17029	TETRYL		
7	15.515	181	6993		0.063	HMX
Total		3192	67995		0.335	
Residual		76	1903		0.000	

LONG PLOT

Injection F: <MC3> 3 3CN1027,9,1

Sample name.....: EXP L1B STD
Sample ID.....: 1-123:IB1
INST ID: IN10, COL. TYPE: LC-CN, ID: LC-CN-07, INJ:100, UV

Acquired on 28-Oct-97 at 02:17:28
Reported on 05-Nov-97 at 10:37:26



INJECTION REPORT

Injection F: <MC3> 3 3CN1027,9,1

Acquired on 28-Oct-97 at 02:17:28
Modified on 05-Nov-81 at 10:28:52
Reported on 05-Nov-97 at 10:29:28

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN10, COL. TYPE: LC-CN, ID: LC-CN-07, INJ:100, UV
Number of samples.: 56
Calibration file..: 3CN1027B Last modified on 05-Nov-81 at 10:28:52
Method file.....: LCCN Last modified on 05-Nov-81 at 10:29:06
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: EXP L1B STD
Sample ID.....: 1-123:IB1
Sample type.....: Standard
Sample amount.....: 1.0000
Number of injections.....: 1
Bottle Number.....: 1

Retention time update.....: No update
Calibration level identifier.....: 1
Response factor update.....: Initialise

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 1.000
Dilution.....: 1.000
Amount (g or mL).....: 1.000

PEAK INFORMATION

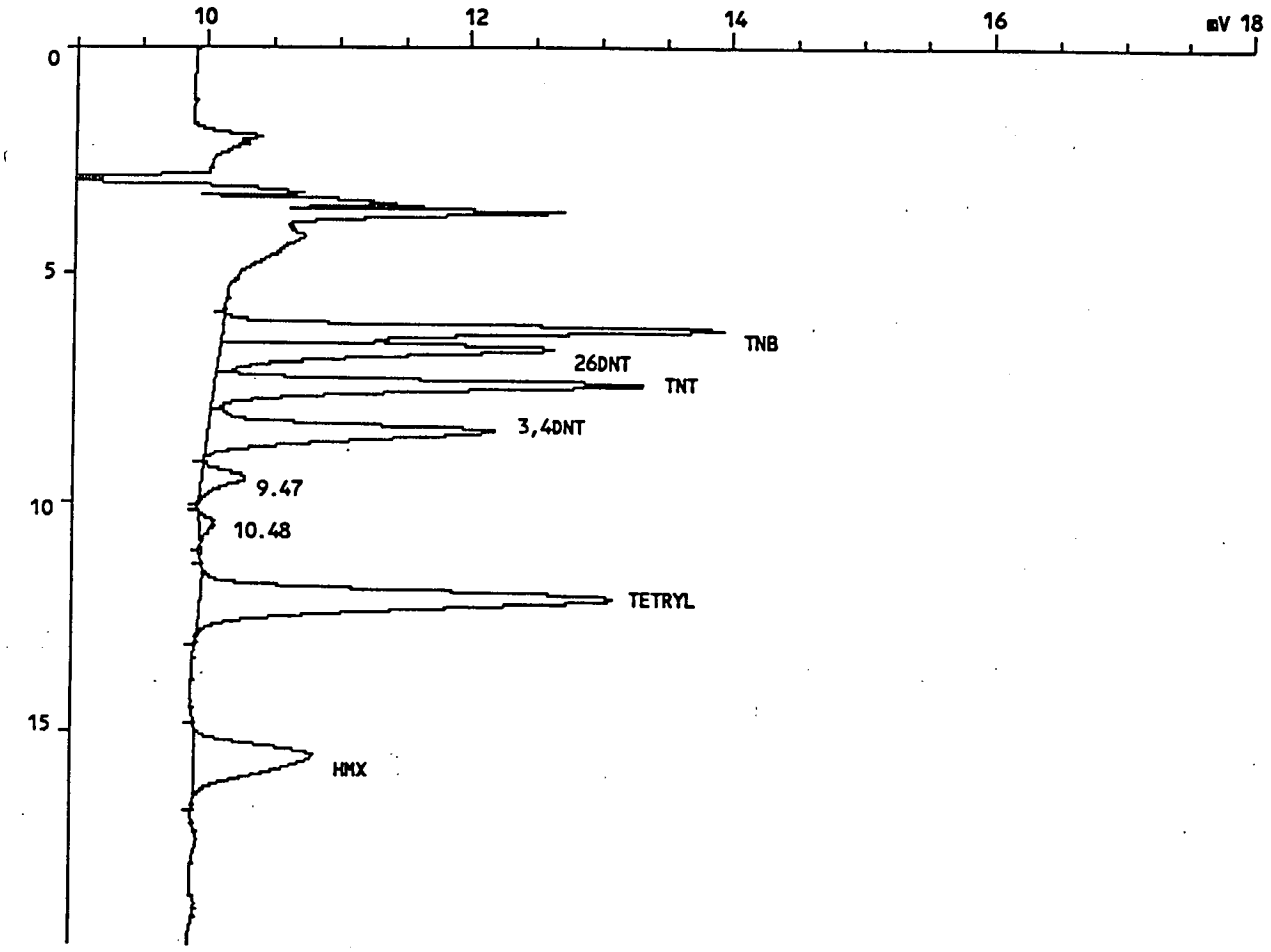
Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
1	5.253	567	9541	0.048	NB
2	5.957	766	13793	0.037	DNB
3	6.955	822	15984	0.047	24DNT
4	7.680	756	16579	0.087	4ADNT
5	8.379	999	24786	0.094	2ADNT
6	9.579	239	7609	0.054	RDX
Total		4150	88292	0.366	
Residual		0	0	0.000	

LONG PLOT

Injection F: <MC3> 3 3CN1027,5,1

Sample name.....: EXP L2A STD
Sample ID.....: 1-117:IA2
INST ID: IN10, COL. TYPE: LC-CN, ID: LC-CN-07, INJ:100, UV

Acquired on 27-Oct-97 at 23:32:03
Reported on 05-Nov-97 at 10:35:04



INJECTION REPORT

Injection F: <MC3> 3 3CN1027,5,1

Acquired on 27-Oct-97 at 23:32:03
Modified on 05-Nov-81 at 10:18:10
Reported on 05-Nov-97 at 10:31:42

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN10, COL. TYPE: LC-CN, ID: LC-CN-07, INJ:100, UV
Number of samples.: 56
Calibration file..: 3CN1027B Last modified on 05-Nov-81 at 10:28:52
Method file.....: LCCN Last modified on 05-Nov-81 at 10:29:06
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: EXP L2A STD
Sample ID.....: 1-117:IA2
Sample type.....: Standard
Sample amount.....: 1.0000
Number of injections.....: 1
Bottle Number.....: 1

Retention time update.....: No update
Calibration level identifier.....: 2
Response factor update.....: Initialise

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 1.000
Dilution.....: 1.000
Amount (g or mL).....: 1.000

PEAK INFORMATION

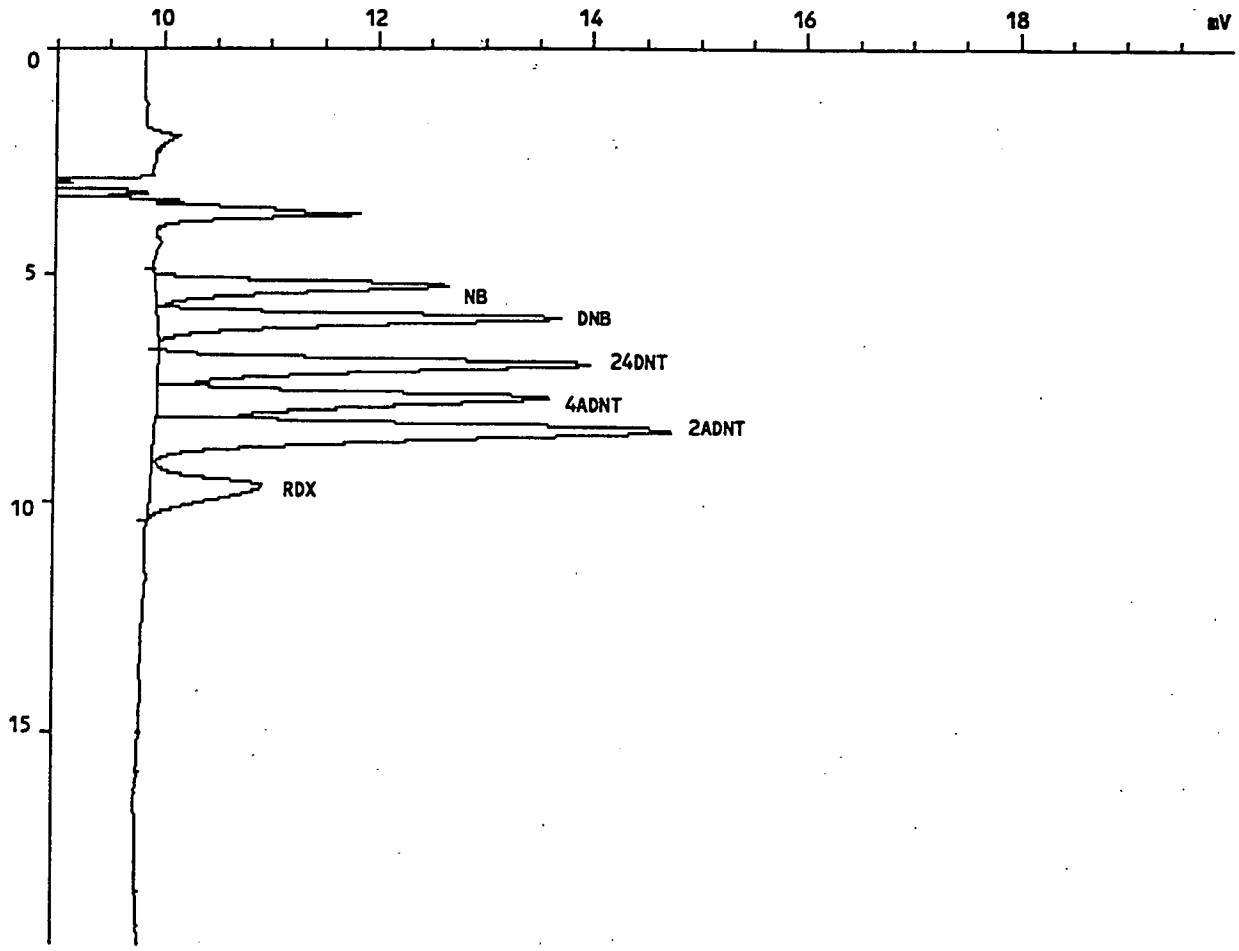
Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
1	6.192	3830	58180	0.222	TNB
2	6.608	2554	48630	0.244	26DNT
3	7.392	3267	57145	0.220	TNT
4	8.421	2188	53552	0.242	3,4DNT
7	12.080	3141	86380	0.429	TETRYL
8	15.568	899	37758	0.338	HMX
Total		15878	341646	1.695	
Residual		454	10099	0.000	

LONG PLOT

Injection F: <MC3> 3 3CN1027,10,1

Sample name.....: EXP L2B STD
Sample ID.....: 1-122:IB2
INST ID: IN10, COL. TYPE: LC-CN, ID: LC-CN-07, INJ:100, UV

Acquired on 28-Oct-97 at 02:58:49
Reported on 05-Nov-97 at 10:38:00



INJECTION REPORT

Injection F: <MC3> 3 3CN1027,10,1

Acquired on 28-Oct-97 at 02:58:49
Modified on 05-Nov-81 at 10:28:44
Reported on 05-Nov-97 at 10:29:44

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN10, COL. TYPE: LC-CN, ID: LC-CN-07, INJ:100, UV
Number of samples.: 56
Calibration file..: 3CN1027B Last modified on 05-Nov-81 at 10:28:52
Method file.....: LCCN Last modified on 05-Nov-81 at 10:29:06
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: EXP L2B STD
Sample ID.....: 1-122:IB2
Sample type.....: Standard
Sample amount.....: 1.0000
Number of injections.....: 1
Bottle Number.....: 1

Retention time update.....: No update
Calibration level identifier.....: 2
Response factor update.....: Initialise

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 1.000
Dilution.....: 1.000
Amount (g or mL).....: 1.000

PEAK INFORMATION

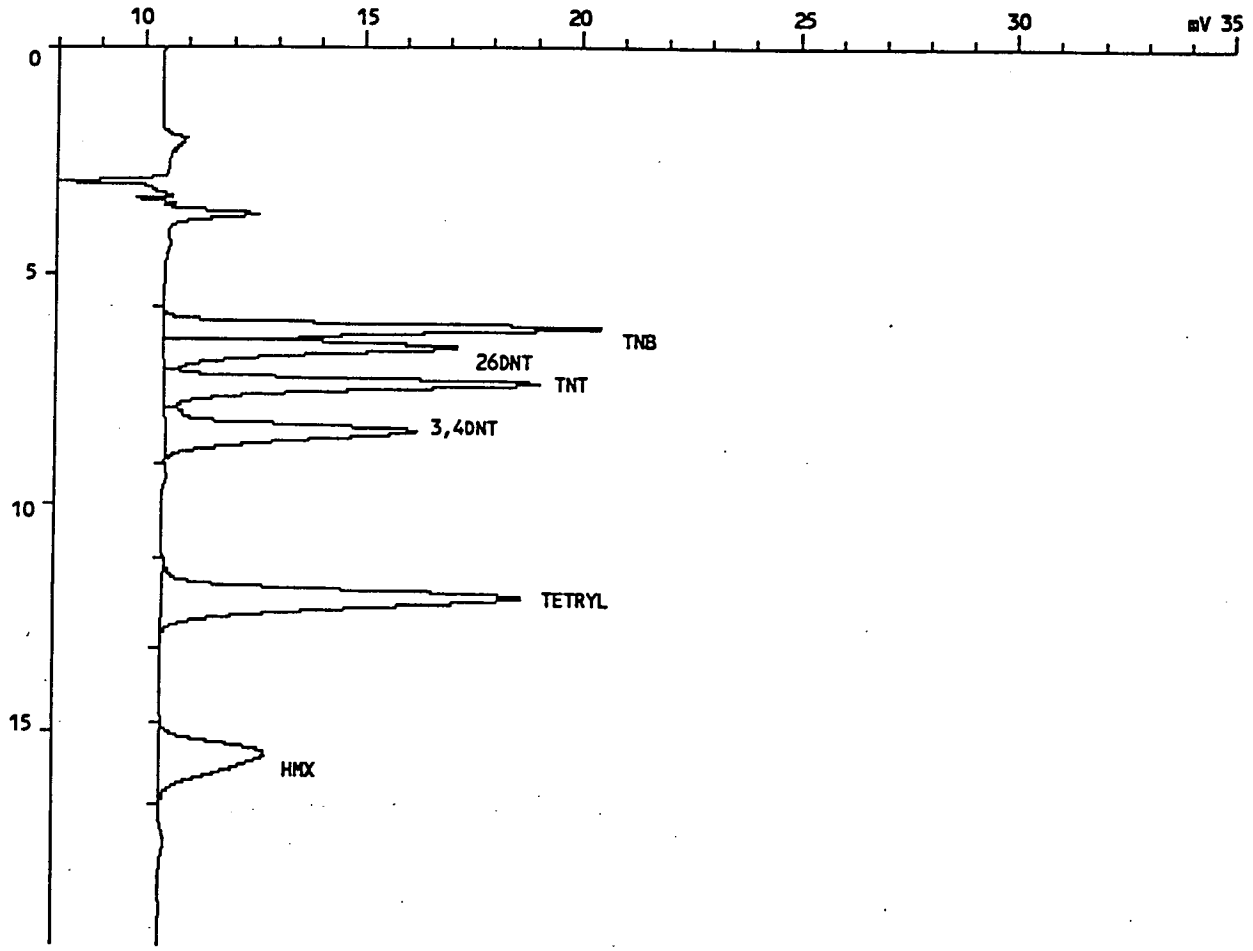
Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
1	5.243	2717	47743	0.238	NB
2	5.957	3757	69281	0.186	DNB
3	6.965	4021	80132	0.234	24DNT
4	7.691	3660	83388	0.438	4ADNT
5	8.395	4818	124064	0.471	2ADNT
6	9.653	1038	34218	0.242	RDX
Total		20010	438826	1.809	
Residual		0	0	0.000	

LONG PLOT

Injection F: <MC3> 3 3CN1027,6,1

Sample name.....: EXP L3A STD
Sample ID.....: 1-116:IA3
INST ID: IN10, COL. TYPE: LC-CN, ID: LC-CN-07, INJ:100, UV

Acquired on 28-Oct-97 at 00:13:24
Reported on 05-Nov-97 at 10:35:41



INJECTION REPORT

Injection F: <MC3> 3 3CN1027,6,1

Acquired on 28-Oct-97 at 00:13:24
Modified on 05-Nov-81 at 10:18:14
Reported on 05-Nov-97 at 10:31:25

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN10, COL. TYPE: LC-CN, ID: LC-CN-07, INJ:100, UV
Number of samples.: 56
Calibration file..: 3CN1027B Last modified on 05-Nov-81 at 10:28:52
Method file.....: LCCN Last modified on 05-Nov-81 at 10:29:06
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: EXP L3A STD
Sample ID.....: 1-116:IA3
Sample type.....: Standard
Sample amount.....: 1.0000
Number of injections.....: 1
Bottle Number.....: 1

Retention time update.....: No update
Calibration level identifier.....: 3
Response factor update.....: Initialise

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 1.000
Dilution.....: 1.000
Amount (g or mL).....: 1.000

PEAK INFORMATION

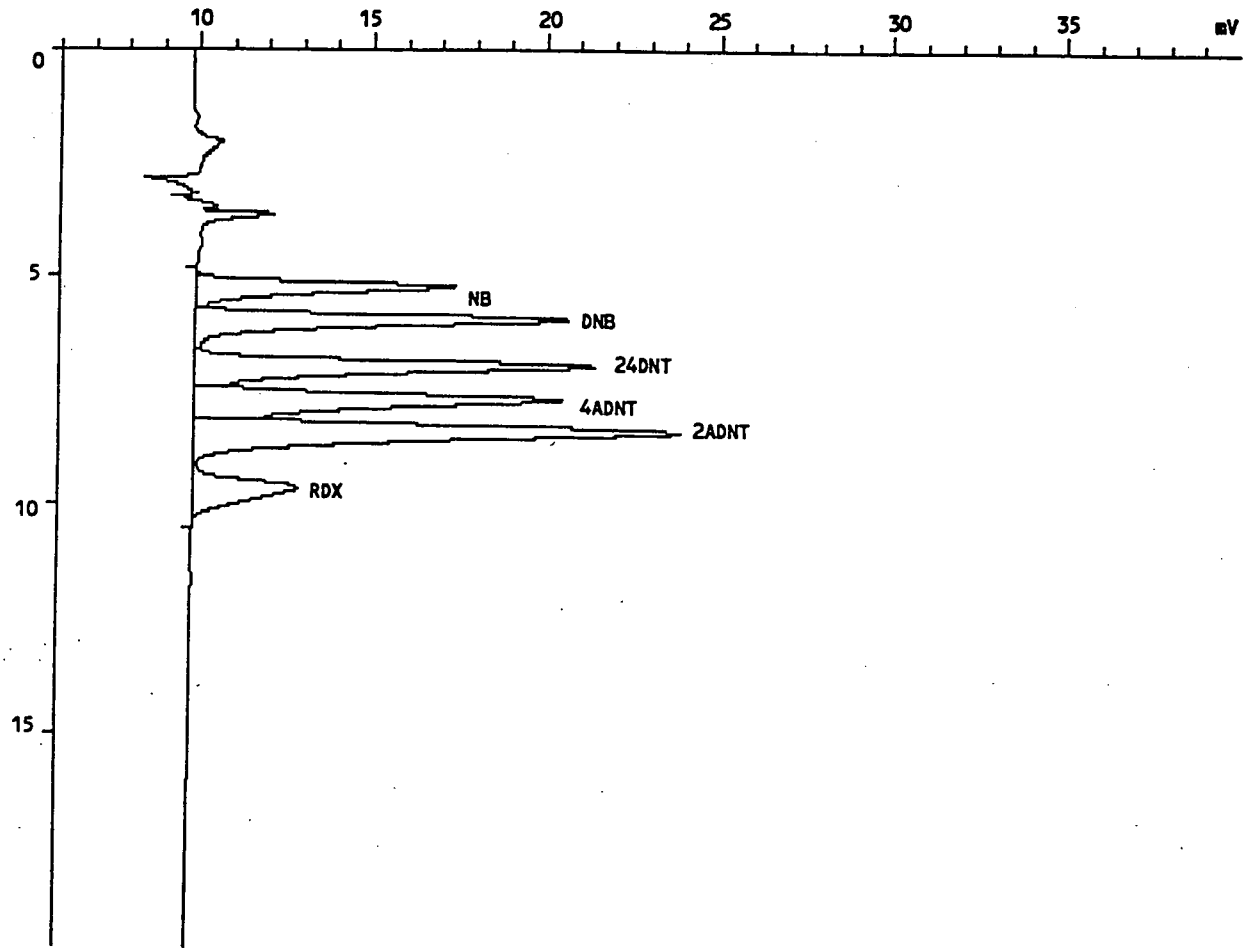
Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
1	6.165	10054	153133	0.584	TNB
2	6.587	6757	128325	0.643	26DNT
3	7.365	8606	151942	0.584	TNT
4	8.379	5749	141258	0.670	3,4DNT
5	12.037	8199	226027	1.124	TETRYL
6	15.493	2380	100405	0.897	HMX
Total		41746	901090	4.502	
Residual		0	0	0.000	

LONG PLOT

Injection F: <MC3> 3 3CN1027,11,1

Sample name.....: EXP L3B STD
Sample ID.....: 1-121:IB3
INST ID: IN10, COL. TYPE: LC-CN, ID: LC-CN-07, INJ:100, UV

Acquired on 28-Oct-97 at 03:40:10
Reported on 05-Nov-97 at 10:38:48



INJECTION REPORT

Injection F: <MC3> 3 3CN1027,11,1

Acquired on 28-Oct-97 at 03:40:10
Modified on 05-Nov-81 at 10:28:40
Reported on 05-Nov-97 at 10:30:01

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN10, COL. TYPE: LC-CN, ID: LC-CN-07, INJ:100, UV
Number of samples.: 56
Calibration file..: 3CN1027B Last modified on 05-Nov-81 at 10:28:52
Method file.....: LCCN Last modified on 05-Nov-81 at 10:29:06
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: EXP L3B STD
Sample ID.....: 1-121:IB3
Sample type.....: Standard
Sample amount.....: 1.0000
Number of injections.....: 1
Bottle Number.....: 1

Retention time update.....: No update
Calibration level identifier.....: 3
Response factor update.....: Initialise

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 1.000
Dilution.....: 1.000
Amount (g or mL).....: 1.000

PEAK INFORMATION

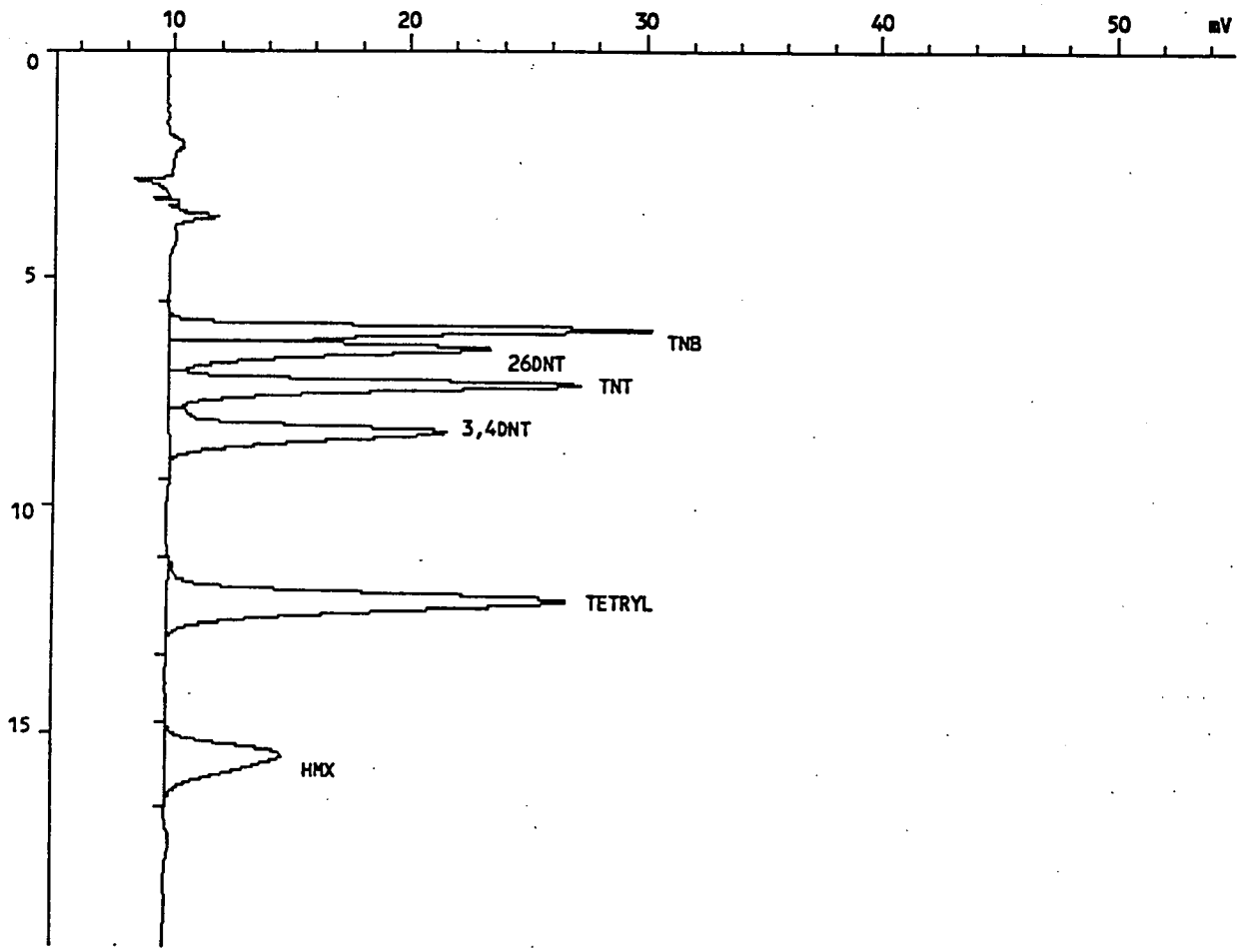
Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
1	5.232	7487	126646	0.631	NB
2	5.952	10782	191347	0.514	DNB
3	6.949	11557	221650	0.649	24DNT
4	7.685	10628	229380	1.204	4ADNT
5	8.373	14012	337472	1.281	2ADNT
6	9.643	3005	92737	0.657	RDX
Total		57471	1199233	4.935	
Residual		0	0	0.000	

LONG PLOT

Injection F: <MC3> 3 3CN1027,7,1

Sample name.....: EXP L4A STD
Sample ID.....: 1-115:IA4
INST ID: IN10, COL. TYPE: LC-CN, ID: LC-CN-07, INJ:100, UV

Acquired on 28-Oct-97 at 00:54:46
Reported on 05-Nov-97 at 10:36:15



INJECTION REPORT

Injection F: <MC3> 3 3CN1027,7,1

Acquired on 28-Oct-97 at 00:54:46
Modified on 05-Nov-81 at 10:18:30
Reported on 05-Nov-97 at 10:31:08

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN10, COL. TYPE: LC-CN, ID: LC-CN-07, INJ:100, UV
Number of samples.: 56
Calibration file..: 3CN1027B Last modified on 05-Nov-81 at 10:28:52
Method file.....: LCCN Last modified on 05-Nov-81 at 10:29:06
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: EXP L4A STD
Sample ID.....: 1-115:IA4
Sample type.....: Standard
Sample amount.....: 1.0000
Number of injections.....: 1
Bottle Number.....: 1

Retention time update.....: No update
Calibration level identifier.....: 4
Response factor update.....: Initialise

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 1.000
Dilution.....: 1.000
Amount (g or mL).....: 1.000

PEAK INFORMATION

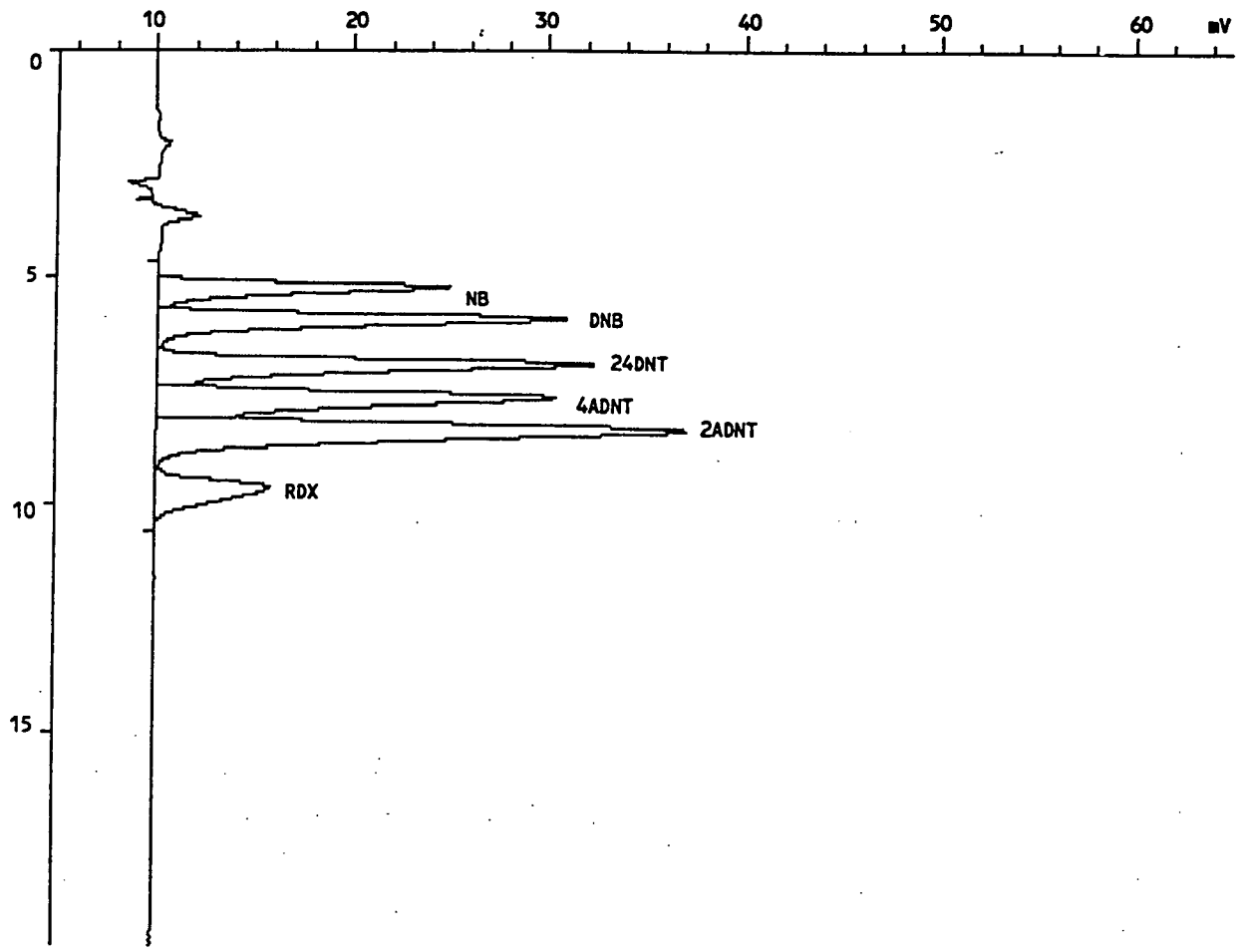
Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
1	6.165	20523	312380	1.203	TNB
2	6.587	13698	259192	1.256	26DNT
3	7.365	17542	310213	1.175	TNT
4	8.384	11758	290701	1.685	3,4DNT
5	12.043	16852	467032	2.338	TETRYL
6	15.493	4950	204417	1.791	HMX
Total		85321	1843935	9.448	
Residual		0	0	0.000	

LONG PLOT

Injection F: <MC3> 3 3CN1027,12,1

Sample name.....: EXP L4B STD
Sample ID.....: 1-120:IB4
INST ID: IN10, COL. TYPE: LC-CN, ID: LC-CN-07, INJ:100, UV

Acquired on 28-Oct-97 at 04:21:32
Reported on 05-Nov-97 at 10:39:24



INJECTION REPORT

Injection F: <MC3> 3 3CN1027,12,1

Acquired on 28-Oct-97 at 04:21:32
Modified on 05-Nov-81 at 10:28:36
Reported on 05-Nov-97 at 10:30:18

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN10, COL. TYPE: LC-CN, ID: LC-CN-07, INJ:100, UV
Number of samples.: 56
Calibration file..: 3CN1027B Last modified on 05-Nov-81 at 10:28:52
Method file.....: LCCN Last modified on 05-Nov-81 at 10:29:06
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: EXP L4B STD
Sample ID.....: 1-120:IB4
Sample type.....: Standard
Sample amount.....: 1.0000
Number of injections.....: 1
Bottle Number.....: 1

Retention time update.....: No update
Calibration level identifier.....: 4
Response factor update.....: Initialise

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 1.000
Dilution.....: 1.000
Amount (g or mL).....: 1.000

PEAK INFORMATION

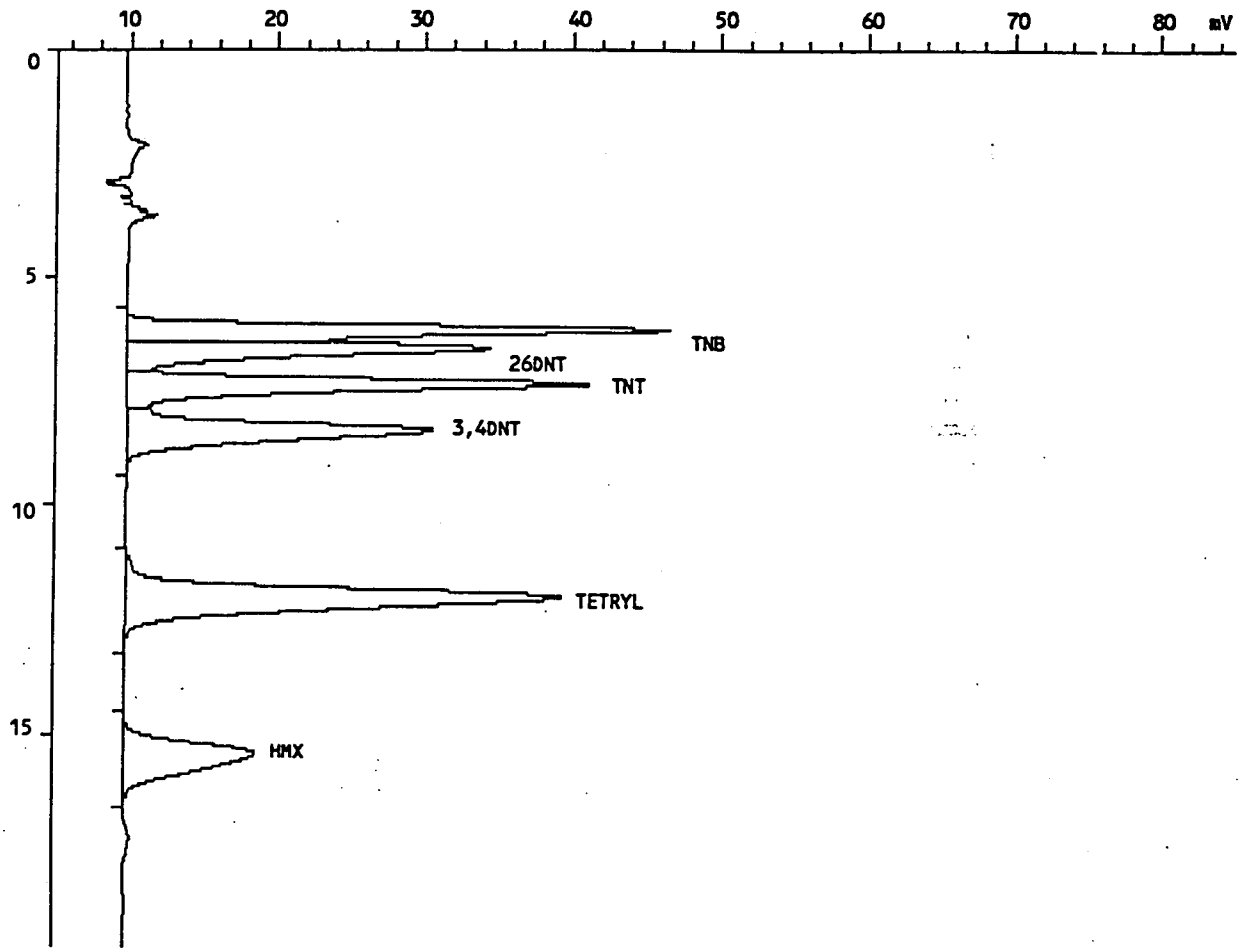
Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
1	5.221	14831	257911	1.285	NB
2	5.936	20843	381461	1.025	DNB
3	6.939	22258	438810	1.284	24DNT
4	7.675	20401	452776	2.376	4ADNT
5	8.357	26977	673302	2.556	2ADNT
6	9.637	5749	181518	1.286	RDX
Total		111060	2385778	9.811	
Residual		0	0	0.000	

LONG PLOT

Injection F: <MC3> 3 3CN1027,8,1

Sample name.....: EXP L5A STD
Sample ID.....: 1-114:IA5
INST ID: IN10, COL. TYPE: LC-CN, ID: LC-CN-07, INJ:100, UV

Acquired on 28-Oct-97 at 01:36:07
Reported on 05-Nov-97 at 10:36:52



INJECTION REPORT

Injection F: <MC3> 3 3CN1027,8,1

Acquired on 28-Oct-97 at 01:36:07
Modified on 05-Nov-81 at 10:18:24
Reported on 05-Nov-97 at 10:30:52

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN10, COL. TYPE: LC-CN, ID: LC-CN-07, INJ:100, UV
Number of samples.: 56
Calibration file..: 3CN1027B Last modified on 05-Nov-81 at 10:28:52
Method file.....: LCCN Last modified on 05-Nov-81 at 10:29:06
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: EXP L5A STD
Sample ID.....: 1-114:IA5
Sample type.....: Standard
Sample amount.....: 1.0000
Number of injections.....: 1
Bottle Number.....: 1

Retention time update.....: No update
Calibration level identifier.....: 5
Response factor update.....: Initialise

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 1.000
Dilution.....: 1.000
Amount (g or mL).....: 1.000

PEAK INFORMATION

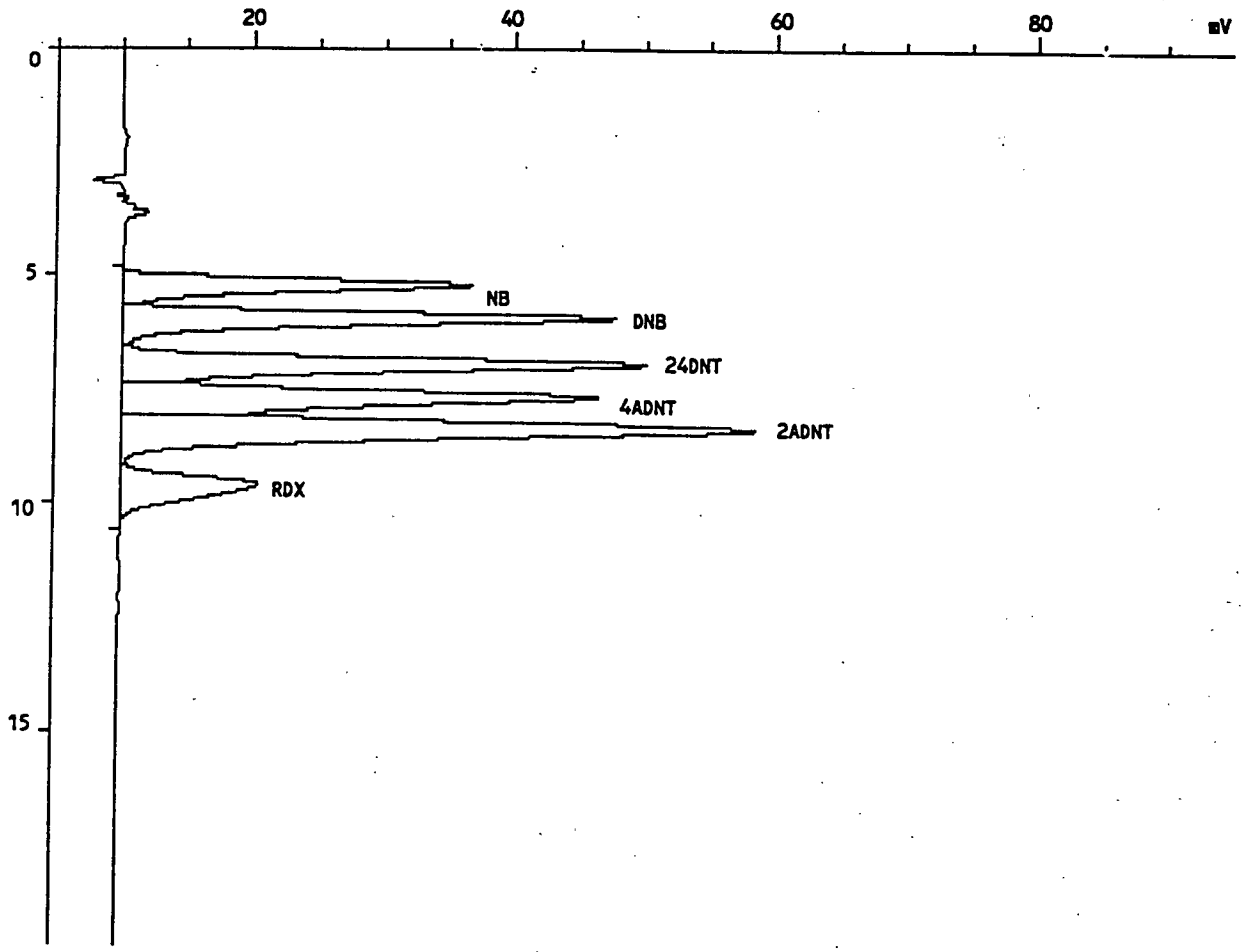
Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
1	6.149	36746	589348	2.270	TNB
2	6.560	24637	515564	2.499	26DNT
3	7.344	31368	604076	2.289	TNT
4	8.357	20766	567503	3.289	3,4DNT
5	12.000	29552	890076	4.456	TETRYL
6	15.435	8841	397348	3.481	HMX
Total		151910	3563916	18.283	
Residual		0	0	0.000	

LONG PLOT

Injection F: <MC3> 3 3CN1027,13,1

Sample name.....: EXP L5B STD
Sample ID.....: 1-119:IB5
INST ID: IN10, COL. TYPE: LC-CN, ID: LC-CN-07, INJ:100, UV

Acquired on 28-Oct-97 at 05:02:53
Reported on 05-Nov-97 at 10:40:03



INJECTION REPORT

Injection F: <MC3> 3 3CN1027,13,1

Acquired on 28-Oct-97 at 05:02:53
Modified on 05-Nov-81 at 10:28:30
Reported on 05-Nov-97 at 10:30:35

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN10, COL. TYPE: LC-CN, ID: LC-CN-07, INJ:100, UV
Number of samples.: 56
Calibration file..: 3CN1027B Last modified on 05-Nov-81 at 10:28:52
Method file.....: LCCN Last modified on 05-Nov-81 at 10:29:06
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: EXP L5B STD
Sample ID.....: 1-119:IB5
Sample type.....: Standard
Sample amount.....: 1.0000
Number of injections.....: 1
Bottle Number.....: 1

Retention time update.....: No update
Calibration level identifier.....: 5
Response factor update.....: Initialise

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 1.000
Dilution.....: 1.000
Amount (g or mL).....: 1.000

PEAK INFORMATION

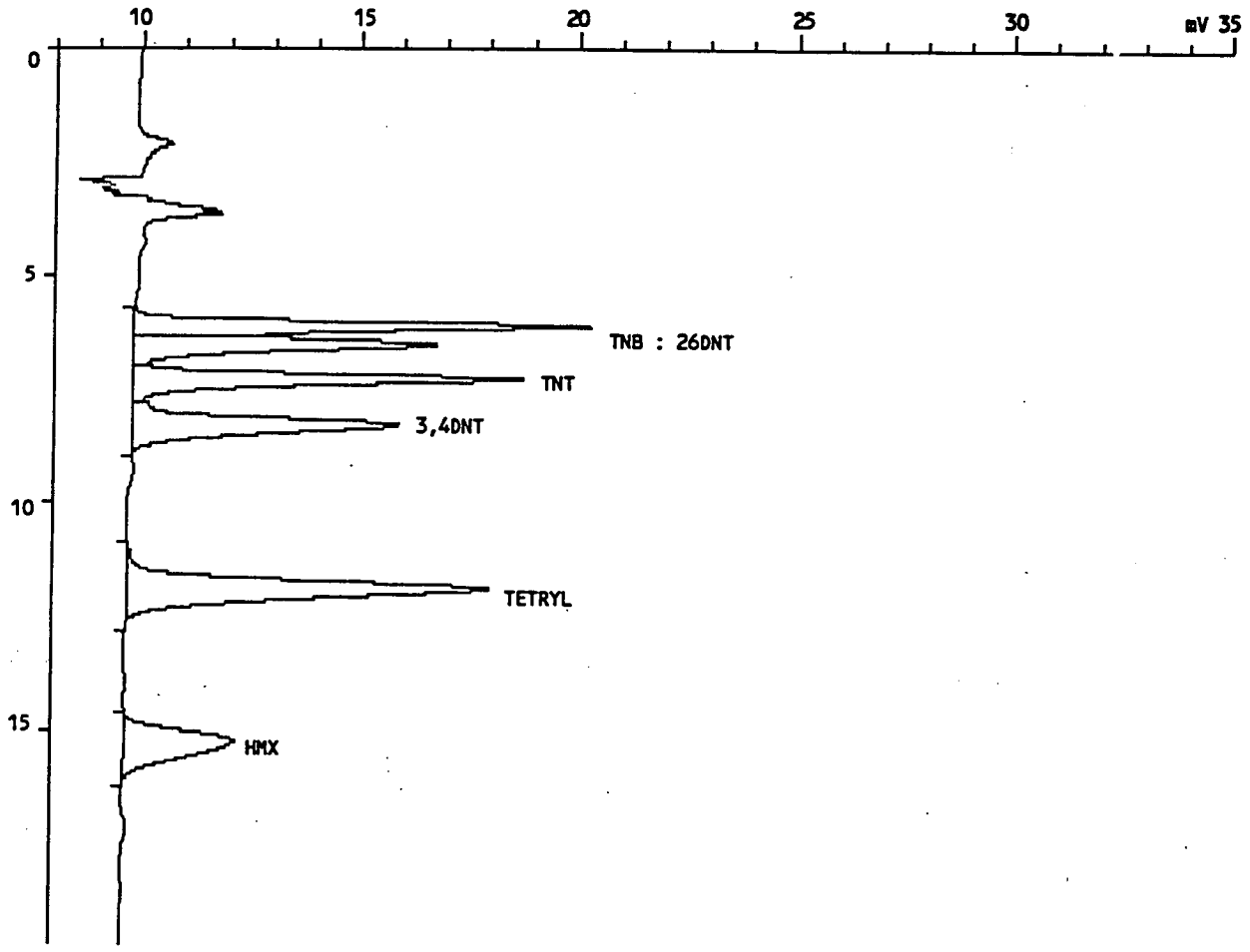
Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
1	5.211	26589	498467	2.483	NB
2	5.920	37679	738945	1.985	DNB
3	6.912	40079	847150	2.479	24DNT
4	7.643	36433	867343	4.551	4ADNT
5	8.325	48514	1308533	4.967	2ADNT
6	9.611	10403	349323	2.475	RDX
Total		199698	4609761	18.940	
Residual		0	0	0.000	

LONG PLOT

Injection F: <MC3> 3 3CN1027,28,1

Sample name.....: EXP L3A STD
Sample ID.....: 1-116:CA1
INST ID: IN10, COL. TYPE: LC-CN, ID: LC-CN-07, INJ:100, UV

Acquired on 28-Oct-97 at 15:21:01
Reported on 05-Nov-97 at 14:51:13



INJECTION REPORT

Injection F: <MC3> 3 3CN1027,28,1

Acquired on 28-Oct-97 at 15:21:01
Modified on 05-Nov-81 at 14:50:30
Reported on 05-Nov-97 at 14:50:35

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN10, COL. TYPE: LC-CN, ID: LC-CN-07, INJ:100, UV
Number of samples.: 56
Calibration file..: 3CN1027A Last modified on 05-Nov-81 at 10:52:08
Method file.....: LCCN Last modified on 05-Nov-81 at 11:44:20
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: EXP L3A STD
Sample ID.....: 1-116:CA1
Sample type.....: Standard
Sample amount.....: 1.0000
Number of injections.....: 1
Bottle Number.....: 1

Retention time update.....: No update
Calibration level identifier.....: 3
Response factor update.....: No update

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 1.000
Dilution.....: 1.000
Amount (g or mL).....: 1000.000

PEAK INFORMATION

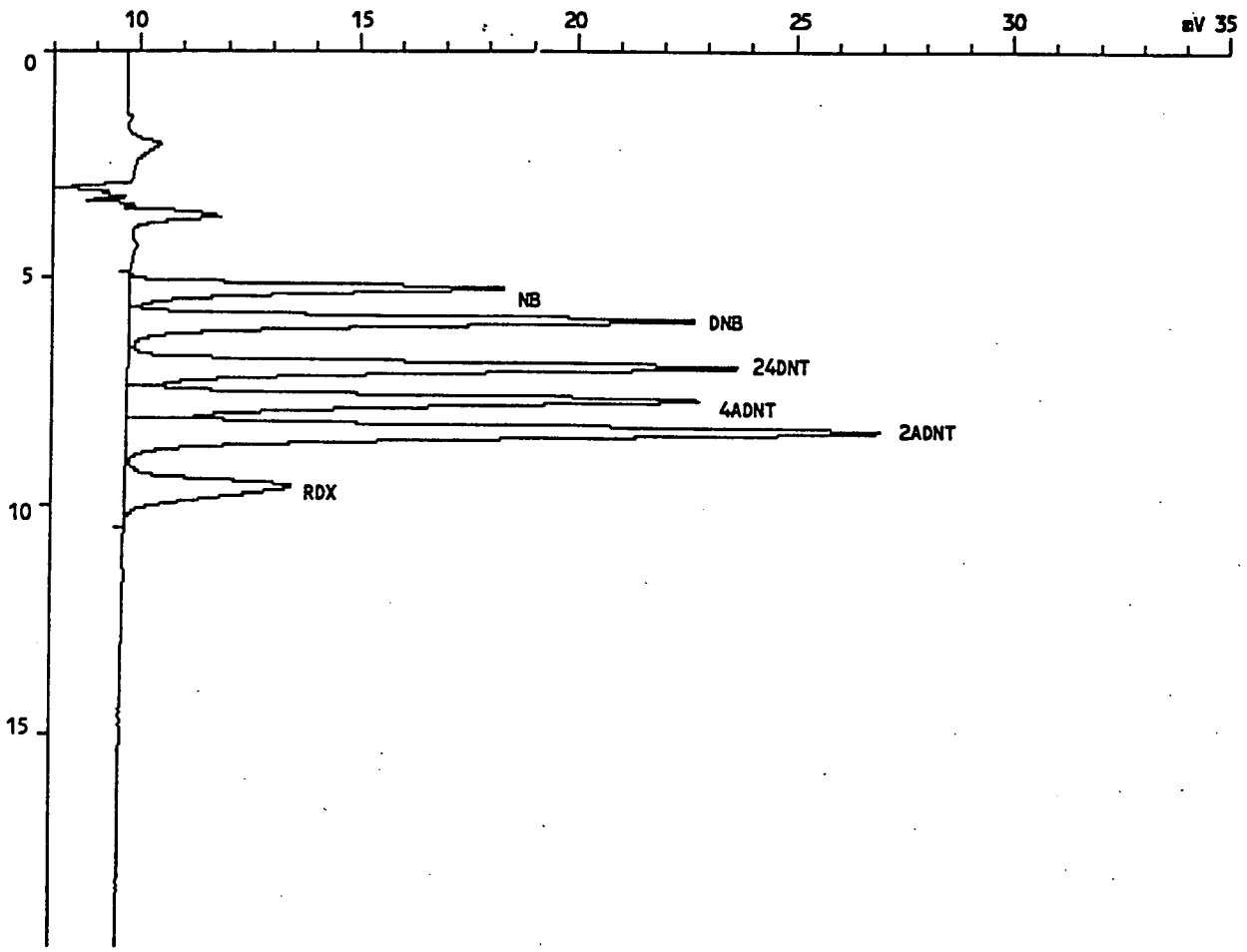
Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
1	6.107	10493	157651	0.609	TNB
2	6.523	6988	127429	0.632	26DNT
3	7.291	8969	154256	0.595	TNT
4	8.288	6126	144860	0.869	3,4DNT
5	11.877	8337	224172	1.135	TETRYL
6	15.216	2529	99981	0.904	HMX
Total		43442	908349	4.743	
Residual		0	0	0.000	

LONG PLOT

Injection F: <MC3> 3 3CN1027,29,1

Sample name.....: EXP L3B STD
Sample ID.....: 1-121:CB1
INST ID: IN10, COL. TYPE: LC-CN, ID: LC-CN-07, INJ:100, UV

Acquired on 28-Oct-97 at 16:02:23
Reported on 05-Nov-97 at 14:52:03



INJECTION REPORT

Injection F: <MC3> 3 3CN1027,29,1

Acquired on 28-Oct-97 at 16:02:23
Modified on 05-Nov-81 at 14:51:42
Reported on 05-Nov-97 at 14:51:45

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN10, COL. TYPE: LC-CN, ID: LC-CN-07, INJ:100, UV
Number of samples.: 56
Calibration file..: 3CN1027B Last modified on 05-Nov-81 at 10:42:36
Method file.....: LCCN Last modified on 05-Nov-81 at 14:51:10
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: EXP L3B STD
Sample ID.....: 1-121:CB1
Sample type.....: Standard
Sample amount.....: 1.0000
Number of injections.....: 1
Bottle Number.....: 1

Retention time update.....: No update
Calibration level identifier.....: 3
Response factor update.....: No update

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 1.000
Dilution.....: 1.000
Amount (g or mL).....: 1000.000

PEAK INFORMATION

Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
1	5.232	8571	129532	0.654	NB
2	5.936	12956	206054	0.565	DNB
3	6.923	13953	238847	0.709	24DNT
4	7.653	13083	247805	1.309	4ADNT
5	8.325	17274	361076	1.392	2ADNT
6	9.589	3773	100724	0.697	RDX
Total		69610	1284039	5.326	
Residual		0	0	0.000	

CALIBRATION FACTOR DATA
FORM 9A-1

EXPLOSIVE CALIBRATION - METHOD SW846-8330
Instrument ID: IN10
Column ID : LC-CN-07

Inj 1

Injection File Name Level 1 : 3CN1110,4
Injection File Name Level 2 : 3CN1110,5
Injection File Name Level 3 : 3CN1110,6
Injection File Name Level 4 : 3CN1110,7
Injection File Name Level 5 : 3CN1110,8

Calibration Date : 11/10/97
Number of Calibration Levels: 5
Line forced thru zero : Yes
Calculation Method : Area

COMPONENT NAME	Area Level 1	CAL FACTOR Level 1	Area Level 2	CAL FACTOR Level 2	Area Level 3	CAL FACTOR Level 3	Area Level 4	CAL FACTOR Level 4	Area Level 5	CAL FACTOR Level 5
TNB	11781	2561	58980	2564	155255	2700	308191	2680	619497	2693
26DNT	8772	1754	44149	1766	116357	1862	230693	1846	465237	1861
TNT	10570	2298	56350	2450	147745	2569	293190	2549	591113	2570
3,4DNT	9495	1356	52251	1493	135244	1639	270561	1640	546051	1655
TETRYL	16114	1790	80720	1794	220668	1961	430603	1914	854145	1898
HMX	7027	1004	35458	1013	95317	1089	191029	1092	381516	1090

CALIBRATION FACTOR DATA
FORM 9A-1

EXPLOSIVE CALIBRATION - METHOD SW846-8330
Instrument ID: IN10
Column ID : LC-CN-07

Inj 1

Injection File Name Level 1 : 3CN1110,9
Injection File Name Level 2 : 3CN1110,10
Injection File Name Level 3 : 3CN1110,11
Injection File Name Level 4 : 3CN1110,12
Injection File Name Level 5 : 3CN1110,13

Calibration Date : 11/10/97
Number of Calibration Levels: 5
Line forced thru zero : Yes
Calculation Method : Area

COMPONENT NAME	Area Level 1	CAL FACTOR Level 1	Area Level 2	CAL FACTOR Level 2	Area Level 3	CAL FACTOR Level 3	Area Level 4	CAL FACTOR Level 4	Area Level 5	CAL FACTOR Level 5
NB	9532	1906	47971	1919	127092	2033	249301	1994	502730	2011
DNB	13648	3412	69049	3452	184705	3694	357696	3577	724567	3623
24DNT	15589	3118	81178	3247	216991	3472	419300	3354	848812	3395
4ADNT	15859	1724	82044	1784	218970	1904	425980	1852	846810	1841
2ADNT	24040	2404	121665	2433	329591	2637	638249	2553	1276751	2554
RDX	5379	1076	30900	1236	86418	1383	168546	1348	336931	1348

CALIBRATION FACTOR DATA
FORM 9B

EXPLOSIVE CALIBRATION - METHOD SW846-8330
Instrument ID: IN10
Column ID : LC-CN-07

Inj 1

Injection File Name Level 1 : 3CN1110,4
Injection File Name Level 2 : 3CN1110,5
Injection File Name Level 3 : 3CN1110,6
Injection File Name Level 4 : 3CN1110,7
Injection File Name Level 5 : 3CN1110,8

Calibration Date : 11/10/97
Number of Calibration Levels: 5
Line forced thru zero : Yes
Calculation Method : Area

Component Name	Mean Cal Factor	% RSD	Corr. Coeff.	Slope area/amt	Intercept
TNB	2640	2.68	0.99999	2690	0.000
26DNT	1818	2.92	0.99998	1857	0.000
TNT	2487	4.70	0.99999	2565	0.000
3,4DNT	1557	8.35	0.99997	1650	0.000
TETRYL	1872	4.07	0.99992	1903	0.000
HMX	1058	4.25	0.99998	1090	0.000

CALIBRATION FACTOR DATA
FORM 9B

EXPLOSIVE CALIBRATION - METHOD SW846-8330
Instrument ID: IN10
Column ID : LC-CN-07

Inj 1
Injection File Name Level 1 : 3CN1110,9
Injection File Name Level 2 : 3CN1110,10
Injection File Name Level 3 : 3CN1110,11
Injection File Name Level 4 : 3CN1110,12
Injection File Name Level 5 : 3CN1110,13

Calibration Date : 11/10/97
Number of Calibration Levels: 5
Line forced thru zero : Yes
Calculation Method : Area

Component Name	Mean Cal Factor	% RSD	Corr. Coeff.	Slope area/amt	Intercept
NB	1973	2.88	0.99997	2008	0.000
DNB	3552	3.31	0.99994	3616	0.000
24DNT	3317	4.16	0.99994	3390	0.000
4ADNT	1821	3.80	0.99994	1846	0.000
2ADNT	2516	3.81	0.99993	2557	0.000
RDX	1278	9.85	0.99992	1349	0.000

CALIBRATION FACTOR DATA
FORM 9C

Initial Calibration Date: 11/10/97
Continuing Calibration #: 2
Continuing Cal Date : 11/11/97
Continuing Cal Level : 3
Instrument ID : IN10
Column ID : LC-CN-07
Injection File Name : 3CN1110B,1
Calculation Mode : Area

Component Name	Response Cont Std	Cont CF	Init CF Mean	%D	Init CF Slope	%D
TNB	164456	2860	2640	8.35	2690	6.31
26DNT	122214	1955	1818	7.58	1857	5.28
TNT	155852	2710	2487	8.97	2565	5.66
3,4DNT	142351	1725	1557	10.85	1650	4.58
TETRYL	229110	2037	1872	8.82	1903	6.99
HMX	101524	1160	1058	9.71	1090	6.47

CALIBRATION FACTOR DATA
FORM 9C

Initial Calibration Date: 11/10/97
Continuing Calibration #: 2
Continuing Cal Date : 11/11/97
Continuing Cal Level : 3
Instrument ID : IN10
Column ID : LC-CN-07
Injection File Name : 3CN1110B,2
Calculation Mode : Area

Component Name	Response Cont Std	Cont CF	Init CF Mean	%D	Init CF Slope	%D
NB	128794	2061	1973	4.46	2008	2.61
DNB	190129	3803	3552	7.06	3616	5.15
24DNT	223032	3569	3317	7.57	3390	5.26
4ADNT	226632	1971	1821	8.23	1846	6.78
2ADNT	337989	2704	2516	7.46	2557	5.76
RDX	87839	1405	1278	9.96	1349	4.21

CALIBRATION FACTOR DATA
FORM 9C

Initial Calibration Date: 11/10/97
Continuing Calibration #: 3
Continuing Cal Date : 11/12/97
Continuing Cal Level : 3
Instrument ID : IN10
Column ID : LC-CN-07
Injection File Name : 3CN1110B,11
Calculation Mode : Area

Component Name	Response Cont Std	Cont CF	Init CF Mean	%D	Init CF Slope	%D
TNB	169569	2949	2640	11.71	2690	9.62
26DNT	127596	2042	1818	12.31	1857	9.91
TNT	163573	2845	2487	14.37	2565	10.90
3,4DNT	146583	1777	1557	14.14	1650	7.69
TETRYL	228788	2034	1872	8.66	1903	6.84
HMX	102788	1175	1058	11.08	1090	7.80

CALIBRATION FACTOR DATA
FORM 9C

Initial Calibration Date: 11/10/97
Continuing Calibration #: 3
Continuing Cal Date : 11/12/97
Continuing Cal Level : 3
Instrument ID : IN10
Column ID : LC-CN-07
Injection File Name : 3CN1110B,12
Calculation Mode : Area

Component Name	Response Cont Std	Cont CF	Init CF Mean	%D	Init CF Slope	%D
NB	127817	2045	1973	3.66	2008	1.84
DNB	190776	3816	3552	7.43	3616	5.51
24DNT	223948	3583	3317	8.02	3390	5.69
4ADNT	226774	1972	1821	8.30	1846	6.84
2ADNT	339108	2713	2516	7.82	2557	6.11
RDX	88162	1411	1278	10.36	1349	4.59

CALIBRATION FACTOR DATA
FORM 9D

EXPLOSIVE CALIBRATION - METHOD SW846-8330

jection volume = 100uL

Amount Units = ng

COMPONENT NAME	AMT ON COL Level 1	AMT ON COL Level 2	AMT ON COL Level 3	AMT ON COL Level 4	AMT ON COL Level 5
TNB	4.600	23.000	57.500	115.000	230.000
26DNT	5.000	25.000	62.500	125.000	250.000
TNT	4.600	23.000	57.500	115.000	230.000
3,4DNT	7.000	35.000	82.500	165.000	330.000
TETRYL	9.000	45.000	112.500	225.000	450.000
HMX	7.000	35.000	87.500	175.000	350.000

CALIBRATION FACTOR DATA
FORM 9D

EXPLOSIVE CALIBRATION - METHOD SW846-8330

ıjection volume = 100uL

Amount Units = ng

COMPONENT NAME	AMT ON COL Level 1	AMT ON COL Level 2	AMT ON COL Level 3	AMT ON COL Level 4	AMT ON COL Level 5
NB	5.000	25.000	62.500	125.000	250.000
DNB	4.000	20.000	50.000	100.000	200.000
24DNT	5.000	25.000	62.500	125.000	250.000
4ADNT	9.200	46.000	115.000	230.000	460.000
2ADNT	10.000	50.000	125.000	250.000	500.000
RDX	5.000	25.000	62.500	125.000	250.000

CALIBRATION FACTOR DATA
FORM 9E

EXPLOSIVE CALIBRATION - METHOD SW846-8330

:strument ID : IN10
olumn ID : LC-CN-07
Calibration Date : 11/10/97
Number of Calibration Levels: 5

COMPONENT NAME	RT Level 1	RT Level 2	RT Level 3	RT Level 4	RT Level 5
TNB	6.34	6.34	6.34	6.36	6.34
26DNT	6.90	6.92	6.92	6.94	6.92
TNT	7.70	7.70	7.71	7.73	7.71
3,4DNT	8.94	8.94	8.95	8.98	8.95
TETRYL	13.12	13.16	13.18	13.23	13.20
HMX	17.81	17.93	17.96	18.04	17.99

COMPONENT NAME	RT Cont CA1	RT Cont CA2	RT Cont CA3	RT Cont CA4
TNB	6.34	6.33	6.36	6.34
26DNT	6.92	6.91	6.95	6.92
TNT	7.70	7.69	7.73	7.69
3,4DNT	8.94	8.91	8.99	8.92
TETRYL	13.15	13.08	13.21	13.07
HMX	17.87	17.72	17.92	17.63

COMPONENT NAME	AVE RT	STD_DEV	3X SD	AVE+-3X SD
TNB	6.34	0.009	0.028	6.31 - 6.37
26DNT	6.92	0.016	0.048	6.87 - 6.97
TNT	7.71	0.015	0.044	7.66 - 7.75
3,4DNT	8.95	0.022	0.067	8.88 - 9.02
TETRYL	13.16	0.050	0.151	13.01 - 13.31
HMX	17.90	0.119	0.358	17.54 - 18.26

CALIBRATION FACTOR DATA
FORM 9E

EXPLOSIVE CALIBRATION - METHOD SW846-8330

:strument ID : IN10
olumn ID : LC-CN-07
Calibration Date : 11/10/97
Number of Calibration Levels: 5

COMPONENT NAME	RT Level 1	RT Level 2	RT Level 3	RT Level 4	RT Level 5
NB	5.42	5.41	5.41	5.42	5.39
DNB	6.19	6.19	6.20	6.21	6.18
24DNT	7.31	7.33	7.33	7.34	7.32
4ADNT	8.31	8.29	8.31	8.33	8.30
2ADNT	9.06	9.07	9.08	9.09	9.07
RDX	10.61	10.60	10.63	10.65	10.64

COMPONENT NAME	RT Cont CA1	RT Cont CA2	RT Cont CA3	RT Cont CA4
NB	5.41	5.38	5.41	5.39
DNB	6.19	6.15	6.19	6.16
24DNT	7.31	7.27	7.32	7.27
4ADNT	8.27	8.21	8.29	8.22
2ADNT	9.03	8.96	9.04	8.97
RDX	10.56	10.46	10.58	10.46

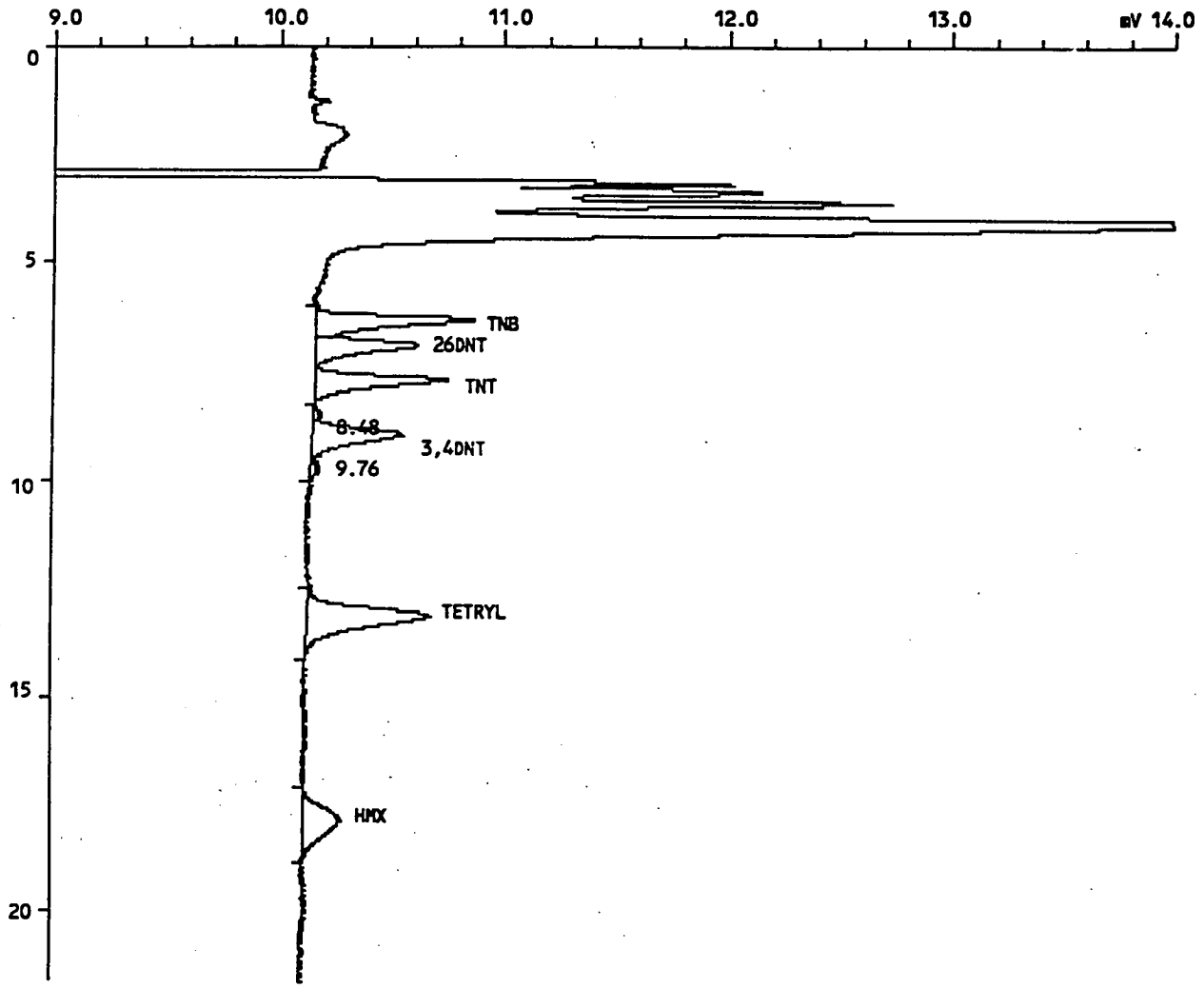
COMPONENT NAME	AVE RT	STD_DEV	3X SD	AVE+-3X SD
NB	5.41	0.015	0.044	5.36 - 5.45
DNB	6.19	0.017	0.052	6.13 - 6.24
24DNT	7.32	0.022	0.066	7.25 - 7.38
4ADNT	8.29	0.036	0.108	8.18 - 8.40
2ADNT	9.05	0.042	0.125	8.93 - 9.18
RDX	10.59	0.063	0.188	10.41 - 10.78

LONG PLOT

Injection F: <MC3> 3 3CN1110,4,1

Sample name.....: EXP L1A STD
Sample ID.....: 1-144:IA1
INST ID: IN10, COL. TYPE: LC-CN, ID: LC-CN-07, INJ:100, UV

Acquired on 10-Nov-97 at 19:53:05
Reported on 21-Nov-97 at 09:18:47



INJECTION REPORT

Injection F: <MC3> 3 3CN1110,4,1

Acquired on 10-Nov-97 at 19:53:05
Modified on 21-Nov-81 at 08:09:04
Reported on 21-Nov-97 at 08:13:11

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN10, COL. TYPE: LC-CN, ID: LC-CN-07, INJ:100, UV
Number of samples.: 30
Calibration file..: 3CN1110B Last modified on 21-Nov-81 at 08:10:12
Method file.....: LCCN Last modified on 21-Nov-81 at 08:10:24
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: EXP L1A STD
Sample ID.....: 1-144:IA1
Sample type.....: Standard
Sample amount.....: 1.0000
Number of injections.....: 1
Bottle Number.....: 1

Retention time update.....: No update
Calibration level identifier.....: 1
Response factor update.....: Initialise

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 1.000
Dilution.....: 1.000
Amount (g or mL).....: 1.000

PEAK INFORMATION

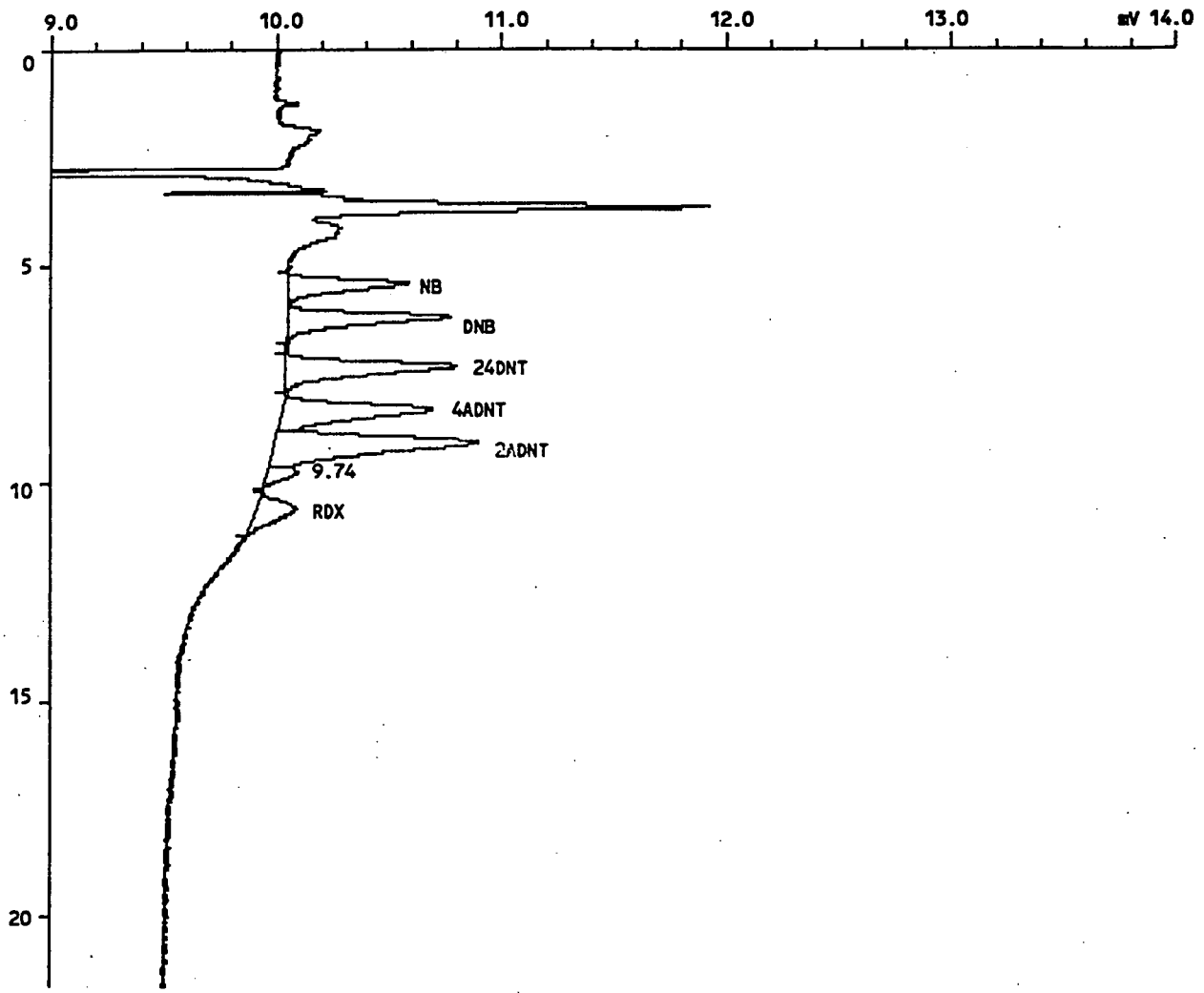
Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
1	6.341	719	11781	0.044	TNB
2	6.896	468	8772	0.047	26DNT
3	7.696	601	10570	0.041	TNT
5	8.939	402	9495	0.025	3,4DNT
7	13.120	548	16114	0.085	TETRYL
8	17.808	165	7027	0.064	HMX
Total		2902	63760	0.306	
Residual		67	890	0.000	

LONG PLOT

Injection F: <MC3> 3 3CN1110,9,1

Sample name.....: EXP L1B STD
Sample ID.....: 1-149:IB1
INST ID: IN10, COL. TYPE: LC-CN, ID: LC-CN-07, INJ:100, UV

Acquired on 10-Nov-97 at 23:19:51
Reported on 21-Nov-97 at 09:22:18



INJECTION REPORT

Injection F: <MC3> 3 3CN1110,9,1

Acquired on 10-Nov-97 at 23:19:51
Modified on 21-Nov-81 at 08:09:50
Reported on 21-Nov-97 at 08:11:47

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN10, COL. TYPE: LC-CN, ID: LC-CN-07, INJ:100, UV
Number of samples.: 30
Calibration file..: 3CN1110B Last modified on 21-Nov-81 at 08:10:12
Method file.....: LCCN Last modified on 21-Nov-81 at 08:10:24
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: EXP L1B STD
Sample ID.....: 1-149:IB1
Sample type.....: Standard
Sample amount.....: 1.0000
Number of injections.....: 1
Bottle Number.....: 1

Retention time update.....: No update
Calibration level identifier.....: 1
Response factor update.....: Initialise

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 1.000
Dilution.....: 1.000
Amount (g or mL).....: 1.000

PEAK INFORMATION

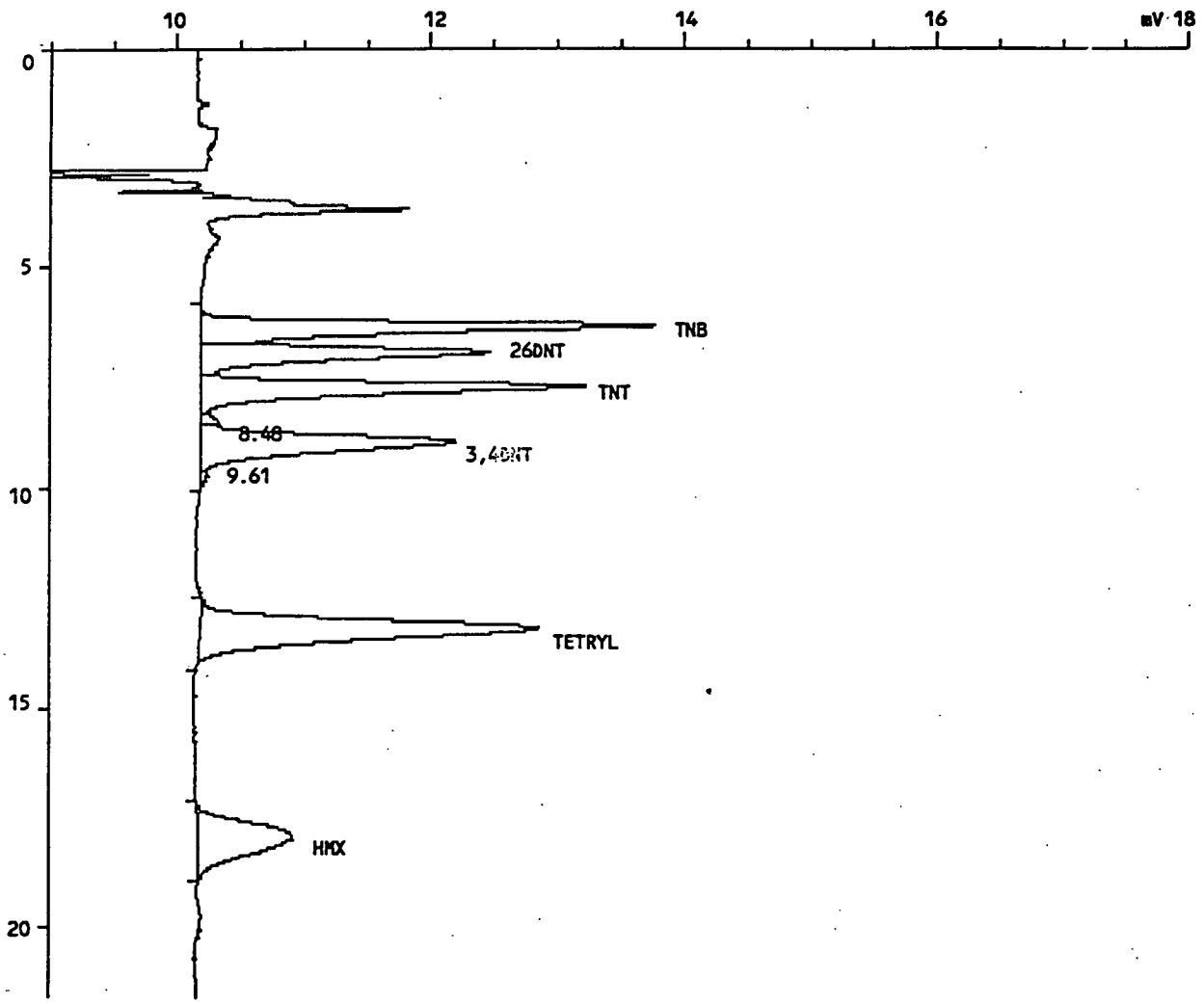
Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
1	5.424	542	9532	0.047	NB
2	6.192	733	13648	0.038	DNB
3	7.312	767	15589	0.046	24DNT
4	8.315	678	15859	0.086	4ADNT
5	9.061	911	24040	0.094	2ADNT
7	10.613	182	5379	0.040	RDX
Total		3812	84046	0.351	
Residual		138	2780	0.000	

LONG PLOT

Injection F: <MC3> 3 3CN1110,5,1

Sample name.....: EXP L2A STD
Sample ID.....: 1-143:IA2
INST ID: IN10, COL. TYPE: LC-CN, ID: LC-CN-07, INJ:100, UV

Acquired on 10-Nov-97 at 20:34:26
Reported on 21-Nov-97 at 09:19:37



INJECTION REPORT

Injection F: <MC3> 3 3CN1110,5,1

Acquired on 10-Nov-97 at 20:34:26
Modified on 21-Nov-81 at 08:09:08
Reported on 21-Nov-97 at 08:12:54

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN10, COL. TYPE: LC-CN, ID: LC-CN-07, INJ:100, UV
Number of samples.: 30
Calibration file..: 3CN1110B Last modified on 21-Nov-81 at 08:10:12
Method file.....: LCCN Last modified on 21-Nov-81 at 08:10:24
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: EXP L2A STD
Sample ID.....: 1-143:IA2
Sample type.....: Standard
Sample amount.....: 1.0000
Number of injections.....: 1
Bottle Number.....: 1

Retention time update.....: No update
Calibration level identifier.....: 2
Response factor update.....: Initialise

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 1.000
Dilution.....: 1.000
Amount (g or mL).....: 1.000

PEAK INFORMATION

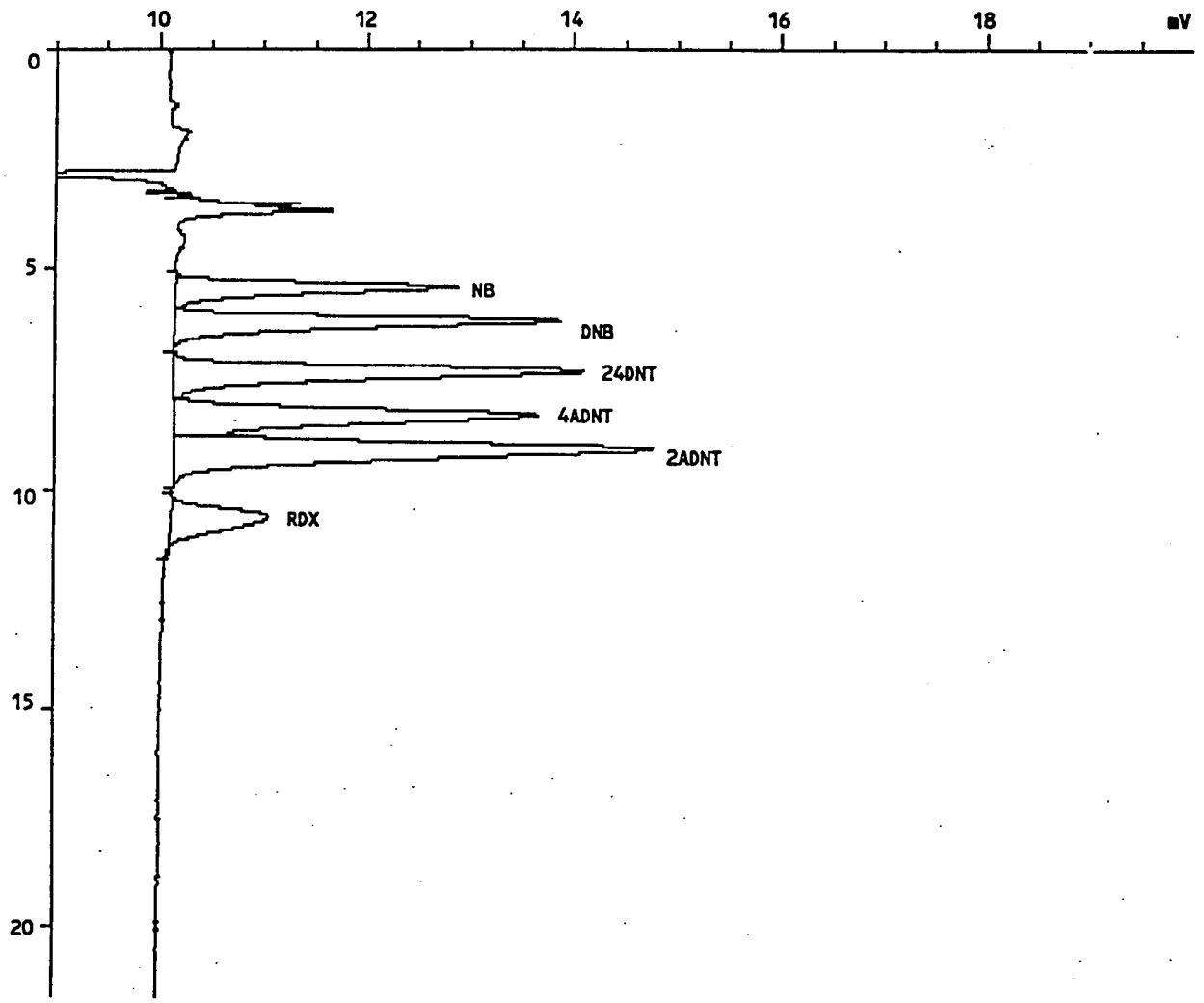
Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
1	6.336	3586	58980	0.219	TNB
2	6.917	2285	44149	0.238	26DNT
3	7.701	3031	56350	0.220	TNT
5	8.944	2020	52251	0.136	3,4DNT
7	13.157	2679	80720	0.424	TETRYL
8	17.931	754	35458	0.325	HMX
Total		14355	327909	1.562	
Residual		209	2428	0.000	

LONG PLOT

Injection F: <MC3> 3 3CN1110,10,1

Sample name.....: EXP L2B STD
Sample ID.....: 1-148:IB2
INST ID: IN10, COL. TYPE: LC-CN, ID: LC-CN-07, INJ:100, UV

Acquired on 11-Nov-97 at 00:01:12
Reported on 21-Nov-97 at 09:22:58



INJECTION REPORT

Injection F: <MC3> 3 3CN1110,10,1

Acquired on 11-Nov-97 at 00:01:12
Modified on 21-Nov-81 at 08:09:54
Reported on 21-Nov-97 at 08:11:30

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN10, COL. TYPE: LC-CN, ID: LC-CN-07, INJ:100, UV
Number of samples.: 30
Calibration file..: 3CN1110B Last modified on 21-Nov-81 at 08:10:12
Method file.....: LCCN Last modified on 21-Nov-81 at 08:10:24
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: EXP L2B STD
Sample ID.....: 1-148:IB2
Sample type.....: Standard
Sample amount.....: 1.0000
Number of injections.....: 1
Bottle Number.....: 1

Retention time update.....: No update
Calibration level identifier.....: 2
Response factor update.....: Initialise

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 1.000
Dilution.....: 1.000
Amount (g or mL).....: 1.000

PEAK INFORMATION

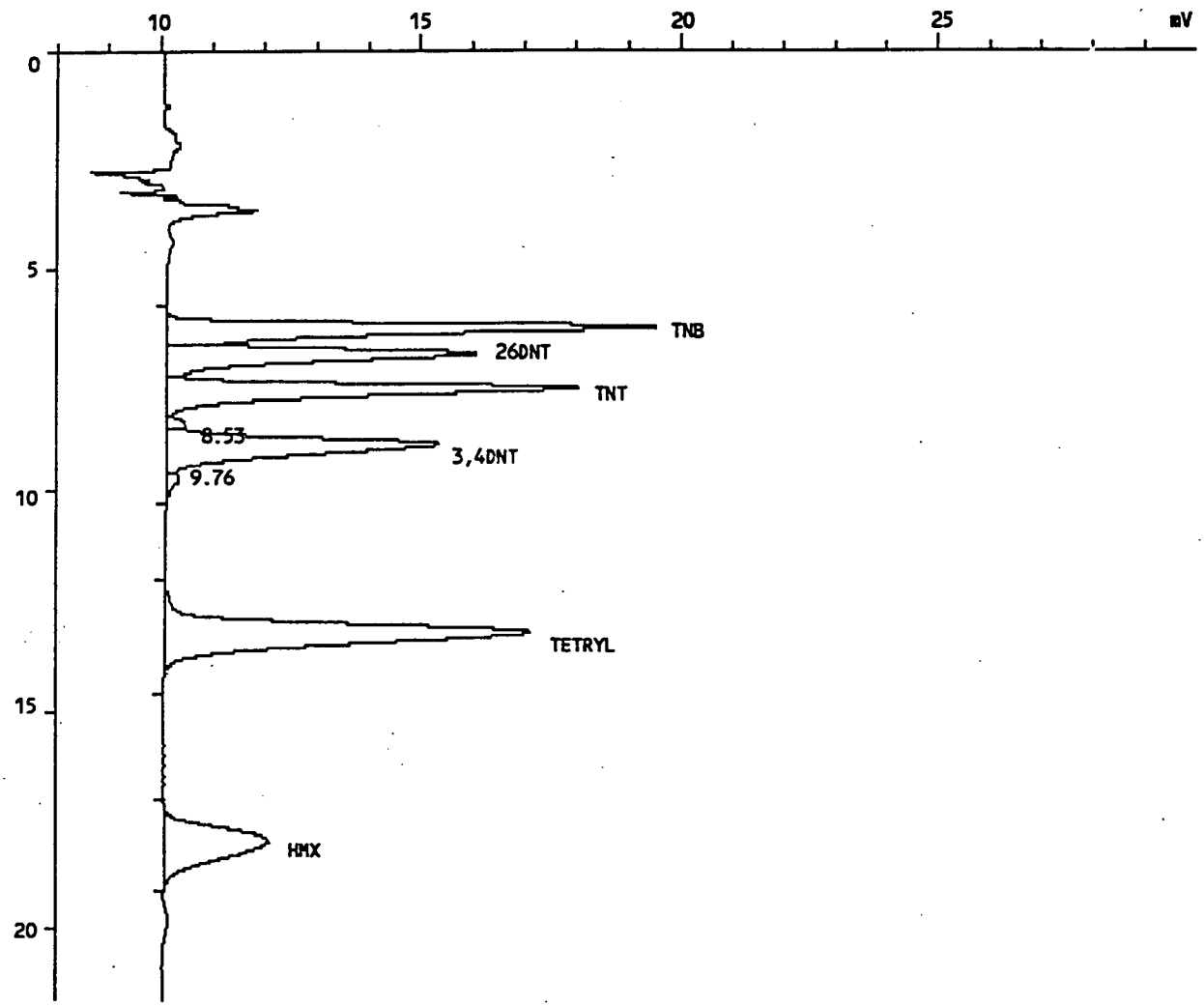
Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
1	5.408	2715	47971	0.239	NB
2	6.192	3729	69049	0.191	DNB
3	7.328	3956	81178	0.239	24DNT
4	8.293	3515	82044	0.445	4ADNT
5	9.067	4632	121665	0.476	2ADNT
6	10.603	948	30900	0.229	RDX
Total		19495	432807	1.819	
Residual		0	0	0.000	

LONG PLOT

Injection F: <MC3> 3 3CN1110,6,1

Sample name.....: EXP L3A STD
Sample ID.....: 1-142:IA3
INST ID: IN10, COL. TYPE: LC-CN, ID: LC-CN-07, INJ:100, UV

Acquired on 10-Nov-97 at 21:15:48
Reported on 21-Nov-97 at 09:20:19



INJECTION REPORT

Injection F: <MC3> 3 3CN1110,6,1

Acquired on 10-Nov-97 at 21:15:48
Modified on 21-Nov-81 at 08:09:12
Reported on 21-Nov-97 at 08:12:38

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN10, COL. TYPE: LC-CN, ID: LC-CN-07, INJ:100, UV
Number of samples.: 30
Calibration file..: 3CN1110B Last modified on 21-Nov-81 at 08:10:12
Method file.....: LCCN Last modified on 21-Nov-81 at 08:10:24
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: EXP L3A STD
Sample ID.....: 1-142:IA3
Sample type.....: Standard
Sample amount.....: 1.0000
Number of injections.....: 1
Bottle Number.....: 1

Retention time update.....: No update
Calibration level identifier.....: 3
Response factor update.....: Initialise

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 1.000
Dilution.....: 1.000
Amount (g or mL).....: 1.000

PEAK INFORMATION

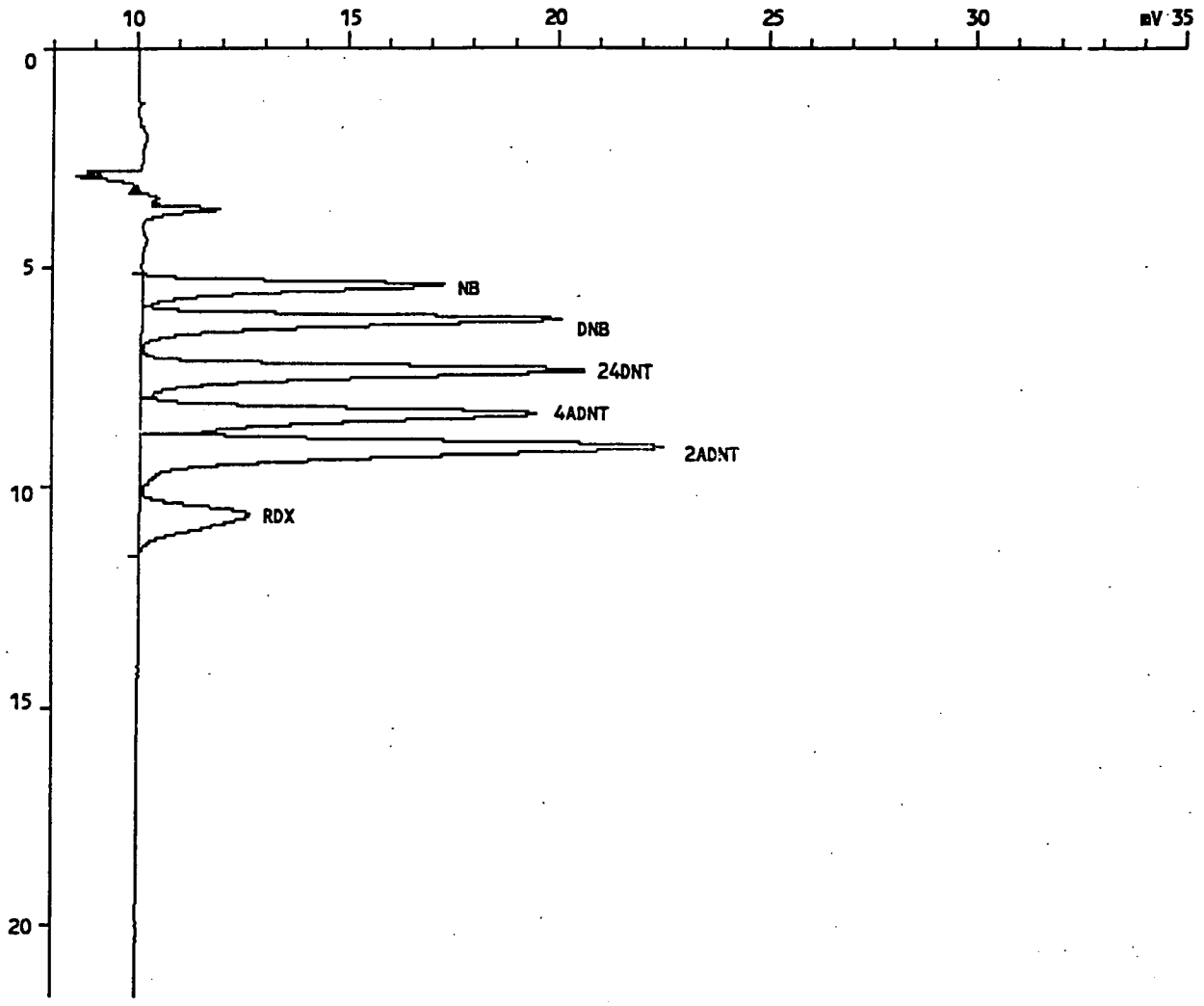
Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
1	6.341	9425	155255	0.577	TNB
2	6.923	5964	116357	0.626	26DNT
3	7.712	7944	147745	0.576	TNT
5	8.955	5259	135244	0.361	3,4DNT
7	13.184	7045	220668	1.159	TETRYL
8	17.957	2008	95317	0.875	HMX
Total		37645	870586	4.175	
Residual		585	9933	0.000	

LONG PLOT

Injection F: <MC3> 3 3CN1110,11,1

Sample name.....: EXP L3B STD
Sample ID.....: 1-147:IB3
INST ID: IN10, COL. TYPE: LC-CN, ID: LC-CN-07, INJ:100, UV

Acquired on 11-Nov-97 at 00:42:34
Reported on 21-Nov-97 at 09:23:40



INJECTION REPORT

Injection F: <MC3> 3 3CN1110,11,1

Acquired on 11-Nov-97 at 00:42:34
Modified on 21-Nov-81 at 08:10:00
Reported on 21-Nov-97 at 08:11:13

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN10, COL. TYPE: LC-CN, ID: LC-CN-07, INJ:100, UV
Number of samples.: 30
Calibration file..: 3CN1110B Last modified on 21-Nov-81 at 08:10:12
Method file.....: LCCN Last modified on 21-Nov-81 at 08:10:24
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: EXP L3B STD
Sample ID.....: 1-147:IB3
Sample type.....: Standard
Sample amount.....: 1.0000
Number of injections.....: 1
Bottle Number.....: 1

Retention time update.....: No update
Calibration level identifier.....: 3
Response factor update.....: Initialise

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 1.000
Dilution.....: 1.000
Amount (g or mL).....: 1.000

PEAK INFORMATION

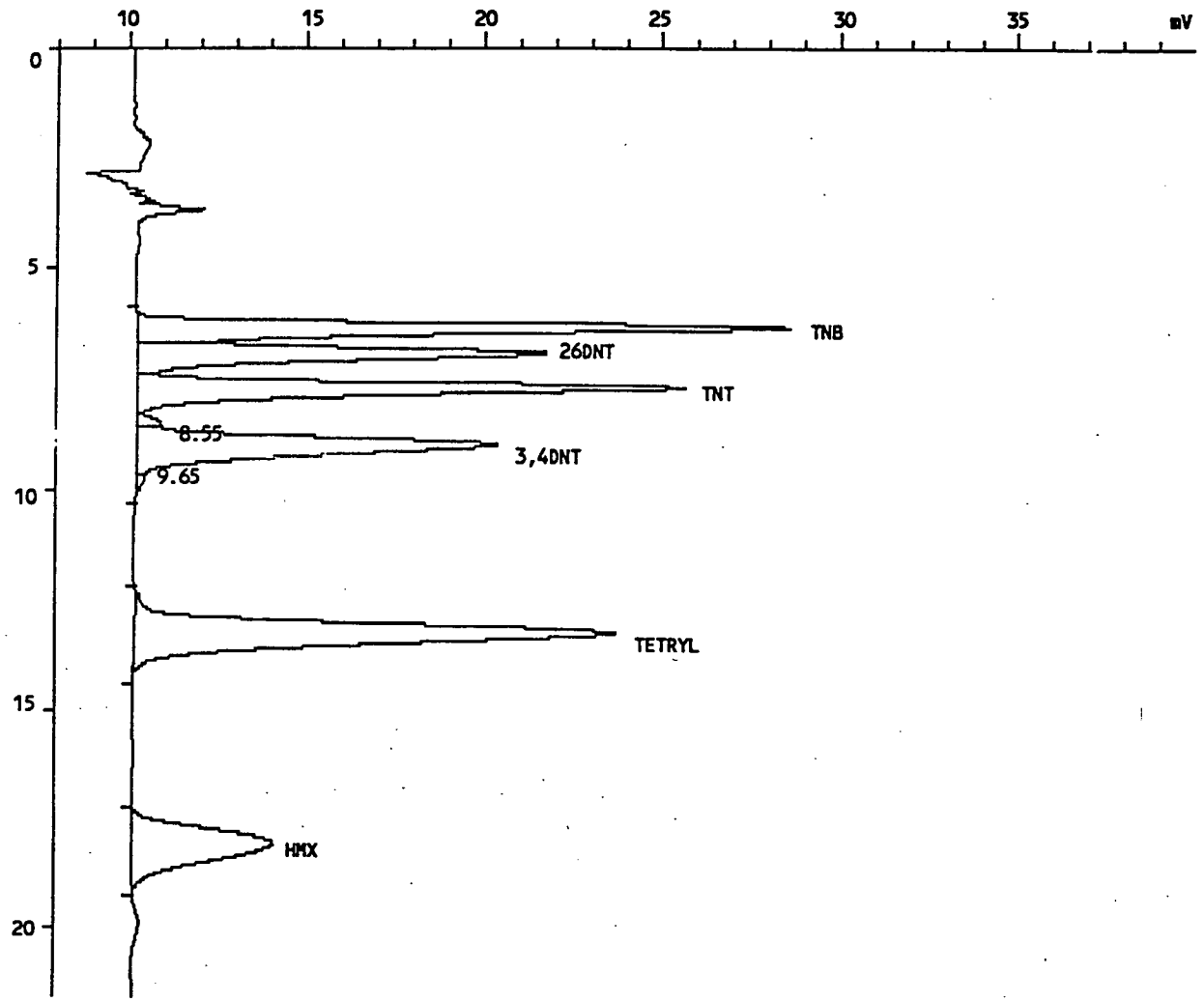
Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
1	5.413	7169	127092	0.633	NB
2	6.197	9964	184705	0.511	DNB
3	7.333	10565	216991	0.640	24DNT
4	8.309	9398	218970	1.186	4ADNT
5	9.077	12438	329591	1.289	2ADNT
6	10.629	2591	86418	0.641	RDX
Total		52125	1163766	4.900	
Residual		0	0	0.000	

LONG PLOT

Injection F: <MC3> 3 3CN1110,7,1

Sample name.....: EXP L4A STD
Sample ID.....: 1-141:IA4
INST ID: IN10, COL. TYPE: LC-CN, ID: LC-CN-07, INJ:100, UV

Acquired on 10-Nov-97 at 21:57:09
Reported on 21-Nov-97 at 09:21:02



INJECTION REPORT

Injection F: <MC3> 3 3CN1110,7,1

Acquired on 10-Nov-97 at 21:57:09
Modified on 21-Nov-81 at 08:09:16
Reported on 21-Nov-97 at 08:12:21

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN10, COL. TYPE: LC-CN, ID: LC-CN-07, INJ:100, UV
Number of samples.: 30
Calibration file..: 3CN1110B Last modified on 21-Nov-81 at 08:10:12
Method file.....: LCCN Last modified on 21-Nov-81 at 08:10:24
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: EXP L4A STD
Sample ID.....: 1-141:IA4
Sample type.....: Standard
Sample amount.....: 1.0000
Number of injections.....: 1
Bottle Number.....: 1

Retention time update.....: No update
Calibration level identifier.....: 4
Response factor update.....: Initialise

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 1.000
Dilution.....: 1.000
Amount (g or mL).....: 1.000

PEAK INFORMATION

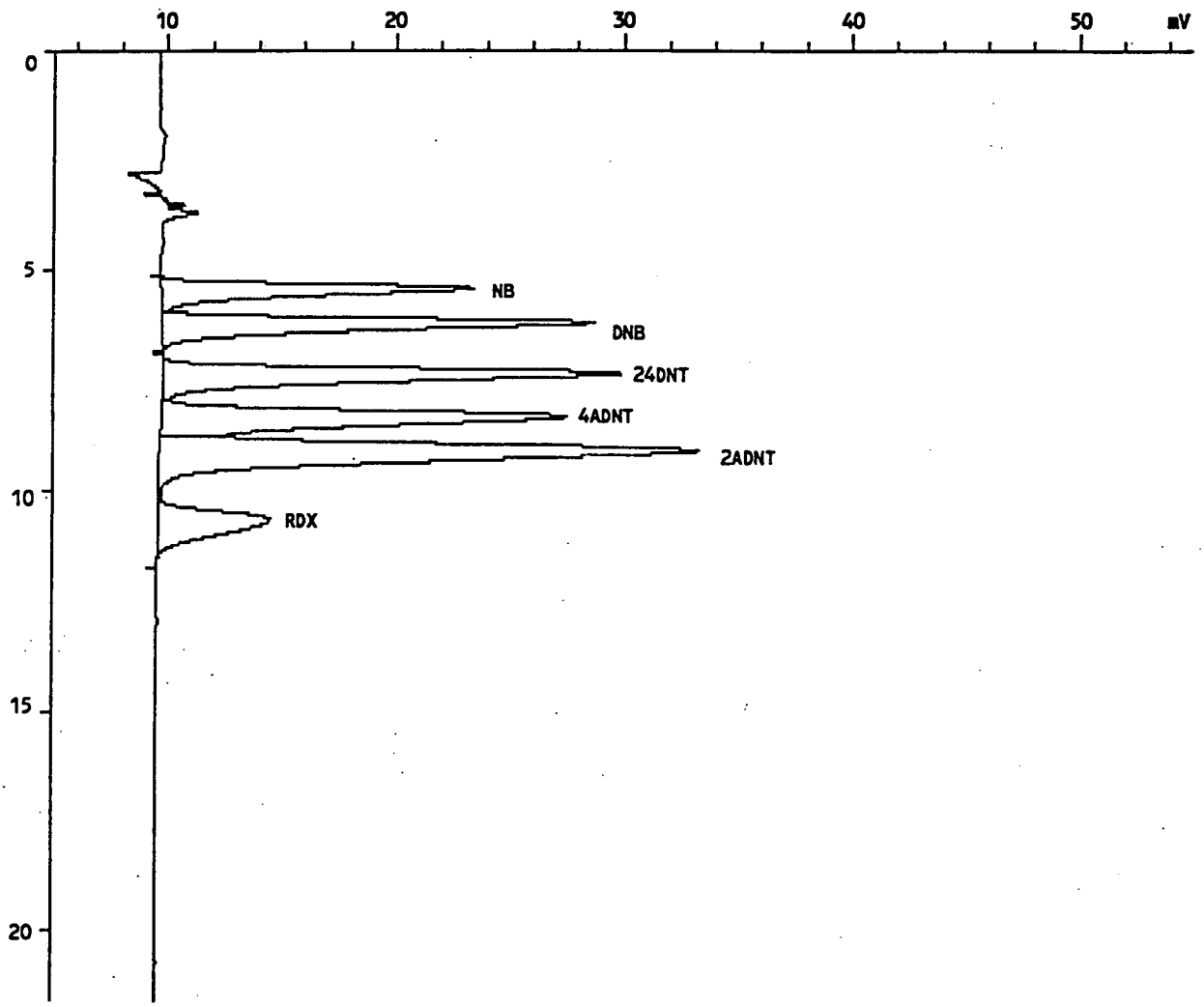
Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
1	6.357	18361	308191	1.146	TNB
2	6.944	11514	230693	1.242	26DNT
3	7.728	15466	293190	1.143	TNT
5	8.981	10202	270561	0.814	3,4DNT
7	13.232	13583	430603	2.262	TETRYL
8	18.037	3933	191029	1.753	HMX
Total		73058	1724267	8.361	
Residual		971	13707	0.000	

LONG PLOT

Injection F: <MC3> 3 3CN1110,12,1

Sample name.....: EXP L4B STD
Sample ID.....: 1-146:IB4
INST ID: IN10, COL. TYPE: LC-CN, ID: LC-CN-07, INJ:100, UV

Acquired on 11-Nov-97 at 01:23:55
Reported on 21-Nov-97 at 09:37:37



INJECTION REPORT

Injection F: <MC3> 3 3CN1110,12,1

Acquired on 11-Nov-97 at 01:23:55
Modified on 21-Nov-81 at 08:10:08
Reported on 21-Nov-97 at 08:10:56

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN10, COL. TYPE: LC-CN, ID: LC-CN-07, INJ:100, UV
Number of samples.: 30
Calibration file..: 3CN1110B Last modified on 21-Nov-81 at 08:10:12
Method file.....: LCCN Last modified on 21-Nov-81 at 08:10:24
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: EXP L4B STD
Sample ID.....: 1-146:IB4
Sample type.....: Standard
Sample amount.....: 1.0000
Number of injections.....: 1
Bottle Number.....: 1

Retention time update.....: No update
Calibration level identifier.....: 4
Response factor update.....: Initialise

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 1.000
Dilution.....: 1.000
Amount (g or mL).....: 1.000

PEAK INFORMATION

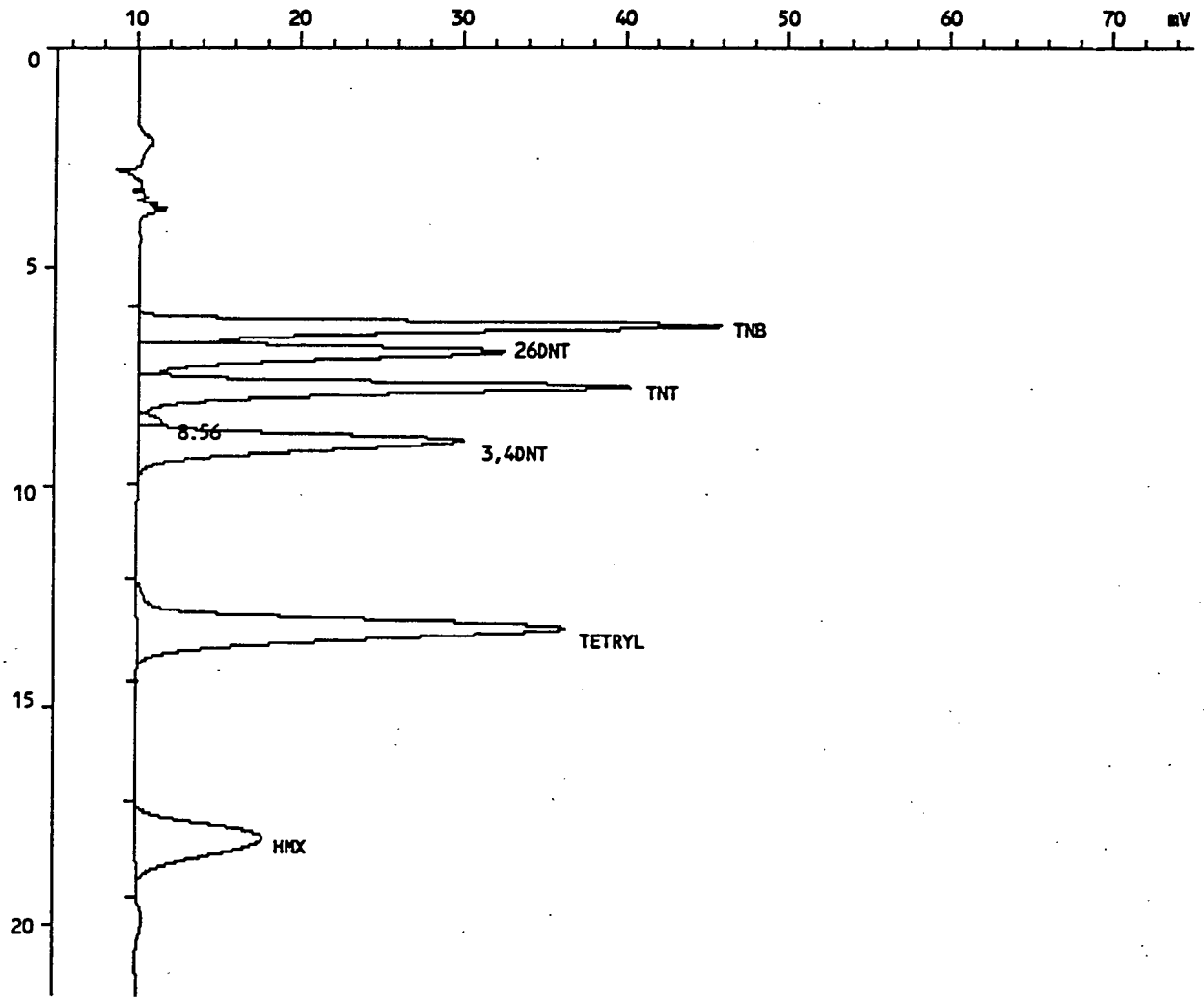
Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
1	5.424	13727	249301	1.241	NB
2	6.208	19010	357696	0.989	DNB
3	7.344	20084	419300	1.237	24DNT
4	8.331	17796	425980	2.308	4ADNT
5	9.093	23669	638249	2.497	2ADNT
6	10.651	4932	168546	1.250	RDX
Total		99217	2259073	9.522	
Residual		0	0	0.000	

LONG PLOT

Injection F: <MC3> 3 3CN1110,8,1

Sample name.....: EXP L5A STD
Sample ID.....: 1-140:IA5
INST ID: IN10, COL. TYPE: LC-CN, ID: LC-CN-07, INJ:100, UV

Acquired on 10-Nov-97 at 22:38:30
Reported on 21-Nov-97 at 09:21:38



INJECTION REPORT

Injection F: <MC3> 3 3CN1110,8,1

Acquired on 10-Nov-97 at 22:38:30
Modified on 21-Nov-81 at 08:09:22
Reported on 21-Nov-97 at 08:12:04

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN10, COL. TYPE: LC-CN, ID: LC-CN-07, INJ:100, UV
Number of samples.: 30
Calibration file..: 3CN1110B Last modified on 21-Nov-81 at 08:10:12
Method file.....: LCCN Last modified on 21-Nov-81 at 08:10:24
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: EXP L5A STD
Sample ID.....: 1-140:IA5
Sample type.....: Standard
Sample amount.....: 1.0000
Number of injections.....: 1
Bottle Number.....: 1

Retention time update.....: No update
Calibration level identifier.....: 5
Response factor update.....: Initialise

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 1.000
Dilution.....: 1.000
Amount (g or mL).....: 1.000

PEAK INFORMATION

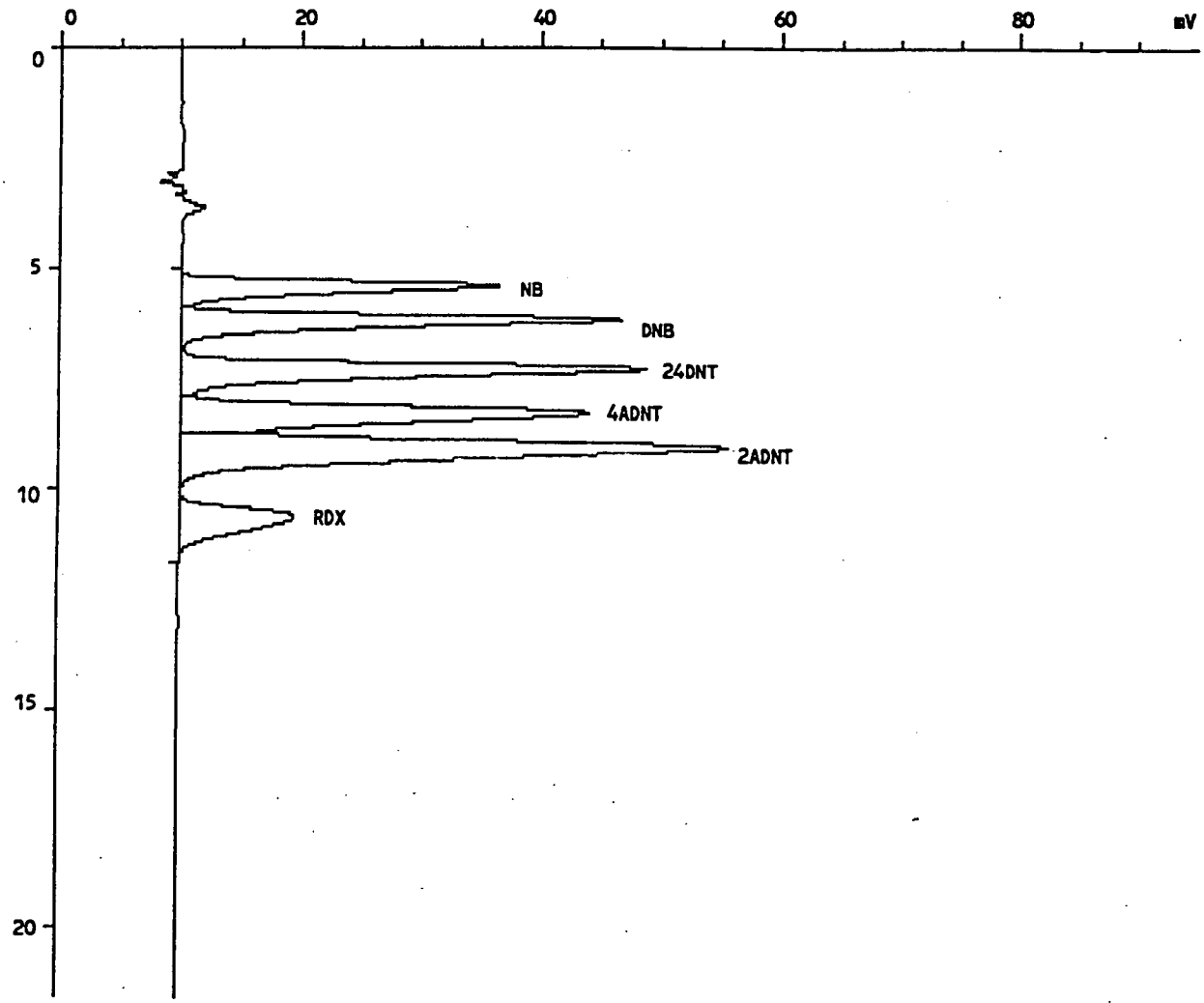
Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
1	6.341	35866	619497	2.303	TNB
2	6.923	22452	465237	2.505	26DNT
3	7.707	30170	591113	2.304	TNT
5	8.955	19936	546051	3.310	3,4DNT
6	13.195	26287	854145	4.488	TETRYL
7	17.989	7799	381516	3.501	HMX
Total		142511	3457559	18.411	
Residual		1439	18195	0.000	

LONG PLOT

Injection F: <MC3> 3 3CN1110,13,1

Sample name.....: EXP L5B STD
Sample ID.....: 1-145:IB5
INST ID: IN10, COL. TYPE: LC-CN, ID: LC-CN-07, INJ:100, UV

Acquired on 11-Nov-97 at 02:05:16
Reported on 21-Nov-97 at 09:35:41



INJECTION REPORT

Injection F: <MC3> 3 3CN1110,13,1

Acquired on 11-Nov-97 at 02:05:16
Modified on 21-Nov-81 at 08:10:12
Reported on 21-Nov-97 at 08:10:40

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN10, COL. TYPE: LC-CN, ID: LC-CN-07, INJ:100, UV
Number of samples.: 30
Calibration file..: 3CN1110B Last modified on 21-Nov-81 at 08:10:12
Method file.....: LCCN Last modified on 21-Nov-81 at 08:10:24
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: EXP L5B STD
Sample ID.....: 1-145:IB5
Sample type.....: Standard
Sample amount.....: 1.0000
Number of injections.....: 1
Bottle Number.....: 1

Retention time update.....: No update
Calibration level identifier.....: 5
Response factor update.....: Initialise

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 1.000
Dilution.....: 1.000
Amount (g or mL).....: 1.000

PEAK INFORMATION

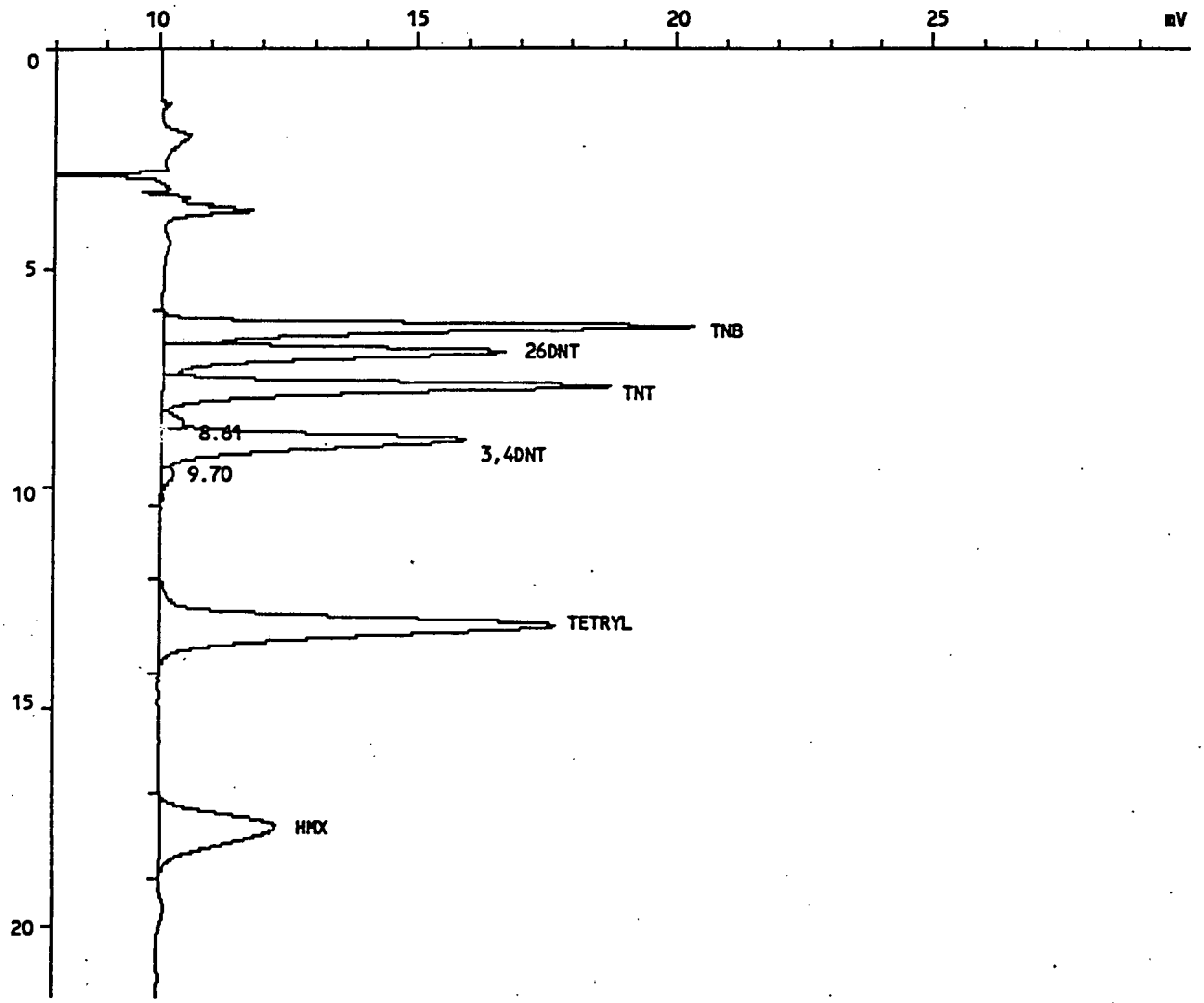
Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
1	5.392	26573	502730	2.503	NB
2	6.176	36834	724567	2.004	DNB
3	7.317	38787	848812	2.504	24DNT
4	8.304	34105	846810	4.588	4ADNT
5	9.072	45537	1276751	4.994	2ADNT
6	10.635	9547	336931	2.498	RDX
Total		191383	4536602	19.092	
Residual		0	0	0.000	

LONG PLOT

Injection F: <MC3> 3 3CN1110B,1,1

Sample name.....: EXP L3A STD
Sample ID.....: 1-142:CA2
INST ID: IN10, COL. TYPE: LC-CN, ID: LC-CN-07, INJ:100, UV

Acquired on 11-Nov-97 at 17:49:57
Reported on 21-Nov-97 at 10:08:59



INJECTION REPORT

Injection F: <MC3> 3 3CN1110B,1,1

Acquired on 11-Nov-97 at 17:49:57
Modified on 21-Nov-81 at 09:43:36
Reported on 21-Nov-97 at 09:43:39

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN10, COL. TYPE: LC-CN, ID: LC-CN-07, INJ:100, UV
Number of samples.: 34
Calibration file..: 3CN1110A Last modified on 21-Nov-81 at 09:39:12
Method file.....: LCCN Last modified on 21-Nov-81 at 09:40:24
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: EXP L3A STD
Sample ID.....: 1-142:CA2
Sample type.....: Standard
Sample amount.....: 1.0000
Number of injections.....: 1
Bottle Number.....: 1

Retention time update.....: No update
Calibration level identifier.....: 3
Response factor update.....: No update

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 1.000
Dilution.....: 1.000
Amount (g or mL).....: 1000.000

PEAK INFORMATION

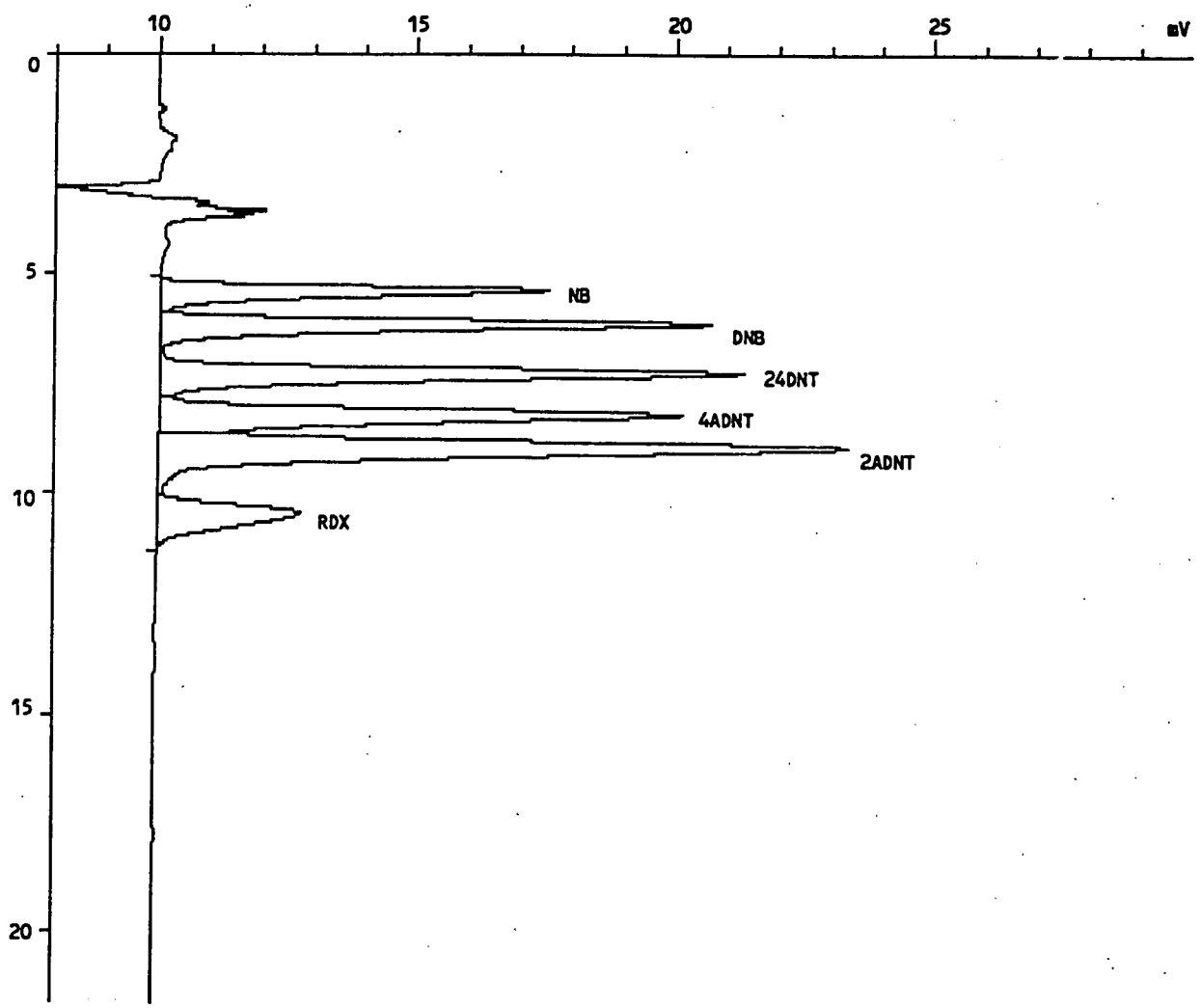
Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
1	6.331	10287	164456	0.623	TNB
2	6.907	6604	122214	0.672	26DNT
3	7.685	8669	155852	0.627	TNT
5	8.907	5908	142351	0.914	3,4DNT
7	13.083	7670	229110	1.224	TETRYL
8	17.717	2239	101524	0.960	HMX
Total		41377	915506	5.020	
Residual		760	13567	0.000	

LONG PLOT

Injection F: <MC3> 3 3CN1110B,2,1

Sample name.....: EXP L3B STD
Sample ID.....: 1-147:CB2
INST ID: IN10, COL. TYPE: LC-CN, ID: LC-CN-07, INJ:100, UV

Acquired on 11-Nov-97 at 18:31:18
Reported on 21-Nov-97 at 10:09:20



INJECTION REPORT

Injection F: <MC3> 3 3CN1110B,2,1

Acquired on 11-Nov-97 at 18:31:18
Modified on 21-Nov-81 at 09:42:54
Reported on 21-Nov-97 at 09:42:56

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN10, COL. TYPE: LC-CN, ID: LC-CN-07, INJ:100, UV
Number of samples.: 34
Calibration file..: 3CN1110B Last modified on 21-Nov-81 at 09:38:30
Method file.....: LCCN Last modified on 21-Nov-81 at 09:40:24
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: EXP L3B STD
Sample ID.....: 1-147:CB2
Sample type.....: Standard
Sample amount.....: 1.0000
Number of injections.....: 1
Bottle Number.....: 1

Retention time update.....: No update
Calibration level identifier.....: 3
Response factor update.....: No update

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 1.000
Dilution.....: 1.000
Amount (g or mL).....: 1000.000

PEAK INFORMATION

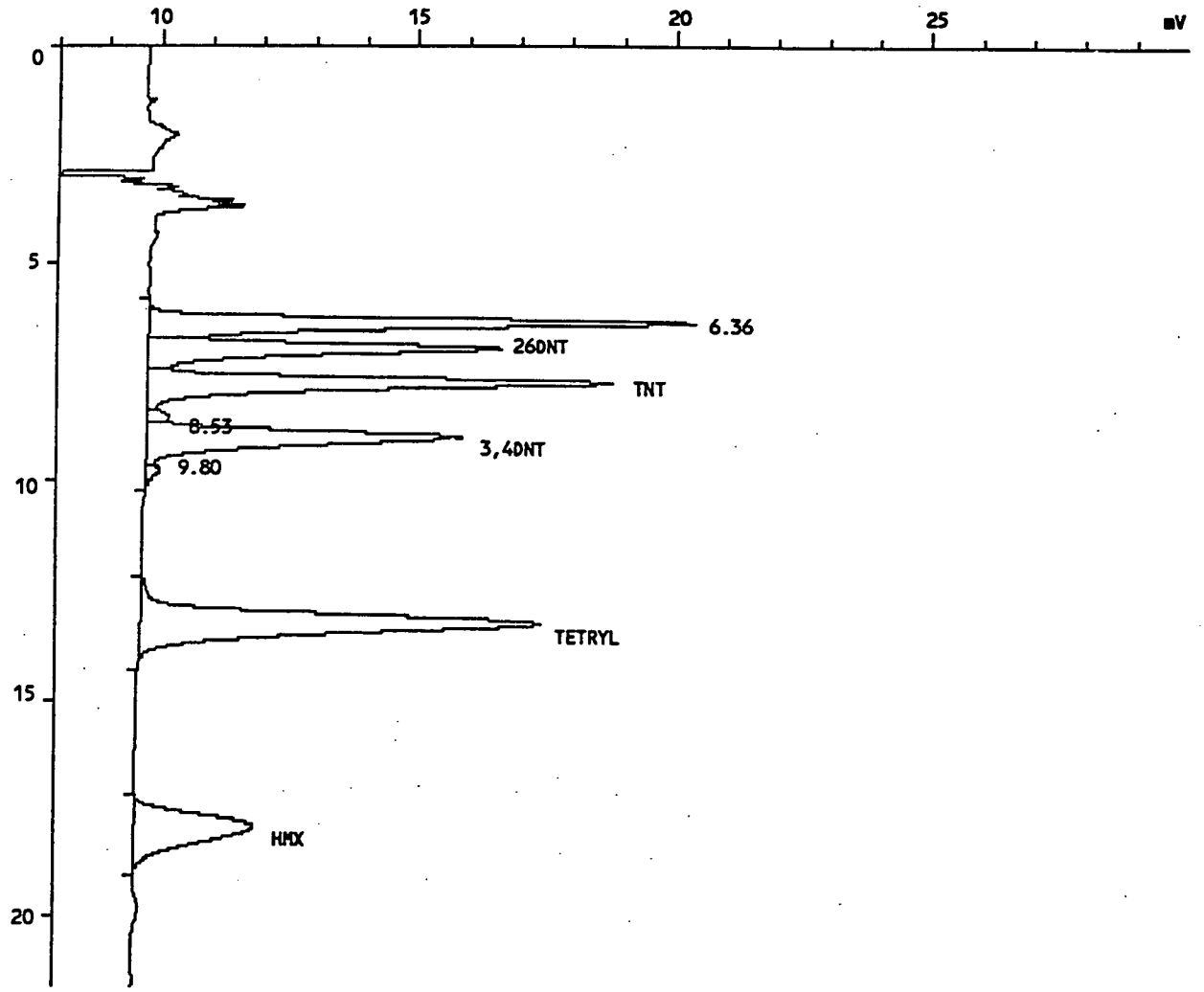
Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
1	5.381	7565	128794	0.653	NB
2	6.149	10648	190129	0.535	DNB
3	7.269	11301	223032	0.672	24DNT
4	8.213	10136	226632	1.245	4ADNT
5	8.960	13389	337989	1.343	2ADNT
6	10.459	2769	87839	0.687	RDX
Total		55809	1194416	5.136	
Residual		0	0	0.000	

LONG PLOT

Injection F: <MC3> 3 3CN1110B,11,1

Sample name.....: EXP L3A STD
Sample ID.....: 1-142:CA3
INST ID: IN10, COL. TYPE: LC-CN, ID: LC-CN-07, INJ:100, UV

Acquired on 12-Nov-97 at 00:43:43
Reported on 21-Nov-97 at 09:44:40



INJECTION REPORT

Injection F: <MC3> 3 3CN1110B,11,1

Acquired on 12-Nov-97 at 00:43:43
Modified on 21-Nov-81 at 09:44:12
Reported on 21-Nov-97 at 09:44:15

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN10, COL. TYPE: LC-CN, ID: LC-CN-07, INJ:100, UV
Number of samples.: 34
Calibration file..: 3CN1110A Last modified on 21-Nov-81 at 09:39:12
Method file.....: LCCN Last modified on 21-Nov-81 at 09:40:24
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: EXP L3A STD
Sample ID.....: 1-142:CA3
Sample type.....: Standard
Sample amount.....: 1.0000
Number of injections.....: 1
Bottle Number.....: 1

Retention time update.....: No update
Calibration level identifier.....: 3
Response factor update.....: No update

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 1.000
Dilution.....: 1.000
Amount (g or mL).....: 1000.000

PEAK INFORMATION

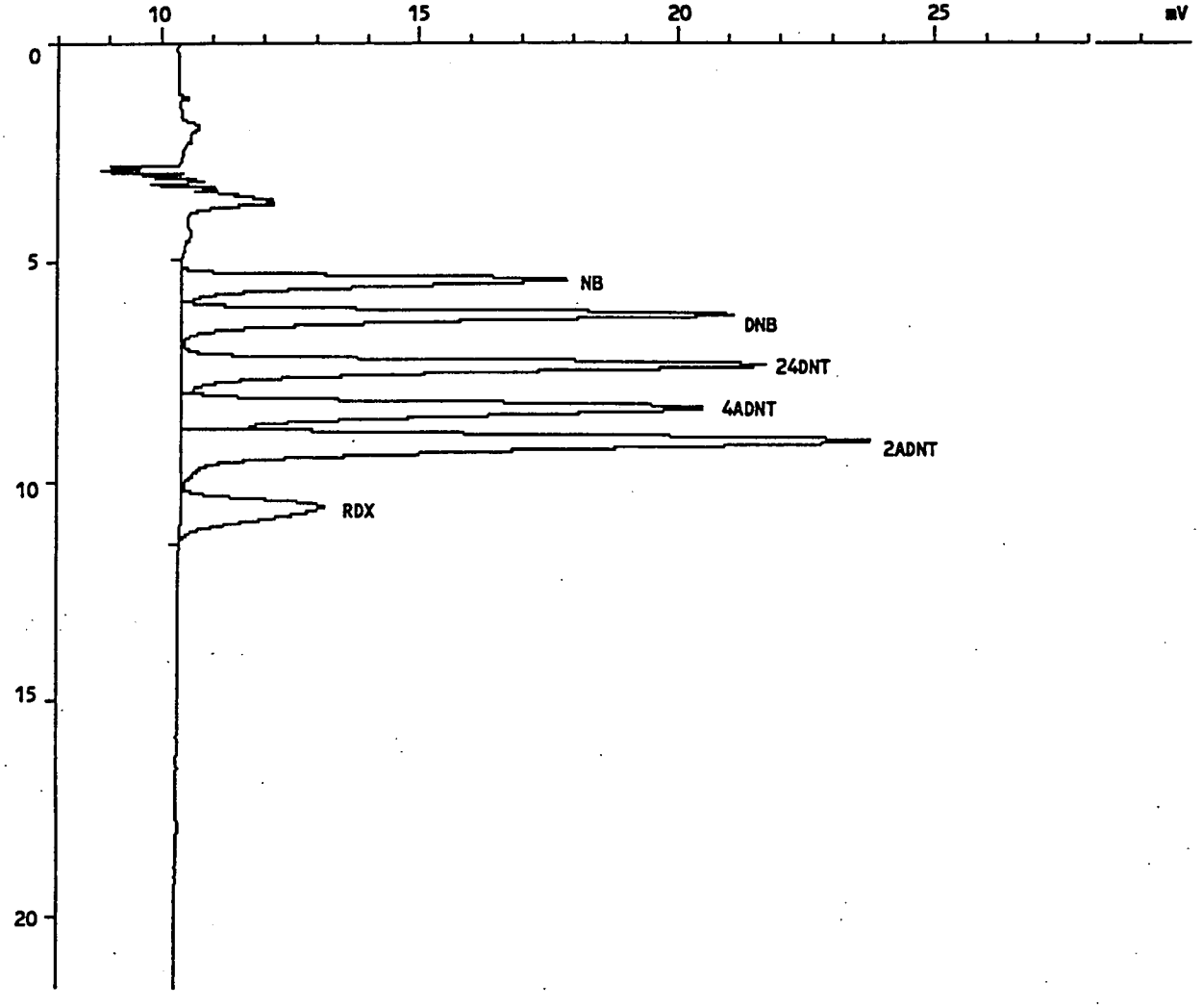
Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
2	6.949	6888	127596	0.702	26DNT
3	7.733	9017	163573	0.658	TNT
5	8.987	6132	146583	0.941	3,4DNT
7	13.205	7792	228788	1.222	TETRYL
8	17.920	2326	102788	0.972	HMX
Total		32155	769328	4.495	
Residual		11343	180995	0.000	

LONG PLOT

Injection F: <MC3> 3 3CN1110B,12,1

Sample name.....: EXP L3B STD
Sample ID.....: 1-147:CB3
INST ID: IN10, COL. TYPE: LC-CN, ID: LC-CN-07, INJ:100, UV

Acquired on 12-Nov-97 at 01:25:07
Reported on 21-Nov-97 at 09:45:28



INJECTION REPORT

Injection F: <MC3> 3 3CN1110B,12,1

Acquired on 12-Nov-97 at 01:25:07
Modified on 21-Nov-81 at 09:45:08
Reported on 21-Nov-97 at 09:45:10

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN10, COL. TYPE: LC-CN, ID: LC-CN-07, INJ:100, UV
Number of samples.: 34
Calibration file..: 3CN1110B Last modified on 21-Nov-81 at 09:38:30
Method file.....: LCCN Last modified on 21-Nov-81 at 09:40:24
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: EXP L3B STD
Sample ID.....: 1-147:CB3
Sample type.....: Standard
Sample amount.....: 1.0000
Number of injections.....: 1
Bottle Number.....: 1

Retention time update.....: No update
Calibration level identifier.....: 3
Response factor update.....: No update

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 1.000
Dilution.....: 1.000
Amount (g or mL).....: 1000.000

PEAK INFORMATION

Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
1	5.408	7474	127817	0.648	NB
2	6.187	10678	190776	0.537	DNB
3	7.317	11327	223948	0.675	24DNT
4	8.288	10125	226774	1.245	4ADNT
5	9.040	13386	339108	1.348	2ADNT
6	10.581	2776	88162	0.690	RDX
Total		55766	1196585	5.143	
Residual		0	0	0.000	

1D.

EXPLOSIVE ANALYSIS DATA SHEET

EPA SAMPLE NO:

INST. BLK.

Lab Name: SWL-TULSA Contract:

Lab Code: SWOK Case No:MKF-OH SDG No:

Matrix: (soil/water) SOIL Lab Sample ID: INST.BLK.

Sample Amt: 2gm % Moisture Date Received: / /

Extraction Volume: 10ml Date Extracted: / /

Extraction Method: SONC Date Analyzed: 10/16/97

GPC Cleanup: (Y/N) N Dilution Factor: 2.00

CAS NO.	COMPOUND	CONCENTRATION UNITS: (ug/L or ug/kg) ug/kg	Q
2691-41-0	HMX	2200	U
121-82-4	RDX	1000	U
99-35-4	TNB	250	U
99-65-0	DNB	250	U
479-45-8	TETRYL	650	U
98-95-3	NB	260	U
118-96-7	TNT	250	U
1946-51-0	4ADNT	250	U
35572-78-2	2ADNT	250	U
606-20-2	26DNT	260	U
121-14-2	24DNT	250	U
88-72-2	2NT	250	U
99-99-0	4NT	250	U
99-08-1	3NT	250	U

SURROGATE AMOUNT SPIKED (ug/kg dry wt.) 20000
3,4-DNT

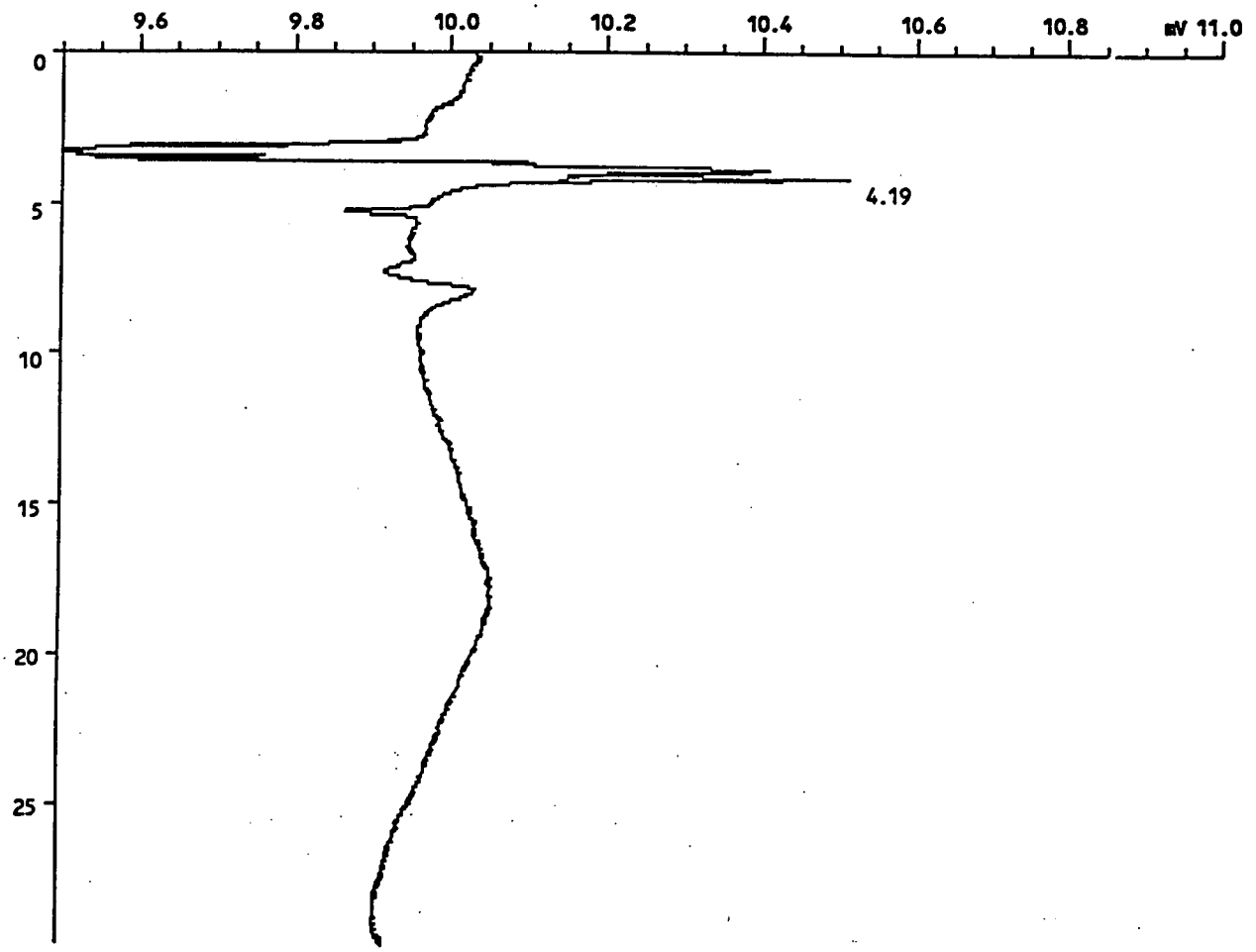
FORM I

LONG PLOT

Injection F: <MC3> 5 5EX1015,1,1

Sample name.....: INST. BLK.
Sample ID.....: INST. BLK.
INST ID: IN13, COL. TYPE: ODS-II, ID: CARB-05, INJ:200,COL#1

Acquired on 15-Oct-97 at 23:47:37
Reported on 20-Nov-97 at 16:49:48



INJECTION REPORT

Injection F: <MC3> 5 5EX1015,1,1

Acquired on 15-Oct-97 at 23:47:37
Modified on 17-Nov-81 at 21:07:32
Reported on 20-Nov-97 at 16:49:11

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN13, COL. TYPE: ODS-II, ID: CARB-05, INJ:200,COL#1
Number of samples.: 31
Calibration file..: 5EX1015 Last modified on 17-Nov-81 at 21:07:32
Method file.....: EXPLOS Last modified on 20-Nov-81 at 16:48:42
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: INST. BLK.
Sample ID.....: INST. BLK.
Sample type.....: Sample
Sample amount.....: 2.0000
Number of injections.....: 1
Bottle Number.....: 1

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 10.000
Dilution.....: 2.000
Amount (g or mL).....: 2.000

PEAK INFORMATION

Peak RT mins	Hght uV	Area uVs	ug/Kg	Peak name
Total	0	0	0.000	
Residual	365	2630	-20.546	

1D
EXPLOSIVE ANALYSIS DATA SHEET

EPA SAMPLE NO:

BL1014SC

ıb Name: SWL-TULSA Contract:

Lab Code: SWOK Case No:MKF-OH SDG No:

Matrix: (soil/water) SOIL Lab Sample ID: BL1014SC

Sample Amt: 2gm % Moisture Date Received: / /

Extraction Volume: 10ml Date Extracted: 10/14/97

Extraction Method: SONC Date Analyzed: 10/17/97

GPC Cleanup: (Y/N) N Dilution Factor: 2.00

CAS NO.	COMPOUND	CONCENTRATION UNITS: (ug/L or ug/kg) ug/kg	Q
2691-41-0	HMX	2200	U
121-82-4	RDX	1000	U
99-35-4	TNB	250	U
99-65-0	DNB	250	U
479-45-8	TETRYL	650	U
98-95-3	NB	260	U
118-96-7	TNT	250	U
1946-51-0	4ADNT	250	U
35572-78-2	2ADNT	250	U
606-20-2	26DNT	260	U
121-14-2	24DNT	250	U
88-72-2	2NT	250	U
99-99-0	4NT	250	U
99-08-1	3NT	250	U

SURROGATE AMOUNT SPIKED (ug/kg dry wt.) 20000
3,4-DNT

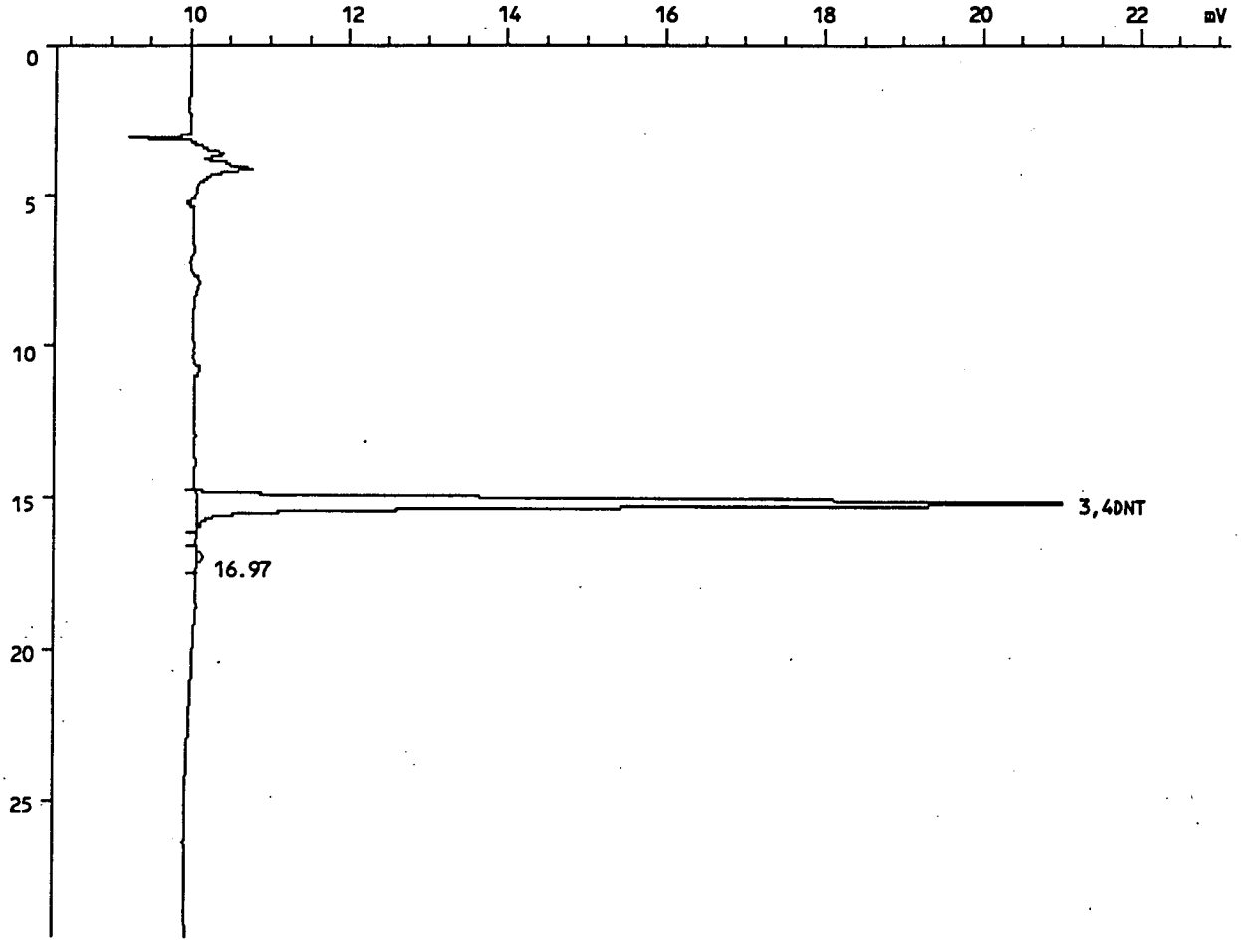
FORM I

LONG PLOT

Injection F: <MC3> 5 5EX1015C,3,1

Sample name.....: BL1014SC
Sample ID.....: BL1014SC
INST ID: IN13, COL. TYPE: ODS-II, ID: CARB-05, INJ:200,COL#1

Acquired on 17-Oct-97 at 22:25:00
Reported on 05-Nov-97 at 08:50:07



INJECTION REPORT

Injection F: <MC3> 5 5EX1015C,3,1

Acquired on 17-Oct-97 at 22:25:00
Modified on 05-Nov-81 at 08:49:46
Reported on 05-Nov-97 at 08:49:50

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN13, COL. TYPE: ODS-II, ID: CARB-05, INJ:200,COL#1
Number of samples.: 27
Calibration file..: 5EX1015 Last modified on 04-Nov-81 at 20:53:04
Method file.....: EXPLOS Last modified on 05-Nov-81 at 08:43:52
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: BL1014SC
Sample ID.....: BL1014SC
Sample type.....: Sample
Sample amount.....: 2.0000
Number of injections.....: 1
Bottle Number.....: 1

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 10.000
Dilution.....: 2.000
Amount (g or mL).....: 2.000

PEAK INFORMATION

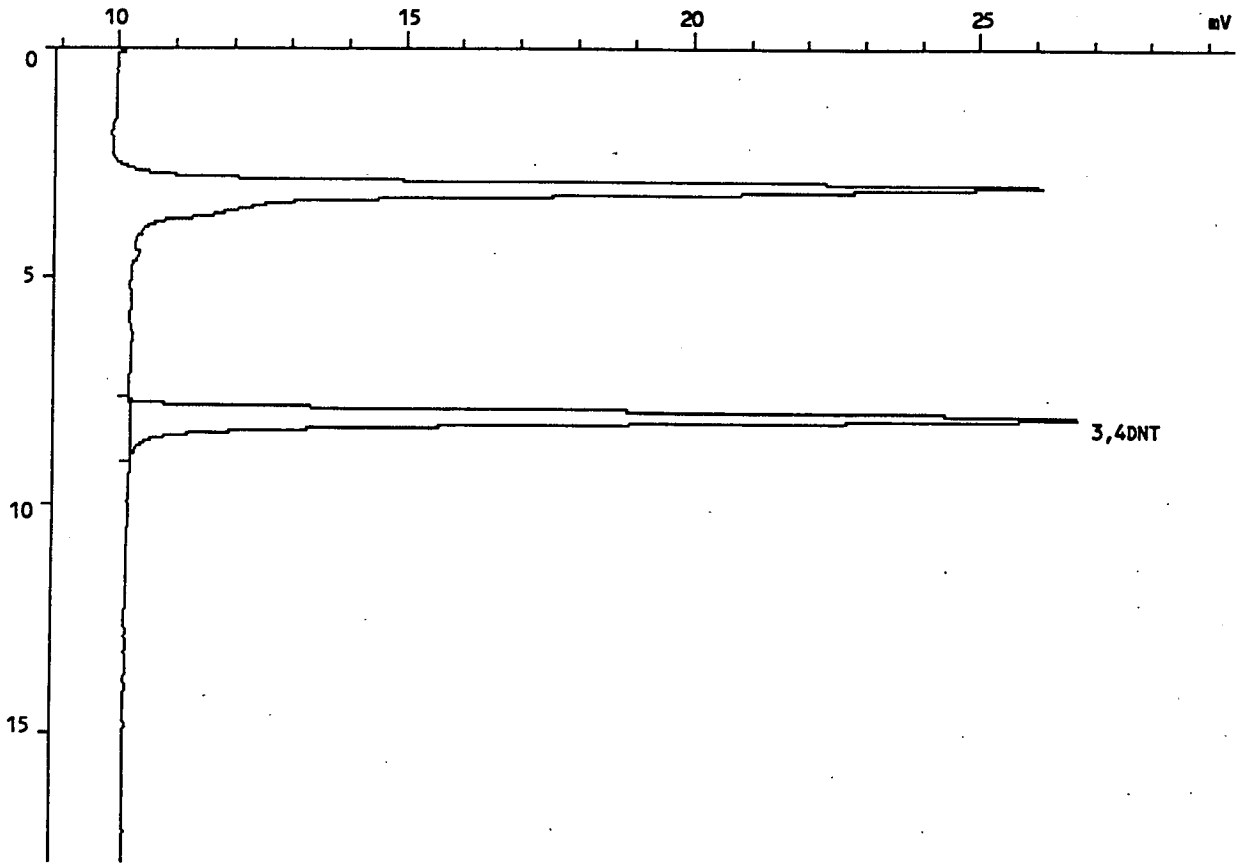
Peak	RT mins	Hght uV	Area uVs	ug/Kg	Peak name
1	15.179	10946	224462	19417.154	3,4DNT 97
Total		10946	224462	19417.154	
Residual		100	2112	182.706	

LONG PLOT

Injection F: <MC3> 2 2CN1024,14,1

Sample name.....: BL1014SC
Sample ID.....: BL1014SC
INST ID: IN12, COL. TYPE: LC-CN, ID: LC-CN-05, INJ:100, UV

Acquired on 25-Oct-97 at 02:33:41
Reported on 05-Nov-97 at 12:04:30



INJECTION REPORT

Injection F: <MC3> 2 2CN1024,14,1

Acquired on 25-Oct-97 at 02:33:41
Modified on 05-Nov-81 at 11:58:54
Reported on 05-Nov-97 at 11:58:54

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN12, COL. TYPE: LC-CN, ID: LC-CN-05, INJ:100, UV
Number of samples.: 67
Calibration file..: 2CN1024A Last modified on 05-Nov-81 at 11:54:16
Method file.....: LCCN Last modified on 05-Nov-81 at 11:58:32
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: BL1014SC
Sample ID.....: BL1014SC
Sample type.....: Sample
Sample amount.....: 2.0000
Number of injections.....: 1
Bottle Number.....: 1

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 10.000
Dilution.....: 2.000
Amount (g or mL).....: 2.000

PEAK INFORMATION

Peak	RT mins	Hght uV	Area uVs	ug/Kg	Peak name
1	8.059	16542	325871	18473.408	3,4DNT
Total		16542	325871	18473.408	
Residual		0	0	0.000	

1D

EXPLOSIVE ANALYSIS DATA SHEET

EPA SAMPLE NO:

CRA/BPSAD1 MS 2
~~0000~~X 20000X
11/29/97

Lab Name: SWL-TULSA Contract:

Lab Code: SWOK Case No:MKF-OH SDG No: 31436

Matrix: (soil/water) SOIL Lab Sample ID: 31436.02MS

Sample Amt: 2gm % Moisture 11.31 Date Received: 10/14/97

Extraction Volume: 10ml Date Extracted: 10/14/97

Extraction Method: SONC Date Analyzed: 10/18/97

GPC Cleanup: (Y/N) N Dilution Factor: 20000.00

CAS NO.	COMPOUND	CONCENTRATION UNITS: (ug/L or ug/kg) ug/kg	Q
2691-41-0	HMX	15100000	J
121-82-4	RDX	40700000	
99-35-4	TNB	2500000	U
99-65-0	DNB	2500000	U
479-45-8	TETRYL	6500000	U
98-95-3	NB	2600000	U
118-96-7	TNT	65400000	
1946-51-0	4ADNT	2500000	U
35572-78-2	2ADNT	2500000	U
606-20-2	26DNT	2600000	U
121-14-2	24DNT	2500000	U
88-72-2	2NT	2500000	U
99-99-0	4NT	2500000	U
99-08-1	3NT	2500000	U

SURROGATE AMOUNT SPIKED (ug/kg dry wt.) 20000
3,4-DNT

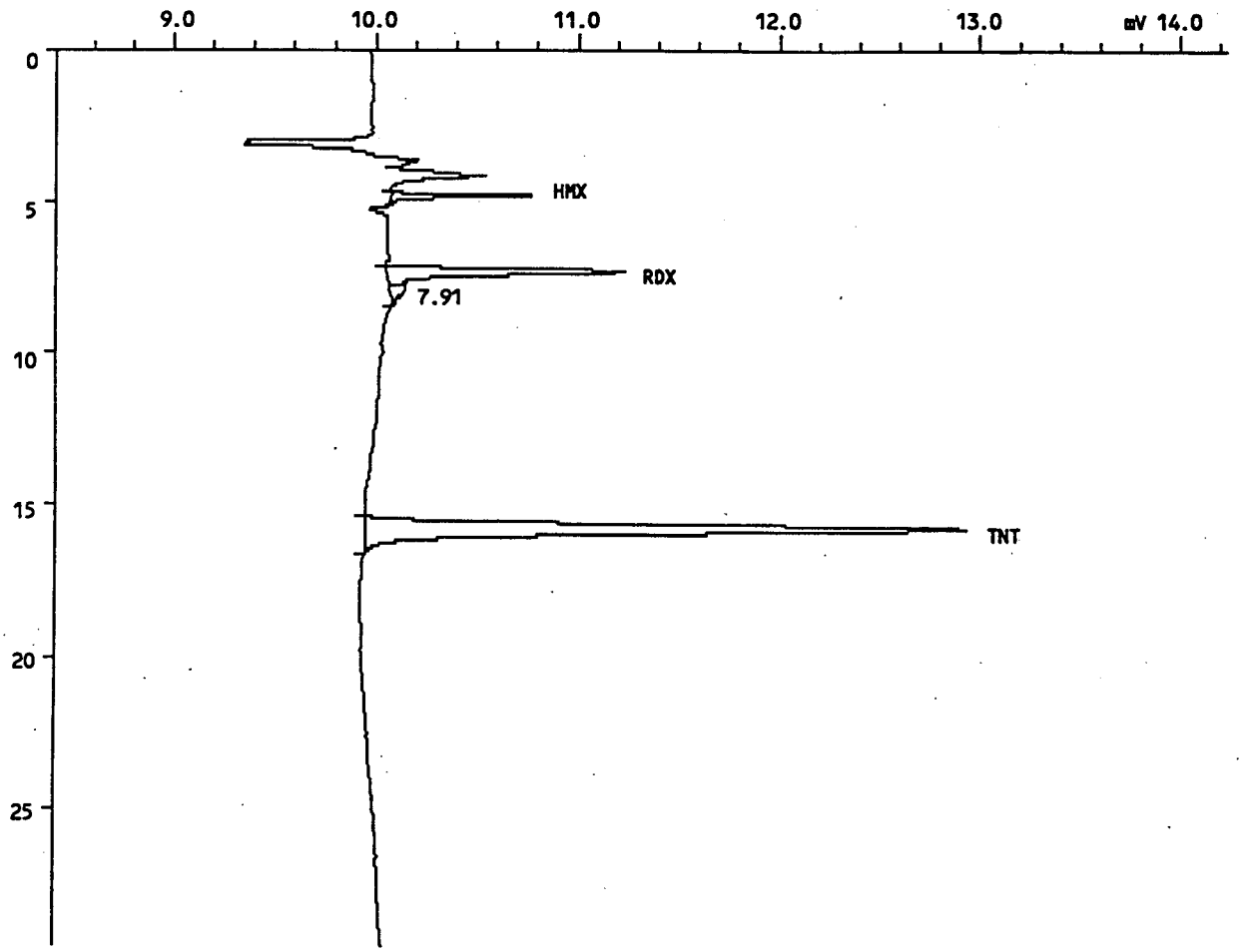
FORM I

LONG PLOT

Injection F: <MC3> 5 5EX1015D,6,1

Sample name.....: CRA/BPSAD1 MS 20000X
Sample ID.....: 31436.02MS
INST ID: IN13, COL. TYPE: ODS-II, ID: CARB-05, INJ:200,COL#1

Acquired on 18-Oct-97 at 20:15:18
Reported on 05-Nov-97 at 12:29:38



INJECTION REPORT

Injection F: <MC3> 5 5EX1015D,6,1

Acquired on 18-Oct-97 at 20:15:18
Modified on 05-Nov-81 at 12:27:58
Reported on 05-Nov-97 at 12:28:39

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN13, COL. TYPE: ODS-II, ID: CARB-05, INJ:200,COL#1
Number of samples.: 26
Calibration file..: 5EX1015 Last modified on 05-Nov-81 at 11:22:30
Method file.....: EXPLOS Last modified on 05-Nov-81 at 08:54:26
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: CRA/BPSAD1 MS 20000X
Sample ID.....: 31436.02MS
Sample type.....: Sample
Sample amount.....: 2.0000
Number of injections.....: 1
Bottle Number.....: 1

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 20.000
Dilution.....: 20000.000
Amount (g or mL).....: 2.000

PEAK INFORMATION

Peak	RT mins	Hght uV	Area uVs	ug/Kg	Peak name
1	4.848	698	5257	15105286.000	HMX
2	7.365	1183	17470	40722108.000	RDX
4	15.803	2987	64134	65375876.000	TNT
Total		4868	86860	121203264.000	
Residual		80	2159	5031516.500	

1D
EXPLOSIVE ANALYSIS DATA SHEET

EPA SAMPLE NO:

CRA/BPSAD1 MSD
20000X
11/27/97

Lab Name: SWL-TULSA Contract:

Lab Code: SWOK Case No:MKF-OH SDG No: 31436

Matrix: (soil/water) SOIL Lab Sample ID: 31436.03MSD

Sample Amt: 2gm % Moisture 11.31 Date Received: 10/14/97

Extraction Volume: 10ml Date Extracted: 10/14/97

Extraction Method: SONC Date Analyzed: 10/18/97

GPC Cleanup: (Y/N) N Dilution Factor: 20000.00

CAS NO.	COMPOUND	CONCENTRATION UNITS: (ug/L or ug/kg) ug/kg	Q
2691-41-0	HMX	18800000	J
121-82-4	RDX	50600000	
99-35-4	TNB	2500000	U
99-65-0	DNB	2500000	U
479-45-8	TETRYL	6500000	U
98-95-3	NB	2600000	U
118-96-7	TNT	77000000	
1946-51-0	4ADNT	2500000	U
35572-78-2	2ADNT	2500000	U
606-20-2	26DNT	2600000	U
121-14-2	24DNT	2500000	U
88-72-2	2NT	2500000	U
99-99-0	4NT	2500000	U
99-08-1	3NT	2500000	U

SURROGATE AMOUNT SPIKED (ug/kg dry wt.) 20000
3,4-DNT

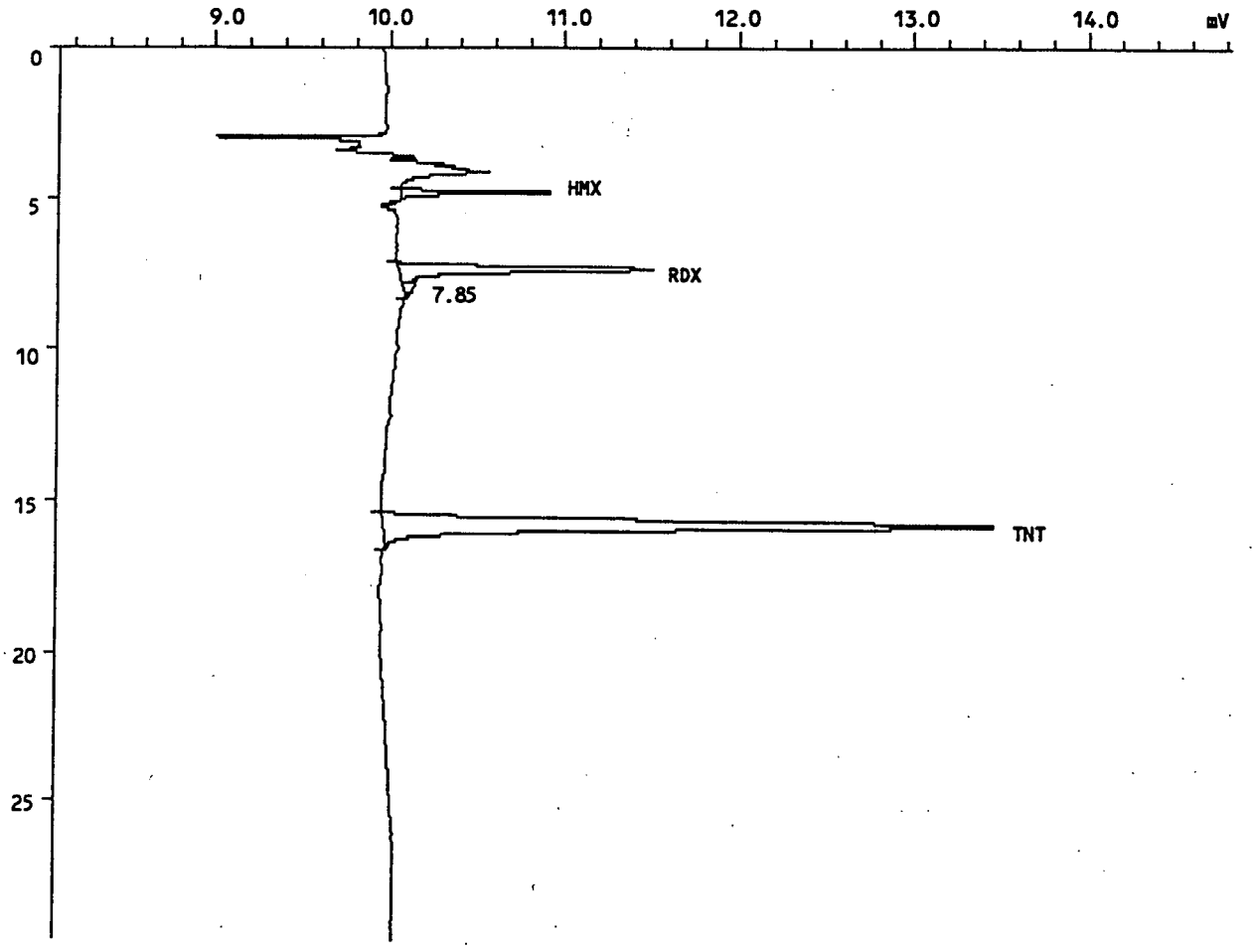
FORM I

LONG PLOT

Injection F: <MC3> 5 5EX1015D,7,1

Sample name.....: CRA/BPSAD1 MSD 20000X
Sample ID.....: 31436.03MSD
INST ID: IN13, COL. TYPE: ODS-II, ID: CARB-05, INJ:200,COL#1

Acquired on 18-Oct-97 at 20:59:59
Reported on 05-Nov-97 at 12:29:57



INJECTION REPORT

Injection F: <MC3> 5 5EX1015D,7,1

Acquired on 18-Oct-97 at 20:59:59
Modified on 05-Nov-81 at 12:28:12
Reported on 05-Nov-97 at 12:28:24

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN13, COL. TYPE: ODS-II, ID: CARB-05, INJ:200,COL#1
Number of samples.: 26
Calibration file..: 5EX1015 Last modified on 05-Nov-81 at 11:22:30
Method file.....: EXPLOS Last modified on 05-Nov-81 at 08:54:26
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: CRA/BPSAD1 MSD 20000X
Sample ID.....: 31436.03MSD
Sample type.....: Sample
Sample amount.....: 2.0000
Number of injections.....: 1
Bottle Number.....: 1

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 20.000
Dilution.....: 20000.000
Amount (g or mL).....: 2.000

PEAK INFORMATION

Peak	RT mins	Hght uV	Area uVs	ug/Kg	Peak name
1	4.843	870	6530	18765732.000	HMX
2	7.360	1472	21696	50572896.000	RDX
4	15.792	3511	75564	77027248.000	TNT
Total		5853	103790	146365872.000	
Residual		66	1406	3278055.000	

1D

EXPLOSIVE ANALYSIS DATA SHEET

EPA SAMPLE NO:

LC1014SC

Lab Name: SWL-TULSA Contract:

Lab Code: SWOK Case No:MKF-OH SDG No:

Matrix: (soil/water) SOIL Lab Sample ID: LC1014SC

Sample Amt: 2gm % Moisture Date Received: / /

Extraction Volume: 10ml Date Extracted: 10/14/97

Extraction Method: SONC Date Analyzed: 10/18/97

GPC Cleanup: (Y/N) N Dilution Factor: 2.00

CAS NO.	COMPOUND	CONCENTRATION UNITS: (ug/L or ug/kg) ug/kg	Q
2691-41-0	HMX	32500	
121-82-4	RDX	13600	
99-35-4	TNB	11200	
99-65-0	DNB	8420	
479-45-8	TETRYL	1410	
98-95-3	NB	7410	
118-96-7	TNT	8560	
1946-51-0	4ADNT	10400	
35572-78-2	2ADNT	7570	
606-20-2	26DNT	7640	
121-14-2	24DNT	5000	
88-72-2	2NT	15900	
99-99-0	4NT	10400	
99-08-1	3NT	10600	

SURROGATE AMOUNT SPIKED (ug/kg dry wt.) 20000
3,4-DNT

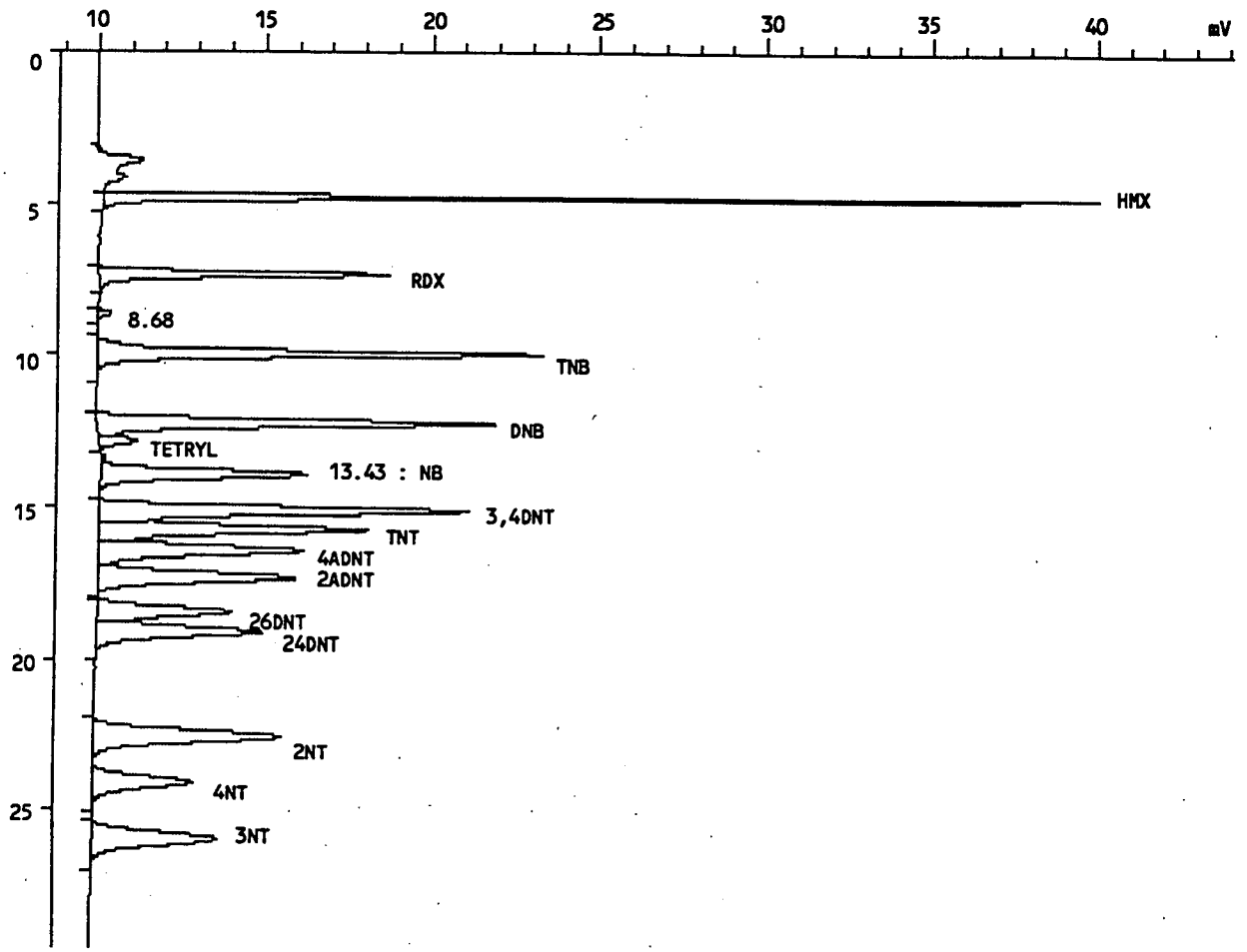
FORM I

LONG PLOT

Injection F: <MC3> 5 5EX1015D,3,1

Sample name.....: LC1014SC
Sample ID.....: LC1014SC
INST ID: IN13, COL. TYPE: ODS-II, ID: CARB-05, INJ:200,COL#1

Acquired on 18-Oct-97 at 18:01:17
Reported on 05-Nov-97 at 09:34:05



INJECTION REPORT

Injection F: <MC3> 5 5EX1015D,3,1

Acquired on 18-Oct-97 at 18:01:17
Modified on 05-Nov-81 at 09:33:34
Reported on 05-Nov-97 at 09:33:41

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN13, COL. TYPE: ODS-II, ID: CARB-05, INJ:200,COL#1
Number of samples.: 26
Calibration file..: 5EX1015 Last modified on 05-Nov-81 at 09:33:30
Method file.....: EXPLOS Last modified on 05-Nov-81 at 08:54:26
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: LC1014SC
Sample ID.....: LC1014SC
Sample type.....: Sample
Sample amount.....: 2.0000
Number of injections.....: 1
Bottle Number.....: 1

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 10.000
Dilution.....: 2.000
Amount (g or mL).....: 2.000

PEAK INFORMATION

Peak	RT mins	Hght uV	Area uVs	ug/Kg	Peak name
1	4.827	29950	226545	32549.516	HMX
2	7.349	8688	116934	13628.680	RDX
4	9.989	13375	214261	11253.221	TNB
5	12.261	11939	214507	8425.253	DNB
6	12.875	1159	20366	1406.473	TETRYL
8	13.963	6268	126438	7411.366	NB
9	15.141	11152	220826	19102.609	3,4DNT 96
10	15.771	8043	168035	8564.482	TNT
11	16.453	6168	148184	10450.235	4ADNT
12	17.344	5935	144872	7569.091	2ADNT
13	18.480	4070	93518	7640.378	26DNT
14	19.136	4965	125263	4998.530	24DNT
15	22.533	5588	160608	15901.792	2NT

Peak	RT mins	Hght uV	Area uVs	ug/Kg	Peak name
16	24.096	2975	89525	10385.683	4NT
17	25.973	3721	118527	10658.943	3NT
Total		123996	2188410	169946.250	
Residual		517	6203	683.167	

1D
EXPLOSIVE ANALYSIS DATA SHEET

EPA SAMPLE NO:

LD1014SC

b Name: SWL-TULSA Contract:

Lab Code: SWOK Case No:MKF-OH SDG No:

Matrix: (soil/water) SOIL Lab Sample ID: LD1014SC

Sample Amt: 2gm % Moisture Date Received: / /

Extraction Volume: 10ml Date Extracted: 10/14/97

Extraction Method: SONC Date Analyzed: 10/18/97

GPC Cleanup: (Y/N) N Dilution Factor: 2.00

CAS NO.	COMPOUND	CONCENTRATION UNITS: (ug/L or ug/kg) ug/kg	Q
2691-41-0	HMX	32600	
121-82-4	RDX	13900	
99-35-4	TNB	10900	
99-65-0	DNB	8590	
479-45-8	TETRYL	968	
98-95-3	NB	7480	
118-96-7	TNT	8840	
1946-51-0	4ADNT	11300	
35572-78-2	2ADNT	7830	
606-20-2	26DNT	7920	
121-14-2	24DNT	5120	
88-72-2	2NT	16000	
99-99-0	4NT	10400	
99-08-1	3NT	10800	

SURROGATE AMOUNT SPIKED (ug/kg dry wt.) 20000
3,4-DNT

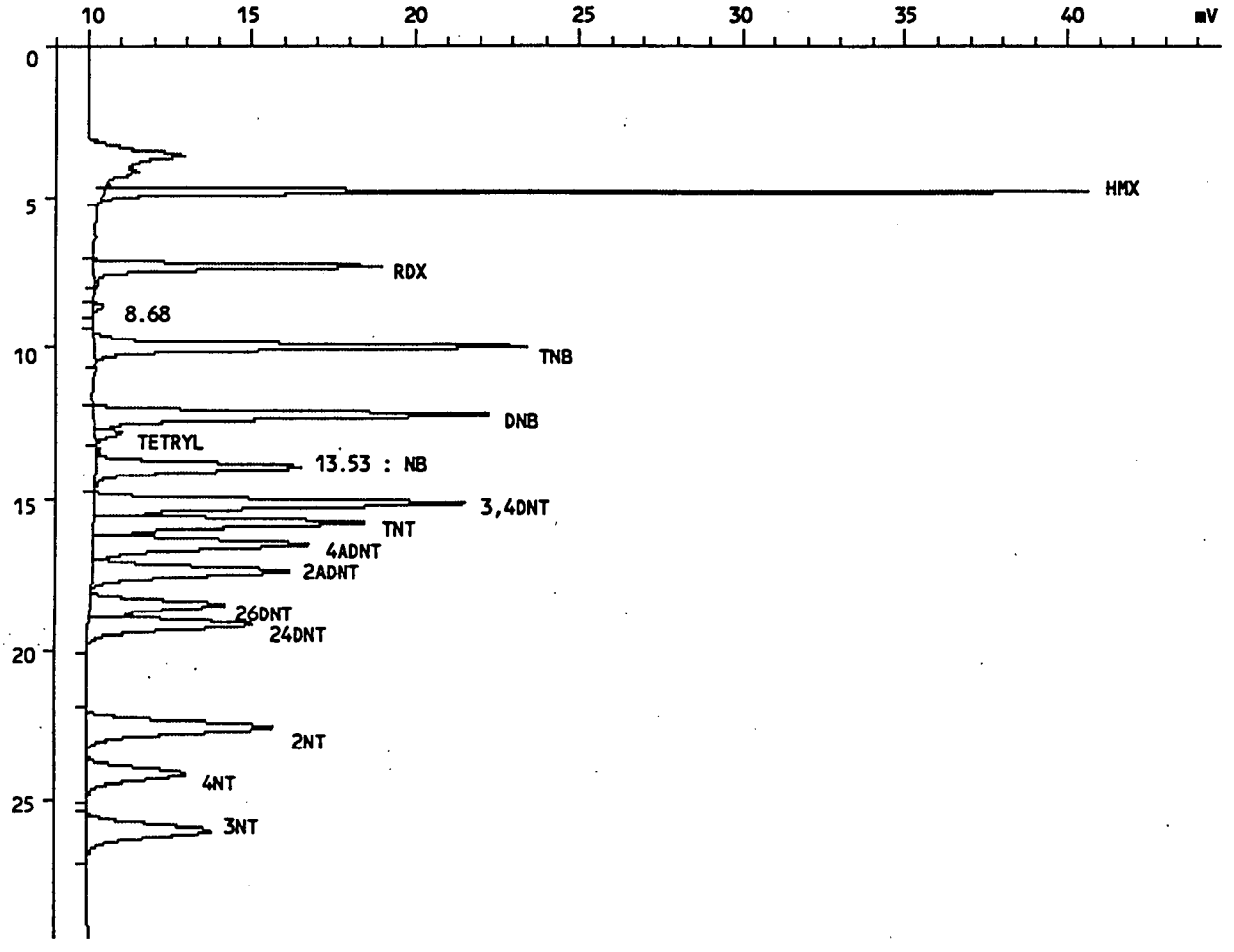
FORM I

LONG PLOT

Injection F: <MC3> 5 5EX1015D,4,1

Sample name.....: LD1014SC
Sample ID.....: LD1014SC
INST ID: IN13, COL. TYPE: ODS-II, ID: CARB-05, INJ:200,COL#1

Acquired on 18-Oct-97 at 18:45:57
Reported on 05-Nov-97 at 09:35:10



INJECTION REPORT

Injection F: <MC3> 5 5EX1015D,4,1

Acquired on 18-Oct-97 at 18:45:57
Modified on 05-Nov-81 at 09:34:24
Reported on 05-Nov-97 at 09:34:30

ANALYSIS INFORMATION

Analysis ID.....: EXP, GC 200
Analyst name.....: SS
Comment.....: INST ID: IN13, COL. TYPE: ODS-II, ID: CARB-05, INJ:200,COL#1
Number of samples.: 26
Calibration file..: 5EX1015 Last modified on 05-Nov-81 at 09:33:30
Method file.....: EXPLOS Last modified on 05-Nov-81 at 08:54:26
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: LD1014SC
Sample ID.....: LD1014SC
Sample type.....: Sample
Sample amount.....: 2.0000
Number of injections.....: 1
Bottle Number.....: 1

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 10.000
Dilution.....: 2.000
Amount (g or mL).....: 2.000

PEAK INFORMATION

Peak	RT mins	Hght uV	Area uVs	ug/Kg	Peak name
1	4.827	30171	226941	32606.447	HMX
2	7.349	8818	119014	13871.112	RDX
4	9.989	13224	207591	10902.880	TNB
5	12.256	12126	218812	8594.341	DNB
6	12.875	803	14024	968.513	TETRYL
8	13.968	6306	127533	7475.568	NB
9	15.147	11286	223330	19319.199	3,4DNT 97
10	15.781	8279	173489	8842.477	TNT
11	16.464	6562	160076	11288.854	4ADNT
12	17.371	6049	149799	7826.467	2ADNT
13	18.507	4140	96983	7923.466	26DNT
14	19.163	5041	128364	5122.253	24DNT
15	22.565	5672	161823	16022.107	2NT

Peak	RT mins	Hght uV	Area uVs	ug/Kg	Peak name
16	24.123	3017	89749	10411.674	4NT
17	25.995	3789	120736	10857.537	3NT
Total		125283	2218264	172032.891	
Residual		423	5220	559.835	

Attachment C

Field Test Kit Summary and Worksheets

TNT SOIL TEST KIT WORKSHEET

Abs background 0.000 Abs control 0.349

1	2	3	4	5	6
SAMPLE #	Abs initial	Abs sample	Abs initial x4	Abs final (Column 3 - Column 4)	TNT CONC ppm (Column 5/0.0323)
Clean 1	0.006	0.018	0.024	–	–
Clean 2	0.011	0.030	0.044	–	–
Reagent	0.001	0.009	0.004	0.005	0.16
A1 1/1000	0.015	0.481	0.06	0.421	13.03 x 1000 = 13,030
A2 1/1000	0.015	0.983	0.06	0.923	28.58 x 1000 = 28,580
1	0.023	0.272	0.092	0.18	5.57
2	0.017	0.047	0.068	–	–
3	0.038	0.177	0.152	0.025	0.77
4 1/10	0.019	0.177	0.076	0.101	3.13 x 10 = 31.3
5	0.019	0.041	0.076	–	–
6	0.018	0.05	0.072	–	–
7	0.017	0.037	0.068	–	–
8	0.017	0.064	0.068	–	–
9 1/20	0.012	0.245	0.048	0.197	121.98
10 1/50	0.013	0.354	0.052	0.302	9.35 x 50 = 468
11	0.016	0.177	0.064	0.113	3.5

TNT SOIL TEST KIT WORKSHEET

$Abs_{background}$ 0.002 $Abs_{control}$ 0.361

1	2	3	4	5	6
SAMPLE #	$Abs_{initial}$	Abs_{sample}	$Abs_{initial}$ x4	Abs_{final} (Column 3 - Column 4)	TNT CONC ppm (Column 5/0.0323)
12	0.015	0.071	0.06	0.011	0.34
13 1/50	0.016	0.189	0.064	0.125	3.869 x 50 = 194
14 1/50	0.016	0.109	0.064	0.045	1.393 x 50 = 69.7
15	0.027	0.74	0.108	0.632	19.6
16 1/5	0.017	0.402	0.068	0.334	10.34 x 5 = 51.7
17	0.021	0.032	0.084	–	–
18	0.027	0.071	0.108	–	–
19 1/10	0.02	0.178	0.08	0.098	3.03 x 10 = 30.3
20	0.022	0.209	0.088	0.121	3.8

RDX SOIL TEST KIT WORKSHEET

SAMPLE #	ABSORBANCE	RDX CONC., PPM $\frac{RDX_{abs} - 0.014}{0.0225}$
Clean 1	0.041	1.2
Clean 2	0.022	0.36
Reagent	0.043	1.3
A1 1/100	0.362	15.47 x 100 = 1547
A2 1/100	0.134	5.33 x 100 = 533
1 1/100	0.398	17.07 x 100 = 1707
2 1/100	0.341	14.53 x 100 = 1453
3 1/1000	0.033	0.844 x 1000 = 844
4 1/1000	0.071	2.53 x 1000 = 2530
5 1/1000	0.04	1.16 x 1000 = 1160
6 1/1000	0.044	1.33 x 1000 = 1330
7	0.72	31.4 over the cal limit
8 1/1000	0.027	0.577 x 1000 = 577
9 1/1000	0.044	1.333 x 1000 = 1333
10 1/1000	0.064	2.222 x 1000 = 2222
11 1/1000	0.032	0.8 x 1000 = 800
12 1/1000	0.04	1.155 x 1000 = 1155
13 1/1000	0.057	1.911 x 1000 = 1911
14 1/1000	0.041	1.2 x 1000 = 1200
15 1/1000	0.039	1.111 x 1000 = 1111
16 1/1000	0.042	1.244 x 1000 = 1244

1) Abs "background" 0.001 2) Abs "control" 0.293 3) Abs "Nitrate/Nitrite" _____

0.297

RDX SOIL TEST KIT WORKSHEET

SAMPLE #	ABSORBANCE	RDX CONC., PPM $\frac{RDX_{abs} - 0.014}{0.0225}$
17	0.423	18.18
18 x/1000	0.04	1.155 x/1000 = 1155
19 x/1000	0.048	1.511 x/1000 = 1511
20 x/1000	0.04	1.555 x/1000 = 1155

1) Abs "background" 0.002 2) Abs "control" 0.262 3) Abs "Nitrate/Nitrite" _____

0.297

0.211

Sheet1

MINE FILL A - Field Audit Demonstration

Sample ID	TNT	RDX
Clean 1	nd	1.2
Clean 2	nd	0.36
Reagent	0.16	1.3
A1	13,030	1547
A2	28,580	533
1	5.57	1707
2	nd	1453
3	0.77	844
4	31.3	2530
5	nd	1160
6	nd	1330
7	nd	31.4
8	nd	577
9	121.98	1333
10	468	2222
11	3.5	800
12	0.35	1155
13	194	1911
14	69.7	1200
15	19.6	1111
16	51.7	1244
17	nd	18.18
18	nd	1155
19	30.3	1511
20	3.8	1155

Attachment D

EPA Outline Summary

EPA OUTLINE FOR THE AUDIT DEMONSTRATION SUMMARY
BIOREMEDIATION FACILITY
NSWC CRANE
CRANE, INDIANA

Comment 1.

Select 12 sampling locations, providing an anticipated range of RDX soil contamination. The range should be distributed as follows:

a. 100 to 1,000 dilution factor needed. (Two samples)
b. Near but within the upper calibration limit of field instrument. (One sample)
c. Within the linear calibration range of field instrument. (Four samples)
d. Just above detection limit of field instrument. (Three samples)
e. Clean soil. (Two samples)

Response 1.

a. Complied with for both RDX and TNT
b. Complied with for TNT. RDX concentrations exceeded upper range.
c. Complied with for TNT. RDX concentrations exceeded upper range.
d. Complied with for TNT. RDX concentrations exceeded upper range.
e. Complied with for TNT. Only one for RDX.

Comment 2.

The Quality Control samples should include the following:

a. Three (composited & split/collocated) field duplicates (Both for colormetric test kits and 8330 method). These should be corresponded to:
   (1) A sample location thought to contain levels of contamination within the linear range of calibration,
   (2) A sample location thought to be just above the field detection limit, and
   (3) Clean soil.
b. Two MS/MSD per method (field test kits and method 8330) For TNT & RDX, spike at 8 to 10 ppm, wet weight. These samples should correspond to samples thought to be just above the detection limit of the respective field methods.

c. Will a "standard soil: be used?
d. Internal QC checks should include calibration verification, surrogate recovery data, LCS, and second column confirmation for 8330.
e. Two equipment rinse blanks.
f. Reagent blanks for RDX and TNT (acetone) - field test kits.
g. Multiple (composited) <u>split</u> sample analysis. (See item #5 below).
h. Method blanks for field and lab methods.

Response 2.

a. Complied with for TNT. RDX out of range but performed in duplicate.
b. MS/MSD samples performed on laboratory analysis only.
c. LCS's were used.
d. Complied with for TNT and RDX.
e. No Rinse blanks collected with these samples.
f. Complied with for TNT and RDX.
g. Samples were homogenized and split in the field.
h Complied with for TNT and RDX.

Comment 3.

Each soil sample should be homogenized at each location prior to splitting. (Follow the procedure suggested by US EPA Region 10. Soil cores (0 to 15 cm, 5.6 cm in diameter) should be placed in resealable plastic bags, and vegetation should be removed. The soil sample should be placed into 23 cm aluminum pie pans. The soil should be broken up using gloved hands and large rocks should be removed. (Sieving may also be applied). A second pie pan should be used to cover the sample, which should then be shaken and swirled vigorously to disperse and homogenize the soil. The sample should be coned and quartered, and subsamples about 5 gm in weight should be removed from each quarter and composited to form the 20 gm sample for analysis. Sufficient sample should be composited and homogenized in this way to support both the RDX, and TNT field tests as well as the 8330 confirmation analyses.

Response 3.

Samples were homogenized, quartered and split per standard procedures.

Comment 4.

Preservation of samples for 8330 samples should involve freezing. (A 4 degree C temperature is not sufficient for the TNT and nitroaromatics.) Also, the TNT field samples should be analyzed within two hours of sample collection.

Response 4.

Samples were frozen on sample collection and then shipped to the laboratory.

Comment 5.

The sample splitting procedure should be performed following homogenization, according to Region 10 guidance. Split the sample in triplicate. One sample of sufficient quantity will go to the lab. The other two will be analyzed in the field (for TNT and RDX). Split 3 soil samples, corresponding to contamination levels thought to be within the calibration range of the field test kits, in this manner again so that the splits can be analyzed multiple times by each method (field

and lab).

Response 5.

Samples were homogenized, quartered and split per standard procedures.

Comment 6.

A five-point initial calibration curve shall be used for all method 8330 explosives compounds. All explosives compounds should be reported.

Response 6.

Complied with for TNT and RDX.

Comment 7.

Provide all calibration HPLC printouts (field and lab), results for MS/MSD, blanks, surrogates, duplicates, LCS (internal QC checks).

Response 7.

Complied with for TNT and RDX.

Comment 8.

Appropriate correlations should be established between 8330 results and field test kit data.

Response 8.

Provided in summary table as percent differences.

Comment 9.

Confirm all 8330 detects using second column confirmation. Reporting limits for 8330 and test kit data should be less than the soil DQL values for respective target compounds in clean soil.

Response 9.

Complied with for TNT and RDX.

Comment 10.

Use high purity acetone for field RDX tests. Histology or commercial grade is not acceptable.

Response 10.

Complied with for TNT and RDX.

Comment 11.

Follow criteria from CRREL Report 90-38 for two times background subtraction for TNT.

Response 11.

Sample analysis performed per methodology.

Comment 12.

Cleanest areas should be sampled first. Referring to item #1, the order of sampling should be E through A.

Response 12.

Concentrations of soil were unknown. Sampling equipment was decontaminated appropriately between sampling locations.

Comment 13.

The deficiency comments generated as a result of the U.S. EPA's review of the "QAPP for Pilot Scale Operations at the Biofacility", dated 11/1/96, Appendix C should be factored into the protocol for both RDX and TNT testing prior to implementation of this Audit Demonstration Plan.

Response 13.

QAPP comments previously incorporated as appropriate.