QUALITY ASSURANCE PROJECT PLAN FOR PILOT SCALE OPERATIONS AT THE BIOFACILITY

NSWC CRANE,
CRANE, INDIANA

U.S. EPA ID NUMBER [IN5 170 023 498]

REVISION 5

June 6, 1997

Prepared for:

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MK Program Manager — Date: 17 June 97

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US NAVY Responsible Authority — Date: 17 Jul 97

USEPA RCRA Project Coordinator — Date: 6/13/97

USEPA Region 5 QAPP Coordinator — Date: 6/13/97

Laboratory Representative — Date: 7/15/97

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 5
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CHICAGO, IL 60604-3590

REPLY TO THE ATTENTION OF: DRP-8J

June 13, 1997

Electronically and Hand Delivered

Mr. Jim Hunsicker, Director
Environmental Protection Department
Department of the Navy
Crane Division
Naval Surface Warfare Center
300 Highway 361
Crane, Indiana 47522-5000

RE: Operational Plan and QAPP
Bioremediation Facility
Naval Surface Warfare Center
Crane, Indiana
IN5 170 023 498

Dear Mr. Hunsicker:

The United States Environmental Protection Agency (U.S. EPA) hereby approves with modifications the final Pilot Scale Operational Plan and the final Quality Assurance Project Plan (QAPP) for the Bioremediation Facility. Enclosed is a final electronic and paper version of each of these documents. The U.S. EPA's modifications strikeout what is no longer applicable, and bolds and/or underlines text that was added. Please be aware that we expect the following from the U.S. Navy:

1. There must be compliance with this specific plan. If changes are anticipated or needed, they must be sent in writing to this office. The Contractor is not given any permission to deviate from the plan without both U.S. EPA and U.S. Navy approval. There are certain areas such as performance criteria in daily monitoring of the windrows that will require the Contractor's judgement. Documentation must support those actions.

2. The U.S. EPA must be notified verbally of the progress of the project on a weekly basis. We are requesting a three day notice prior to clean soil testing, and a two week notice prior to contaminated soil testing. Daily performance monitoring records shall be faxed by the next day.

3. The U.S. EPA Region 5 Office in conjunction with the Region 10 office will be on-site to take split samples on Day 0 of the contaminated soil portion of the test.

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4. The U.S. Navy and the U.S. EPA must meet to discuss the clean soil testing results prior to beginning the contaminated soil testing.

5. Any explosive hazard analysis modifications to equipment or the operations must be in place prior to the contaminated soil testing. Any changes in the approved plan in order to allow for explosives safety must be submitted as an amendment request.

6. The U.S. EPA plans to perform a field inspection with record auditing sometime during the pilot scale test. This may be an announced or a surprise inspection.

7. We would encourage good photo documentation and video of the entire process to support the program.

8. The U.S. EPA believes that the yucca plants growing in the higher contaminated soil areas of Mine Fill A may provide some potential future research in phytoremediation technologies. We are asking that the U.S. Navy transplant these plants with some soil still surrounding the roots and maintain them until we can work on a joint effort into this research. We will begin discussing this with our Office of Research and Development in July.

Our Agency is happy to see the finalization and approval of these plans. We hope that the Pilot Scale Test will be a success. If you have any questions regarding this matter, or have any questions during testing, please contact me at (312) 886-6146.

Sincerely,

Carol Witt-Smith
Corrective Action Expert
WMB, IL/IN/MI Section

cc:

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QUALITY ASSURANCE PROJECT PLAN

FOR PILOT SCALE OPERATIONS AT THE BIOFACILITY

NSWC CRANE
CRANE, INDIANA

CONTRACT #N62467-93-D-1106
DELIVERY ORDER #0009
STATEMENT OF WORK #007

REVISION 4

June 6, 1997

Prepared for:

SOUTHERN DIVISION
NAVAL FACILITIES ENGINEERING COMMAND
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TABLES

TABLE		PAGE
4-8	NPDES DISCHARGE LIMITATIONS	4-30
6-1	CALIBRATION CRITERIA - LABORATORY METHODS	6-4
7-1	SUMMARY OF SOPs	7-3
9-1	DATA SET DELIVERABLES	9-5
11-1	SUMMARY OF MAINTENANCE - FIELD EQUIPMENT	11-2

FIGURES

FIGURE		PAGE
1-1	SUMMARY OF PILOT SCALE OPERATIONS	1-27
1-2	VICINITY MAP OF NSWC CRANE, INDIANA	1-28
1-3	LOCATION OF SOLID WASTE MANAGEMENT UNITS	1-29
1-4	MINE FILL A	1-30
1-5	LAYOUT OF CENTRAL MINE FILL A	1-31
2-1	ORGANIZATION CHART	2-7
4-1	SAMPLING AREAS - MINE FILL A	4-31
4-2	EXAMPLE GRID LAYOUT - BUILDING 153/2793 VICINITY	4-32
4-3	PILOT SCALE - WINDROW SAMPLE LOCATIONS	4-33
5-1	FIELD CUSTODY DOCUMENT	5-10

APPENDICES

APPENDIX		PAGE
A	FIELD DATA SHEETS	A-1
B	LABORATORY REPORTING LIMITS FOR PILOT SCALE BIOFACILITY PROJECT ANALYTICAL METHODS	B-1
C	(RESERVED)	
D	LABORATORY SAMPLE CUSTODY PROCEDURES	D-1
E	PROCEDURES FOR FIELD INSTRUMENTS	E-1
F	LABORATORY STANDARD OPERATING PROCEDURES	F-1
G	EXAMPLE DATA DELIVERABLE PACKAGE (EXPLOSIVES)	G-1
H	NEESA 20.2-047B - SAMPLING AND CHEMICAL ANALYSIS QUALITY ASSURANCE REQUIREMENTS FOR THE NAVY INSTALLATION RESTORATION PROGRAM	H-1

SECTION 1
PROJECT DESCRIPTION

1.1 INTRODUCTION

1.1.1 Project Background

Naval Surface Warfare Center (NSWC) Crane Division, is located in southwestern Indiana and provides support for equipment, shipboard weapons systems, and ordnance. NSWC Crane also supports the Crane Army Ammunition Activity (CAAA), including production and renovation of conventional ammunition, storage, shipment, and demilitarization and disposal of conventional ammunition.

Promulgation of the USEPA's Resource Conservation and Recovery Act (RCRA) hazardous waste regulatory program provided the impetus to identify and control environmental contamination from past practices at NSWC Crane. Corrective action programs established as part of the RCRA Hazardous and Solid Waste Amendments (HSWA) required NSWC Crane to address past releases of hazardous waste or hazardous constituents at selected Solid Waste Management Units (SWMUs).

Following promulgation of the USEPA's RCRA hazardous waste regulatory program, NSWC Crane filed notification and application to operate as a treatment, storage and disposal (TSD) facility in October 1980. Interim status was granted, subject to operating requirements and applicable technical standards found in Title 40 Code of Federal Regulations, Part 265 (40 CFR 265).

In April 1981, the Navy implemented the Navy Assessment and Control of Installation Pollutants (NACIP), subsequently known as the Installation Restoration Program (IRP). The purpose of the NSWC Crane IRP was to identify and control environmental contamination from past practices. As part of the IRP, an Initial Assessment Study (IAS) was conducted in April and May 1981, by a team from the Naval Energy and Environmental Support Activity (NEESA), the Ordnance Environmental Support Office, and the Army Corps of Engineers. Based on information from historical records, aerial photographs, field inspections and personnel interviews, the IAS identified seventeen potentially impacted sites at NSWC Crane. Fourteen of the seventeen sites were recommended for confirmation studies to include sampling and monitoring.

Pursuant to RCRA regulations and the IRP, hydrogeologic investigations, including ground water monitoring, were initiated at ten sites in 1981. Eight of these ten sites were later identified as warranting further investigation in the IAS. In November 1984, the Naval Facilities Engineering Command - Northern Division (NORTHDIV) contracted the Hazardous Material Technical Center (HMTC) to perform confirmation studies at several sites at NSWC Crane.

Corrective action programs established as part of the RCRA HSWA required NSWC Crane to address past releases of hazardous waste or hazardous constituents at the SWMUs. Accordingly, NSWC Crane submitted a Hazardous Waste Management Report to the USEPA in January 1985. The report listed the IAS-identified hazardous waste sites as SWMUs. Following the Hazardous Waste Management Report, a RCRA Facility Assessment was conducted to characterize the potential for releases of hazardous waste or constituents from 100 SWMUs.

On December 23, 1989, the USEPA issued the federal portion of the Final RCRA Permit for NSWC Crane to the US Navy and issued the permit renewal for a period of five years on July 31 1995. This permit contains both the Federal Permit Conditions and State Permit Conditions, which were issued separately by the State of Indiana RCRA program. This permit established the HSWA Corrective Action Requirements and Compliance Schedules obligating the US Navy to perform RCRA Facility Investigations (RFIs) at 30 SWMUs, conduct Corrective Measures Studies (CMSs) and implement corrective measures if needed.

The RCRA Permit for NSWC Crane requires that RFI Phase I Environmental Monitoring Reports (EMRs) be submitted from many of the SWMUs at NSWC Crane. The purpose of the EMR is to identify and assess IRP Corrective Action SWMUs posing potential threat to human health or the environment due to contamination from past hazardous material operations. The EMRs for SWMUs #12/14 (Mine Fill A) and #13/14 (Mine Fill B) were completed in August 1992 (Halliburton NUS, 1992). The Pilot Scale Phase of the Interim Measures Cleanup which will be performed under this scope of work are a result of the findings presented in the EMRs.

1.1.2 Scope of Work and Overall Objectives

The scope of the first phase of interim measures cleanup includes Pilot Scale Operation at the Bioremediation Facility (Biofacility), located within NSWC Crane, under the direction of the Southern Division Naval Facilities Engineering Command (Southern Division). Remediation activities will be performed as specified by Delivery Order #0009, Statement Of Work #007, under the Environmental Remedial Action Contract (ERAC) No. N62467-93-D-1106.

The objective of the Pilot Scale testing is to define and optimize design and operation criteria for the overall purpose of interim measures cleanup (soil excavation, treatment and disposal) at four SWMUs at NSWC Crane (#03/11 Ammunition Burning Ground, #10/15 Rockeye, #12/14 Mine Fill A, and #13/14 Mine Fill B). Interim measures actions will be completed in compliance with State of Indiana and EPA Region 5 guidance and requirements. Data collected as part of this Pilot Scale Operation will be used to guide future remediation activities in the Full Scale Operations for interim measures cleanup.

Prior to instituting full scale interim measures cleanup, the Pilot Scale study will be conducted to evaluate and finalize soil treatment goals at the Biofacility, using soil excavated from a single SWMU, #12/14, Mine Fill A. Mine Fill A was chosen based on current operations, past activities, and contaminant levels.

The primary objective of the Pilot Scale phase is to:

- Determine optimum mix design and operational parameters which will meet Pilot Scale process goals of 30 mg/kg for TNT and RDX, as these are primary target compounds; and initial remedial levels for HMX and other target compounds (i.e. explosives).

Initial remedial goals are drawn from Preliminary Remedial Goals (PRGs) developed by Region 9 (USEPA, 1996b). TNT and RDX PRGs will not be used for this project. Rather, two process goals have been developed for these explosives, based on composting practices at other sites. Further background information on the initial remedial goals and TNT and RDX process goals is provided in Section 1.4.

Additional Pilot Scale objectives include:

- Identifying areas for Pilot Scale excavation at Mine Fill A, with respect to explosives,

- Determining "startup values" of explosives in soil entering the Biofacility,

- Determining if soil entering the Biofacility is hazardous with respect to metals and VOCs. Soil containing hazardous levels of metals or VOCs will be not be accepted into the Biofacility for treatment,

- Demonstrating effectiveness of composting on a large scale, and

- Properly disposing of waste water produced (i.e. storm water collected, decontamination water) in compliance with NPDES permit and POTW requirements, as applicable.

Performance goals serve to connect the Pilot Scale results and startup of Full Scale Operations. These goals include:

- Finalizing process goals for TNT, **HMX** and RDX **and other constituents**, which will be used during full scale operation.

- Providing data to evaluate the biological treatment efficiency based on the National Contingency Plan (NCP) remediation criteria for innovative treatment technologies.

Pilot Scale Operations will include:

1. Initial characterization of soil, to determine (a) areas for Pilot Scale excavation, (b) that soil to be excavated and treated at the Biofacility is not hazardous with respect to metals or VOCs, and © initial explosives concentrations in soil to be treated.

2. Excavation and treatment of approximately 200 cubic yards of soil from Mine Fill A.

3. Process data analysis and optimization of mixtures throughout treatment.

4. Confirmation sampling (treated compost piles). Confirmation sampling at the excavations will be deferred to Full Scale Operations. The excavation(s) created as part of the Pilot Scale phase will be sampled prior to temporary backfilling to determine what levels of explosives, metals and VOCs remain.

5. Data review to determine if treated soil must be reprocessed, placed in an on-site landfill, removed for off-site treatment, or placed back in the excavation as clean fill during Full Scale Operations.

6. Continuous evaluation of target parameters and initial remedial goals/levels and development of site-specific risk-based concentrations to confirm one or more of the following outcomes, depending on the compost piles evaluated:

    a. Mixture (recipe) is <u>not</u> suitable for meeting treatment goals. The pile(s) must be reprocessed using a different mixture, or

    b. Treatment <u>is</u> <u>successful.</u> The mixture (recipe) is finalized and proceed to determine if:

    -Compost is suitable for placement in on-site landfill, or

    -Compost is suitable for placement as backfill.

    c. Treatment <u>is</u> <u>unsuccessful</u> using all recipes. Treated compost must be shipped off-site for disposal or retreated using a new mixture developed during further bench scale testing. Off-site disposal sampling will be completed during Full Scale Operations.

These activities are summarized in Figure 1-1.

1.1.3 QAPP Preparation Guidelines

This Quality Assurance Project Plan (QAPP) presents the organization, objectives, planned activities, and specific Quality Assurance/Quality Control (QA/QC) procedures associated with the Pilot Scale study. Sampling and data evaluation activities are summarized in Figure 1-1, Sampling and Analysis Logic Diagram. Specific protocols for sampling, sample handling and storage, chain-of-custody, and laboratory analyses will be described. All QA/QC procedures will be structured in accordance with applicable technical standards, certain US Environmental Protection Agency's (USEPA) requirements, regulations, guidance, and technical standards. This QAPP was prepared in accordance with a guidance manual entitled, "Region 5 Model RCRA Quality Assurance Project Plan," May, 1993.

1.2 SITE/FACILITY DESCRIPTION

A brief description of the facility and associated features are presented in the sections below.

1.2.1 Location

NSWC Crane is located in southwestern Indiana and occupies the northern half of Martin County and parts of Lawrence, Green and Daviess counties, approximately 30 miles southwest of Bloomington (Figure 1-2). The site encompasses more than 100 square miles (62,463 acres) including 800-acre Lake Greenwood. NSWC Crane provides Naval support for equipment, shipboard weapons systems and ordnance. In addition, NSWC Crane supports the CAAA including production and renovation of conventional ammunition, as well as storage, shipment, demilitarization and disposal of conventional ammunition.

Mine Fill A is located in the west-central portion of NSWC, and occupies the south-central portion of Navy-designated Sheet 17 of the NSWC General Development Maps. Mine Fill A is accessible from NSWC Highway H-45 (Figure 1-3).

1.2.2 Facility/Size and Borders

Mine Fill A consists of 29 buildings and is bordered to the north/northwest by Highway H-45 and Highway H-17 along its remaining perimeter. The entire site occupies approximately 50 acres. The Pilot Scale will require 200 cubic yards of soil which will be excavated in the vicinity of Building 153. If sufficient soil is not available, soil in the vicinity of Building 158 may be used. Note Figures 1-4 and 1-5 for the locations of these buildings.

1.2.3 Natural and Manmade Features

Buildings at Mine Fill A provide storage, receiving, and filling of munitions/bomb materials. Local facilities include Mine Fill B to the southwest, an inert storage area to the west, the pyrotechnics production area to the north, and the Supply Department, a restricted explosive area and high explosives magazines to the northeast (Halliburton NUS, 1992).

Surface drainage is primarily off the eastern and western borders of Mine Fill A, via ditches and culverts. Surface discharge is forwarded to intermittent drainageways which flow to Turkey Creek, which flows south-southwest to merge with Boggs Creek (Halliburton NUS, 1992).

1.2.4 Topography

The topography is gently sloping from the northeast towards the southwest. Surface elevation varies from approximately 745 feet mean sea level (MSL) to the north to approximately 725 feet MSL along the western edge of Mine Fill A. See Section 5.1.1.4 of the August 1992 EMR (Halliburton NUS, 1992) for information concerning the site's topography.

1.2.5 Local Geology

Mine Fill A is covered with well-drained to moderately well-drained soil consisting of a broad range of grain sizes, depending on depth. Surface soils include a mixture of clay, silty clay and silt. Deeper soils include clay, sandy clay, silty sand and silty gravel. Depth to bedrock (Pennsylvanian Age Raccoon Group - sandstone, shale,

limestone) ranges from 45 to over 65 inches, depending on earth moving activities in the area. See Section 5.1.1.6 of the August 1992 EMR (Halliburton NUS, 1992) for more information.

1.3 SITE/FACILITY HISTORY

1.3.1 General History - NSWC Crane

In 1940, Congress authorized construction of a Naval Ammunition Depot (NAD) in southern Indiana, and in late 1941, the Burns City Ammunition Depot was commissioned. In 1943, NAD Burns City was renamed NAD Crane, and the Town of Crane was built to house the rapidly growing number of civil service employees. NAD Crane's overall mission was to load, prepare, renovate, receive, store and issue ammunition to the fleet.

During World War II, NAD Crane's mission expanded to include pyrotechnics production, mine filling, rocket assembly, field storage, torpedo storage, and ordnance spare parts and mobile equipment storage. During the 1950s, several new departments were created, the Ammunition Loading and Production Engineering Center (ALPEC) was transferred to Crane, and the Central Ammunition Supply Control Office (CASCO) was established. NAD Crane supplied ammunition to the fleet during the Korean and Vietnam conflicts.

In 1976, NAD Crane was renamed NSWC Crane. Its new mission was to provide support for ships and crafts equipment, shipboard weapons systems and assigned ordnance items, and to perform additional functions as directed.

In 1977, the Single Manager Concept was implemented. The CAAA was created, and the Army assumed ordnance production, storage, and related responsibilities as a tenant organization. Other functions remained Navy, and currently the Navy retains ownership of all real estate and facilities at NSWC Crane. Responsibility for overall station safety, security and environmental protection remains with the Commanding Officer, NSWC Crane.

Until the late 1970s and early 1980s, a variety of liquid wastes from both ordnance and non-ordnance activities were disposed on site without treatment. For example, ordnance-impacted waste waters were discharged into unlined ponds or into storm drains and ditches; waste waters generated from cleaning operations were discharged into an unlined pond; oily waters, battery acids, paint thinner, acetone, boiler blowdown and solvents were dumped behind buildings or into ravines; and waste hydraulic oil was spread on dirt roads. Since that time, treatment plants have been built, waste water streams have been connected to the sanitary sewer, and oils and solvents have been collected, segregated, and either recycled, sold, or properly disposed.

Air pollution control devices were not installed until the late 1970s. Therefore, ventilation systems at bomb loading facilities and the incinerator had no scrubber systems until the 1970s. Waste management activities included the burning of solid wastes and subsequent disposal of ash in unlined pits or gullies. Also, dye and dye-impacted materials have been disposed in on-site trenches.

Operations at NSWC Crane also included radiological activities and some radioactive materials were disposed on site during the early 1950s. Known burial grounds were excavated, decontaminated and released for unrestricted use in 1974.

1.3.2 Mine Fill A History

In September, 1942, Mine Fill A began production with the cast loading of depth charge bombs. Numerous items, including rocket heads, depth charges, projector charges and ammunition, were produced. The area was also utilized during the Korean and Vietnam Wars. Production rates were not available for the Korean War, but over six million bombs were manufactured at Mine Fill A and Mine Fill B during the Vietnam War era alone. Mine Fill A continued to produce ordnance until 1975, when production was suspended. Production was resumed in 1980. More details on production may be found in Sections 5.4.1 and 6.4.1 (similar operations at Mine Fill B), in the August 1992 EMR.

1.3.3 Past Data Collection Activities - Mine Fill A

Based on operational history, physical condition, location, waste characteristics, visual evidence, and analytical data, releases of explosives contamination to surface water and soil have occurred at Mine Fill A. Effluent, runoff and ditch water samples were collected in 1972 and 1979. TNT, RDX and HMX were detected in concentrations ranging from non-detected to 35.3 mg/L (RDX).

Several soil borings were installed in the vicinity of Buildings 153 and 158 in 1985. Results are summarized in Table 1-1, with estimated locations provided in Figure 1-5. The samples were analyzed for TNT, RDX and HMX only, and results showed the highest levels of explosives are in the vicinity of Building 153. Further discussion of surface water, ground water and soil sampling results may be found in Section 5.3.2 of the August 1992 EMR (Halliburton NUS, 1992).

1.3.4 Current Status

Mine Fill A is currently an active site. As of 1992, Mine Fill A was used for the production of 2,000-pound aerial bombs. Demilitarization activities are also conducted at Mine Fill A. Activities include the application of enamel and bituminous solvent coatings, rotary grit blasting, spray painting, propellant removal, and fuse and configuration changes. Ordnance compound particulate matter was exhausted through the ventilation system. Rinse and waste water containing TNT, Composition B, HBX-1, HBX-3, and H6 are potential sources. Chemical composition of these and other wastes generated at Mine Fill A are provided in Table 1-2.

According to the August 1992 EMR produced by Halliburton NUS, other potential wastes at Mine Fill A include sludge (containing explosives), pink water (TNT), grit blast residue (containing cadmium, chromium, lead and barium) and paint waste (containing chromium, lead, methylene chloride, acetone, ethanol, isopropanol, methanol, methyl ethyl ketone, methyl isobutyl ketone, naphtha, toluene and xylene).

Based on reports and documents reviewed for the site, and a current assessment of available information, concentrations of individual ordnance compounds in soils ranged from undetected to 15,300 mg/kg (RDX). It was not verified whether volatile organic compounds (VOCs) were tested or detected in Mine Fill A soils. Five paint booths are

authorized to operate in Building 155, and are equipped with particulate control features. However, volatile organics samples will be collected during initial excavation site characterization, based on field observations, and if paint wastes were associated with Buildings 153 or 158.

Approximately 11,000 cubic yards of impacted soil within the Mine Fill A are targeted for interim measures cleanup. A portion of this soil targeted for excavation will be transported to the Biofacility for Pilot Scale operations. Based on previous soil boring results (Table 1-1), the areas around Building 153 show higher levels of explosives contamination than Building 158.

Confirmation soil sampling within each excavated area will be discussed in the Full Scale Operational Plan and QAPP, which will be issued at the completion of the Pilot Scale Operation.

1.4 PROJECT OBJECTIVES

1.4.1 Specific Objectives and Associated Tasks

For this Pilot Scale operation, it will be necessary to gather sufficient information to evaluate the soil excavated for treatment and the effectiveness of each compost mix. Associated data collection activities are summarized in Table 1-3 and as follows:

Data Collection Task 1 - Initial Excavation Site Characterization

Previous data collected at the site (Mine Fill A) indicates that high levels of explosives contamination were detected in the vicinity of Buildings 153 and 158. Based on site history, wastes present at Mine Fill A may include TNT, RDX, HMX, metals and VOCs. Although explosives were detected in high concentrations, it is not clear if soil in the vicinity of these buildings is non-hazardous, with respect to 40 CFR 261. The Pilot Scale phase will focus on explosives and site-specific chemicals only.

This task has three analytical objectives:

1. Analyze the soil, primarily in the vicinity of Building 153 or Building 158 (backup in case Building 153 cannot be used during the Pilot Scale) for explosives. This data will primarily be used to identify areas to be excavated for Pilot Scale Operations. This data will not be sufficient to indicate full extent of contamination at Mine Fill A. Additional excavation at Mine Fill A will be completed during Full Scale Operations. This data will also be used as "entry " or "startup" concentrations for composting.

2. Analyze the soil for site-specific VOCs and metals to determine if other waste products are present. Certain observations will dictate a need for VOCs testing. **Observations and criteria include visibly stained soils, unusual odors, and PID readings exceeding resident background levels. If one or more of these criteria is discovered, additional samples for VOCs will be collected. Analysis will be approved by the Project Manager and U.S. Navy designee, as required. It is anticipated that "low level" (if any) VOC contamination will be present.** Site specific VOCs include methylene chloride, acetone, methyl ethyl ketone, methyl isobutyl ketone, toluene and xylene. These constituents will serve as indicators of the presence of paint wastes. Depending on VOC results, additional testing for naphtha, methanol, isopropanol and ethanol may be required. Site specific metals include barium, cadmium, chromium and lead.

3. Analyze the soil to determine if it is hazardous with respect to metals and VOCs. Soil will not be accepted into the Biofacility for treatment if it is hazardous with respect to these constituents.

Soil borings, approximately two-feet deep, will be installed using a drive sampler at or near the previous sampling locations shown in Figure 1-5. Soil will be collected from two depths (6 inches and 2 feet below ground surface) and tested for explosives, site-specific metals (barium, cadmium, total chromium and lead), TCLP-metals, and total VOCs (TCLP list and site-specific VOCs). One sample will be collected for every 25 cubic yards (approximately) to be potentially excavated.

Soil samples collected from each boring will also be screened with a PID and examined. Should PID readings be above background levels for the site, or staining/odors be observed, additional samples may be collected for VOCs analysis.

Based on explosives testing results, approximately 200 cubic yards will be excavated for the Pilot Scale and temporarily staged at Mine Fill A. If sufficient explosives-contaminated soil is not available at Mine Fill A for the Pilot Scale, excavation will focus on the use of Mine Fill B soils.

Soil will be excavated and passed through a screen to remove coarse fractions. Coarse fractions (gravel, cobbles, boulders) from the soil screen will be washed, as prescribed in the Operational Plan, and used as natural backfill.

Data Collection Task 2 - Equipment Wash Water/ Decontamination Water Sampling

Decontamination procedures described in Section 6.2 of the Pilot Scale Operational Plan will yield a quantity of water which may be used during initial windrow formation. Once the treatment process is underway, decontamination water will no longer be added to the process; only potable water will be used for moisture addition. Rock wash and other decontamination fluids will then be treated at a waste water treatment facility at Crane. The water will be sampled for explosives and other parameters, as required by the on-site treatment facility.

Solids and liquid which collect in Biofacility sumps will also be sampled for explosives. Similarly, the sump solids and liquids will be added to the composting process during the initial phase only.

Data Collection Task 3 - Storm Water Retention Pond Sampling

Residual surface runoff will be collected into storm water retention ponds. This water will be sampled as described in Section 4.0, and may be potentially used for water addition during windrow formation. Data will be evaluated to determine if water can be discharged per the limitations and requirements of Crane's NPDES permit.

If water cannot be discharged, it will be sampled for site-specific metals to permit use in

composting activities. If the water cannot be used during composting, it will be either treated and disposed at the on-site POTW or disposed of ~~off-site~~ **according to Navy EPD approved procedures.**

Data Collection Task 4 - Process Sampling

Each amendment mixture will be sampled prior to adding soil to be treated. The amendment material will be tested for TOC and TKN to develop a "startup" ratio of nitrogen to organic carbon. This data will be compared to TOC and TKN results from process testing.

Process parameters which will be tested on-site include pH, temperature, oxygen and % moisture. These parameters are further discussed in Section 7.2 and 8 of the Operational Plan.

Process parameters requiring off-site analysis include total metals (barium, cadmium, chromium, lead), TCLP-metals, TKN, TOC, and explosives (full Method 8330 list). Depending on the results of VOCs testing during Task 1, VOCs may be added to the list of process parameters.

Approximately 12 samples will be collected per compost pile at time intervals defined in Section 4.0. Explosives, TKN and TOC will be tested throughout the treatment process. Total metals (barium, cadmium, chromium, lead) and TCLP-metals will be tested at Day 0 and Da y ~~28~~ **60** (end of process). If metals are not detected in any particular pile, based on the Day 0 sampling results, metals testing at Day 28 **60** may not be required.

During the Pilot Scale, the full suite of explosive compounds (standard list - Method 8330) will be monitored during treatment. "Startup" concentrations and rates of decomposition of daughter compounds and other compounds versus TNT, RDX and HMX will be evaluated. The data will be evaluated to determine if direct correlation is found between percent reduction of TNT, RDX and HMX and percent reduction of other explosives, as TNT, RDX, and HMX are primary target compounds.

At the completion of the treatment process ~~(Day 28)~~, (**Day 60**) confirmation samples will be collected and submitted to the off-site laboratory for analysis of explosives, metals

(barium, cadmium, total chromium, lead), and TCLP-metals, depending on initial excavation site characterization results. Confirmation sampling will not be completed unless concentrations of TNT and RDX are at or below process goals.

Confirmation sample results will be compared to those levels in Table 1-4 to determine if TNT and RDX are below process goals and other parameters are below initial remedial goals (industrial PRGs). HMX degradation will also be closely evaluated. This data will be used to determine if treatment must continue using the same mixture, the mixture must be changed, treatment is complete or treatment has failed using all mixtures.

If treated soil does not meet process goals (i.e. TNT, RDX**, HMX**), it will be retreated using a different process mixture (recipe). Following reprocessing, if again the treated soil does not meet process goals, does not indicate a 40 percent or greater reduction in target compounds, and is unsuitable for placement in the landfill, it may require off-site disposal. This issue will be discussed during Full Scale Operations.

Data Collection Task 5 - Post-Excavation Sampling

Once soil boring sampling is completed and the results reviewed (Task 1), a minimum of 200 cubic yards will be excavated for the Pilot Scale test. Additional soil excavation at Mine Fill A will be deferred to the Full Scale Operations phase.

Once soil has been removed, the Pilot Scale excavation at Mine Fill A will be sampled and temporarily backfilled from an approved source at Crane. The on-site borrow source has previously been sampled and approved based on analytical results which will be supplied prior to temporary backfilling operations.

A sampling grid will be laid out in the base of the Pilot Scale excavation(s), with a nodal spacing of 20 feet. Samples will be collected six inches below ground surface from each 20-foot by 20-foot grid block (base) and analyzed for explosives, site-specific VOCs (methylene chloride, acetone, methyl ethyl ketone, methyl isobutyl ketone, toluene and xylene) and metals (barium, cadmium, chromium and lead). Sidewalls will

be sampled every 20 feet and will not be gridded. The purpose of this sampling is to determine the level of contamination remaining in the excavation, with respect to explosives and potential site-specific contaminants. The data will be used during the Full Scale Operations to guide further excavation.

1.4.2 Project Target Constituents and Laboratory Data Use

1.4.2.1 Target Constituents

Three primary target constituents have been identified for the Pilot Scale operations: TNT, RDX and HMX. These parameters are present in many of the waste streams generated at Mine Fill A and have been detected in soil and surface water. Additional target constituents include site-specific metals and VOCs as these have also been identified in waste stored or generated at Mine Fill A.

1.4.2.2 Initial Remedial Goals

Soil will be excavated and treated to concentration levels which are protective of human health and ecology, as treated soil will be placed in an on-site landfill or used as backfill in the original excavation (Full Scale Operations task). Soil which is not successfully treated will **be managed in accordance with this QAPP.** Bench scale testing may be performed as an alternative to off-site disposal, to determine if other mixtures (recipes) can be used to improve treatment.

Pilot Scale testing success will be governed primarily by process goals for TNT and RDX (30 mg/kg, respectively) and remedial goal for HMX (34,000 mg/kg). Decreases in concentrations of other explosives will also be carefully evaluated. Process goals for TNT and RDX are further discussed below.

Preliminary Remedial Goals (PRGs) developed by Region 9 (USEPA 1996b) will be used as initial remedial goals for the remaining target constituents during the Pilot Scale phase. Industrial levels for the target constituents are listed in Table 1-4. Concentrations were derived by Region 9 using EPA toxicity values from the integrated risk information system (IRIS), updated through July 1996, HEAST, updated through May 1995, and EPA's National Center for Environmental Assessment.

Due to ongoing operations at Mine Fill A, industrial levels will be used as "ceiling levels" or initial remedial goals for treatment during the Pilot Scale. Reporting limits listed in Table 1-4 are provided by Southwest Laboratory (Broken Arrow, Oklahoma).

Initial remedial goals will be reviewed in the framework of the conceptual model for Mine Fill A, as well as the other SWMUs being considered for interim measure action. The conceptual model will be updated by the USEPA Region 5, using data collected during the Pilot Scale phase and assumptions and models used in current risk assessment activities. Initial remedial levels and any data generated during the Pilot Scale will be provided to the EPA to assist in risk assessment activities for NSWC Crane.

Two explosives, 2,4-DNT and 2,6-DNT, are considered Group B (probable human) carcinogens. PRGs for these explosives are 1,400 mg/kg and 680 mg/kg, respectively.

TNT and RDX Process Goals

As was demonstrated at the Umatilla Army Depot (UMDA) Project and the US Naval Submarine Base (project) site in Bangor, Washington, TNT and RDX are significantly degraded during composting. At the Umatilla site, concentrations of TNT and RDX were reduced from 1,574 ppm (TNT) and 944 ppm (RDX) to <30 mg/kg each after 20 days of treatment; and further reduced to 4 ppm (TNT) and 2 ppm (RDX), after 40 days of treatment (US Army Environmental Center, 1993, USEPA, 1994). Composting reduced the concentration of TNT in one kilogram of soil from 822 ppm to 8 ppm after 60 days of treatment at the Bangor site. Follow-up toxicological and leachability tests at the Umatilla site showed that a reduction in concentrations of TNT and RDX in treated soils provided a parallel reduction in toxicity and leachability of these contaminants (USEPA, 1996a).

An initial process goal of 30 mg/kg for TNT and RDX will be used during the Pilot Scale, as this level has been applied at previous sites to minimize the phytotoxicity effects of munitions compounds on plant species (USEPA, 1996a). This is especially important for areas which will be backfilled with treated soil and revegetated. The PRG established for soil cleanup at the Bangor facility was set at 30 mg/kg for TNT, as this level corresponded to "lifetime excess cancer risks between 10E-5 and 10E-6 under CERCLA and are protective for non-carcinogenic effects" (USEPA, 1993).

1.4.2.3 Performance Goals

The rate of degradation of TNT and RDX will be evaluated, based on the recipe used, and process goals for TNT and RDX will be finalized for full scale operation. Remedial goals and final process goals will be discussed, reviewed and approved by the Navy, IDEM and EPA prior to Full Scale Operations. These activities constitute the first performance goal.

The second performance goal will be to provide data to evaluate the biological treatment efficiency based on the CERCLA remediation criteria for innovative treatment technologies. The NCP preamble indicates 90% to 99% reduction of toxicity and mobility (leachability) as a guide for effective treatment. Previous studies have indicated that reductions in parent compounds and first series intermediate compounds were providing parallel reductions in toxicity and leachability based on combined chemical, toxicology and leachability data (USEPA, 1996b). Therefore, a 90% to 99% reduction in concentrations of contaminant compounds corresponds to a 90% to 99% reduction in toxicity and leachability and meets the NCP treatment performance guideline for composting of TNT, RDX and HMX. Applicability of this guideline will be finalized prior to Full Scale Operations.

1.4.3 Data Quality Objectives

Data Quality Objectives (DQOs) are qualitative and quantitative statements which specify the quality of the data required to support decisions made during interim measures cleanup, and are based on the end uses of the data to be collected. As such, different data uses may require different levels of data quality. There are at least five analytical levels which address various data uses and the QA/QC effort and methods required to achieve the desired level of quality. This project will utilize NEESA Level C for all off-site analytical work.

The intended data quality objectives for precision, accuracy and completeness for laboratory data are summarized in Tables 3-1 through 3-4.

1.5 SAMPLE LOCATIONS

Sample locations and rationale are fully described in Section 4.0. Locations will be reviewed by EPA Region 5 and IDEM, as applicable, prior to start of work.

1.5.1 Sample Network by Task and Matrix

Analytical constituents per task are summarized in Table 1-5. This is a summary of parameters relating to the Pilot Scale study. Sample totals are provided in Table 4-6, Section 4.0.

1.5.2 Excavation Site Maps of Sampling Locations

Sample location maps will be generated prior to mobilization at Mine Fill A. Preliminary sampling areas for Mine Fill A are provided in Figure 4-1. Depending on the nature of encountered field conditions some of these locations may be changed. Sampling locations will be finalized/approved by the Project Manager or designee, as overseen by the Navy, EPA and/or IDEM representatives.

TABLE 1-1 SUMMARY OF PREVIOUS SOIL BORING RESULTS - MINE FILL A

Sample I.D./Location	TNT (ppm)	HMX (ppm)	RDX (ppm)
MFA-1/45 feet away from northeast corner of box bin at Building 153	69	292	2,620
MFA-2/at base of berm 10 feet south of box bin	3,790	1,340	15,300
MFA-3/10 feet out from end of drain pipe	8	908	8,150
MFA-4/10 feet down ditch from MFA-3	<3.6	64	374
MFA-5/28 feet east of Building 158, in ditch	64	99	906
MFA-7/two feet southeast of static pole south of Building 158, in run-off ditch	<3.6	<5.0	<7.2
MFA-8/near base of berm, 15 feet west of corner of box bin	5.2	52	349

Notes:
1. Soil samples were collected from bores installed 20" below ground surface. Samples were analyzed for TNT, RDX and HMX. Reference for data and locations found on Figure 1-5 is *Final RCRA Facility Investigation Phase I, Environmental Monitoring Report, Solid Waste Management Units #19/00, #08/17, #12/14 and #13/14, Naval Surface Warfare Center, Crane Division, Crane, Indiana, Halliburton NUS (August 1992).*

TABLE 1-2. SUMMARY OF CHEMICALS PRESENT AT MINE FILL A	
Chemical	**Constituents/Composition**
TNT	2,4,6 Trinitrotoluene
RDX	Hexahydro-1,3,5-trinitro-1,3,5-triazine
HMX	Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine
Minol	TNT/Ammonium Nitrate/Aluminum
HBX	RDX/TNT/Aluminum/Wax/Calcium Chloride
H-6	RDX (45%), TNT (30%), Aluminum (20%), D-2 Wax (5%)
Tritanol	TNT (80%), Aluminum (20%)
Composition B	RDX (60%), TNT (40%), wax (1%)
Tetrytol	Tetryl/TNT
Tetryl	Trinitrophenylmethylnitramine

Notes:
1. Reference: *Final RCRA Facility Investigation Phase I, Environmental Monitoring Report, Solid Waste Management Units #19/00, #08/17, #12/14 and #13/14, Naval Surface Warfare Center, Crane Division, Crane, Indiana, Halliburton NUS (August 1992)*

TABLE 1-3 DATA COLLECTION ACTIVITIES - PILOT SCALE			
TASK	**DATA COLLECTED**	**DATA USE**	**DATA QUALITY LEVEL**
1. Initial Excavation Site Characterization (NOTE: One sample will be collected per location for VOCs, covering both lists)	-site-specific VOCs (methylene chloride, acetone, methyl ethyl ketone, methyl isobutyl ketone, toluene and xylene) -total VOCs (TCLP list) -explosives (Method 8330 list) -metals (barium, cadmium, chromium, lead) -TCLP metals (regulated list only)	-identify areas for Pilot Scale excavation -determine "startup" levels of explosives to be treated -determine if site-specific chemicals are present in soil to be treated -determine if soil is hazardous with respect to metals and specific VOCs	-NEESA Level C QA/QC
2. Equipment Wash Water/ Decontamination Water Sampling	-explosives (Method 8330 list) -POTW requirements, -**RCRA metals**	-results used for discharge to on-site treatment system -determine levels of explosives entering treatment system -**metals for informational (loading) purposes**	-NEESA Level C QA/QC
3. Storm Water Retention Pond Sampling	-NPDES parameters (see Table 4-8)	-determine if NPDES criteria are met for discharge to a permitted outfall	-NEESA Level C QA/QC

TABLE 1-3 DATA COLLECTION ACTIVITIES - PILOT SCALE			
TASK	**DATA COLLECTED**	**DATA USE**	**DATA QUALITY LEVEL**
4. Process Sampling (plus Confirmation Sampling - Day ~~28~~ **60**) - Compost Piles, amendments (TOC and TKN only)	-TOC, TKN -explosives (Method 8330 list) -metals (barium, cadmium, chromium, lead) -TCLP - metals (regulated list only) -pH -% moisture -oxygen	-measure the effectiveness and progress of the compost cycle (TOC, TKN, explosives, pH, %moisture, oxygen will be tested at day ~~0, 7, 14, 21, 28~~; **0, 5, 10, 20, 30, 40, and 60** metals will be tested at day 0 and ~~28~~ **60)** -measure the starting levels of nitrogen, carbon in amendment materials (to determine initial ratios) -determine if process goals have been met with respect to explosives -preliminary determination if treated compost can be placed in on-site landfill or used as backfill in the Mine Fill A excavations (lab results will be compared to preliminary remedial goals during the Pilot Scale)	-NEESA Level C QA/QC

TABLE 1-3 DATA COLLECTION ACTIVITIES - PILOT SCALE			
TASK	**DATA COLLECTED**	**DATA USE**	**DATA QUALITY LEVEL**
5. *Post-Excavation Sampling-* (confirmation sampling will be deferred to Full Scale Operations)	-*site-specific* VOCs (methylene chloride, acetone, methyl ethyl ketone, methyl isobutyl ketone, toluene and xylene) -explosives (Method 8330 list) -metals (barium, cadmium, chromium, lead)	-determine the levels of explosives, metals, and site-specific VOCs remaining in soil after Pilot Scale excavation is complete and prior to backfilling. **The purpose of post-excavation sampling is only informational. Until full scale operations begin, the excavation will be temporarily backfilled. The data will be used during Full Scale Operations to dictate or plan further excavation at Mine Fill A. Confirmation sampling for the purpose of "closing" an excavation will be completed during Full Scale Operations.**	-NEESA Level C QA/QC
Notes: 1. Methods or parameters listed under "data collected" are off-site analytical methods, unless otherwise noted.			

TABLE 1-4. INITIAL REMEDIAL GOALS AND ACCEPTABLE REPORTING LIMITS - Pilot Scale Study				
Parameter	**Method of Analysis**	**Soil goal-industrial limit (mg/kg) or TCLP regulatory limit**	**Soil Reporting Limit (mg/kg)**	**Water Reporting Limit (ug/L) (for Blank analyses)**
Explosives				
TNT	8330	30f	0.25	6.4
RDX	8330	30f	1	14.0
HMX	8330	34,000a	2.2	13.0

TABLE 1-4. INITIAL REMEDIAL GOALS AND ACCEPTABLE REPORTING LIMITS - Pilot Scale Study

Parameter	Method of Analysis	Soil goal-industrial limit (mg/kg) or TCLP regulatory limit	Soil Reporting Limit (mg/kg)	Water Reporting Limit (ug/L) (for Blank analyses)
Tetryl	8330	6,800a	0.65	10.0
TNB	8330	34b	0.25	7.3
1,3-DNB	8330	**68 a, f**	0.25	4.0
Nitrobenzene (NB)	8330	94a	0.26	7.0
4-Am-DNT	8330	2.8c	0.25	**20** ~~0.06 (low level)~~
2-Am-DNT	8330	2.8c	0.25	**14** ~~0.03 (low level)~~
2,4-DNT	8330	**2.8c**	0.25	5.7
2,6-DNT	8330	**2.8c**	0.26	9.4
2-nitrotoluene	8330	6,800d	0.25	12.0
3-nitrotoluene	8330	6,800d	0.25	7.9
4-nitrotoluene	8330	6,800d	0.25	8.0
Total Metals (site-specific)				
Barium	6010	100,000a	0.5	5
Cadmium	6010	850a	0.4	4
Chromium (total)	6010	450a	0.5	5
Lead	6010	1,000a	4 (0.3 - trace ICP)	40
TCLP-metals (regulatory limit units: mg/L)				

TABLE 1-4. INITIAL REMEDIAL GOALS AND ACCEPTABLE REPORTING LIMITS - *Pilot Scale Study*

Parameter	Method of Analysis	Soil goal-industrial limit (mg/kg) or TCLP regulatory limit	Soil Reporting Limit (mg/kg)	Water Reporting Limit (ug/L) (for Blank analyses)
Arsenic	1311, 6010	5.0 mg/L	N/A	100 ug/L
Barium	1311, 6010	100	N/A	5
Cadmium	1311, 6010	1.0	N/A	4
Chromium	1311, 6010	5.0	N/A	5
Lead	1311, 6010	5.0	N/A	40
Mercury	1311, 7470	0.2	N/A	1.5
Selenium	1311, 6010	1.0	N/A	100
Silver	1311, 6010	5.0	N/A	5
Volatile Organics (the following compounds were listed in Table 5-7 of the August 1992 EMR for Mine Fill A)				
Dichloro-methane (methylene chloride)	8240	18a	0.005	5
Acetone	8240	8,600a	0.01	10
Methyl ethyl ketone	8240	27,000a	0.01	10

TABLE 1-4. INITIAL REMEDIAL GOALS AND ACCEPTABLE REPORTING LIMITS - Pilot Scale Study

Parameter	Method of Analysis	Soil goal-industrial limit (mg/kg) or TCLP regulatory limit	Soil Reporting Limit (mg/kg)	Water Reporting Limit (ug/L) (for Blank analyses)
Methyl isobutyl ketone	8240	2,800a	0.01	10
Toluene	8240	880a	0.005	5
Xylene (total)	8240	320a	0.005	5
Total VOCs (TCLP regulated list)				
Benzene	8240	1.4a	0.005 mg/kg	5 ug/L
Carbon Tetrachloride	8240	0.5a	0.005	5
Chlorobenzene	8240	220a	0.005	5
Chloroform	8240	0.53a	0.005	5
1,2-Dichloroethane	8240	0.55a	0.005	5
1,1-Dichloroethene	8240	0.08a	0.005	5
Methyl Ethyl Ketone	8240	27,000a	0.01	10
Tetrachloro-ethylene	8240	170a	0.005	5
Tetrachloro-ethene	8240	7a	0.005	5
Vinyl Chloride	8240	0.035a	0.01	10

TABLE 1-4. INITIAL REMEDIAL GOALS AND ACCEPTABLE REPORTING LIMITS - Pilot Scale Study

Parameter	Method of Analysis	Soil goal-industrial limit (mg/kg) or TCLP regulatory limit	Soil Reporting Limit (mg/kg)	Water Reporting Limit (ug/L) (for Blank analyses)

Notes:

1. Analytical methods listed above are SW-846 methods, unless otherwise indicated.
2. All other parameters not listed here will be reported at standard PQLs (i.e. TOC, TKN)
3. Parameter lists required to be reported per method per data collection task are provided in Table 1-5.
4. N/V = no value available
5. N/A = not applicable

a. Reference: EPA Region 9 Preliminary Remediation Goals, August 1, 1996 **(Note that if high levels of total chromium are detected, hexavalent chromium samples may be collected. Analysis will not be permitted until after U.S. EPA's approval of an SOP for hexavalent chromium analysis.)**

b. Value for 1,3,5-Trinitrobenzene

c. Value for dinitrotoluene mixture (see reference a).

d. Value for 2-and 3-nitrotoluene.

e. These reporting limits will be confirmed with the laboratory.

f. Target parameter presumes collection of 3 isomers and applies most conservative available industrial PRG value to non resolved isomers.

TABLE 1-5 SUMMARY OF SAMPLING PARAMETERS PER TASK		
Task	**Analytical Method Requested**	**Parameters to be Reported**
1. Initial excavation site Characterization -identify areas for Pilot Scale excavation -determine "startup" levels of explosives -determine if additional site-specific (potential) contaminants are present -determine if soil is hazardous with respect to metals and VOCs	-Site-specific VOCs (Method 8240) -total VOCs (TCLP regulated list using Method 8240) (NOTE: one sample will be collected for VOCs analysis, covering both lists) -Explosives (Method 8330) -Site-specific metals (6010) -TCLP-metals (1311, 6010, 7470)	-8240: methylene chloride, acetone, xylene, toluene, methyl ethyl ketone, methyl isobutyl ketone -benzene, carbon tetrachloride, chloroform, chlorobenzene, 1,2-dichloroethane, 1,1-dichloroethene, methyl ethyl ketone, tetrachloroethene, trichloroethene, vinyl chloride -full list for explosives -6010: barium, cadmium, total chromium, lead -regulated list only
2. Equipment Wash Water/ Decontamination Water Sampling	-explosives (Method 8330) -POTW requirements -**RCRA metals**	-full Method 8330 list -dependent on POTW -**Methods 6010 and 7420**
3. Storm Water Retention Pond Sampling	-TSS (EPA 160.2) -COD (EPA 410.4) -BOD (EPA 405.1) -Oil and Grease (EPA 413.1) -Nitrate (EPA 300.0) -Ammonia-N (EPA 350.2)	list as shown
4. Process Sampling and Confirmation Sampling (Day ~~28~~ **60**); amendments will be sampled once for TOC and TKN only	-Explosives (Method 8330) -TOC (EPA 9060) -TKN (EPA 351.3) -metals (6010) - day 0 and ~~28~~ **60** only -TCLP-metals(1311, 6010, 7470) - day 0 and ~~28~~ **60** only	-full list for explosives -TOC, TKN -7470/7471: mercury -6010: barium, cadmium, total chromium, lead -regulated list only (see Pilot Scale Operational Plan for additional field parameters)

TABLE 1-5 SUMMARY OF SAMPLING PARAMETERS PER TASK

Task	Analytical Method Requested	Parameters to be Reported
5. Sampling- Excavations	-Site-specific VOCs -Explosives (Method 8330) -Site-specific metals (6010)	-8240: methylene chloride, acetone, xylene, toluene, methyl ethyl ketone, methyl isobutyl ketone -full list for explosives -6010: barium, cadmium, total chromium, lead

Notes:
1. If soil requires off-site treatment, samples will be collected during Full Scale Operations.

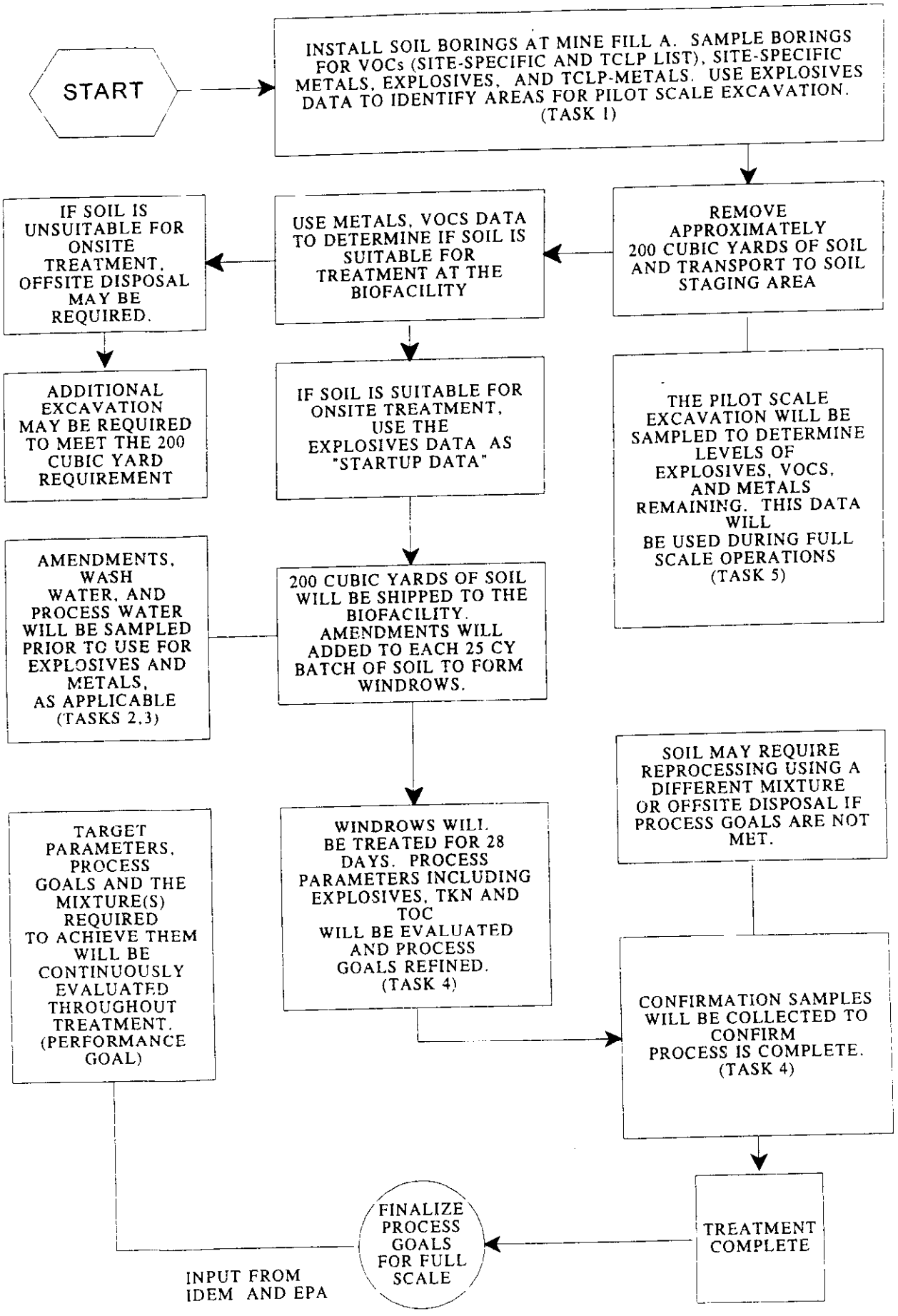


FIGURE 1-1. SAMPLING AND ANALYSIS LOGIC DIAGRAM

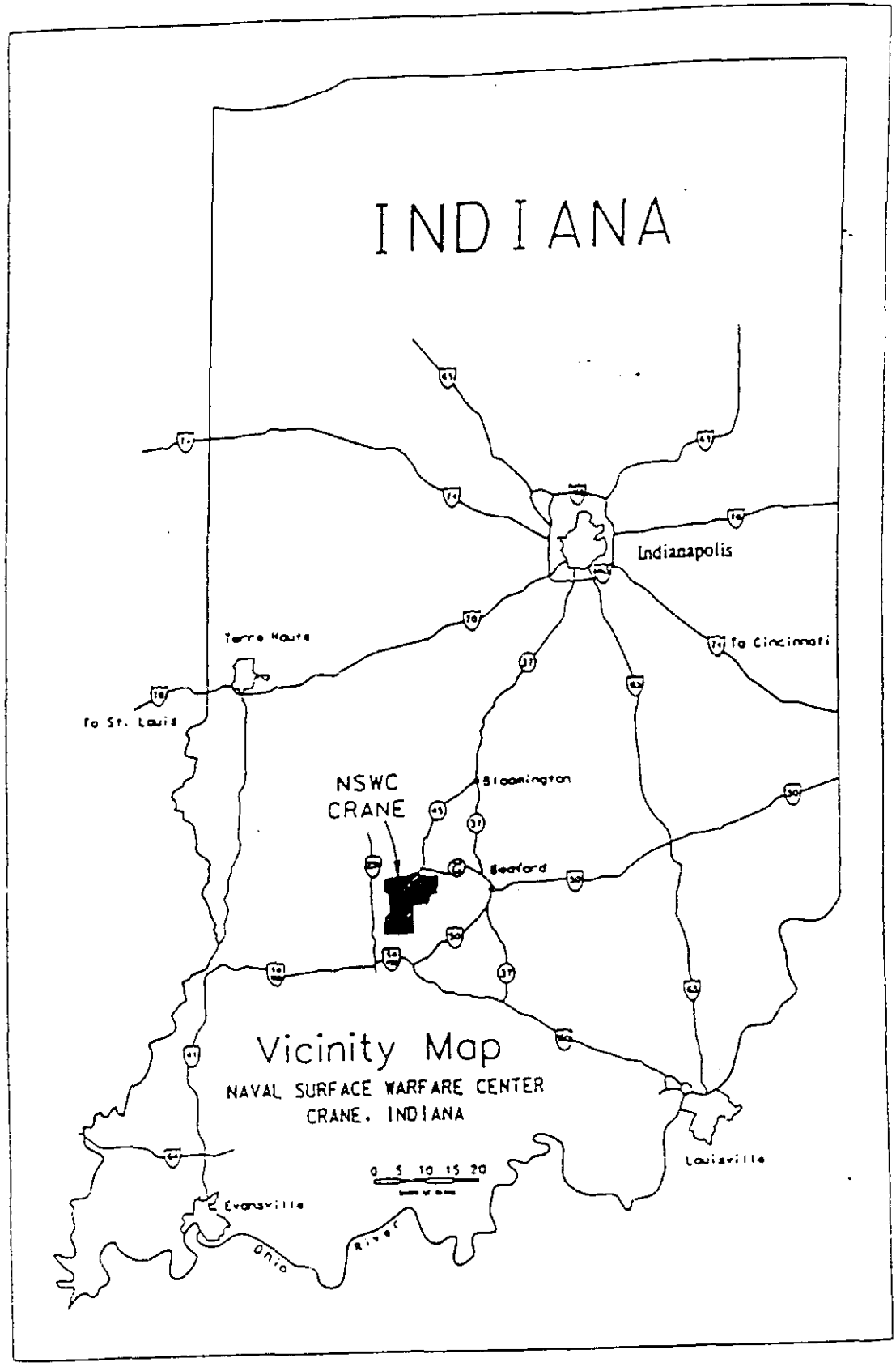
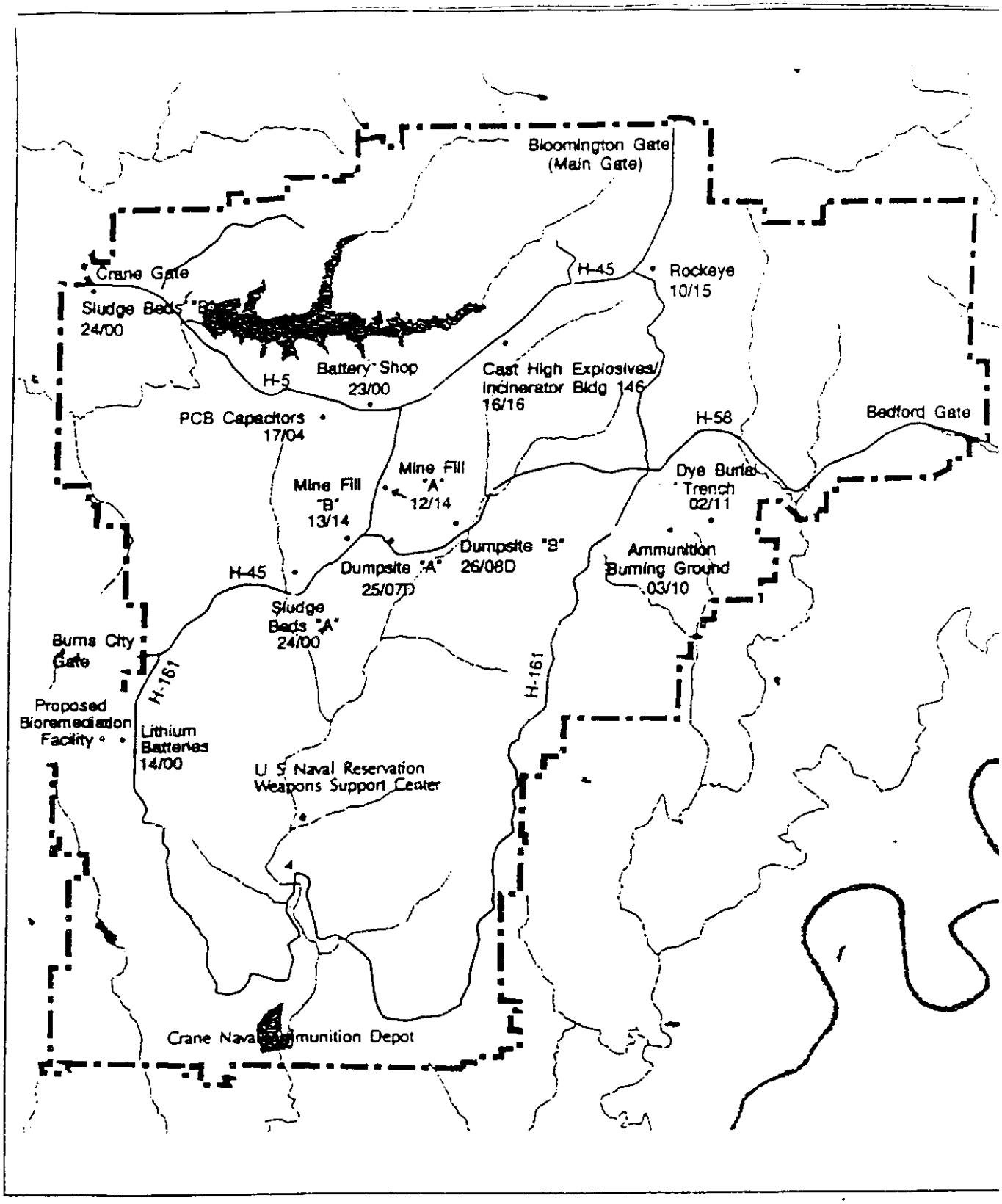
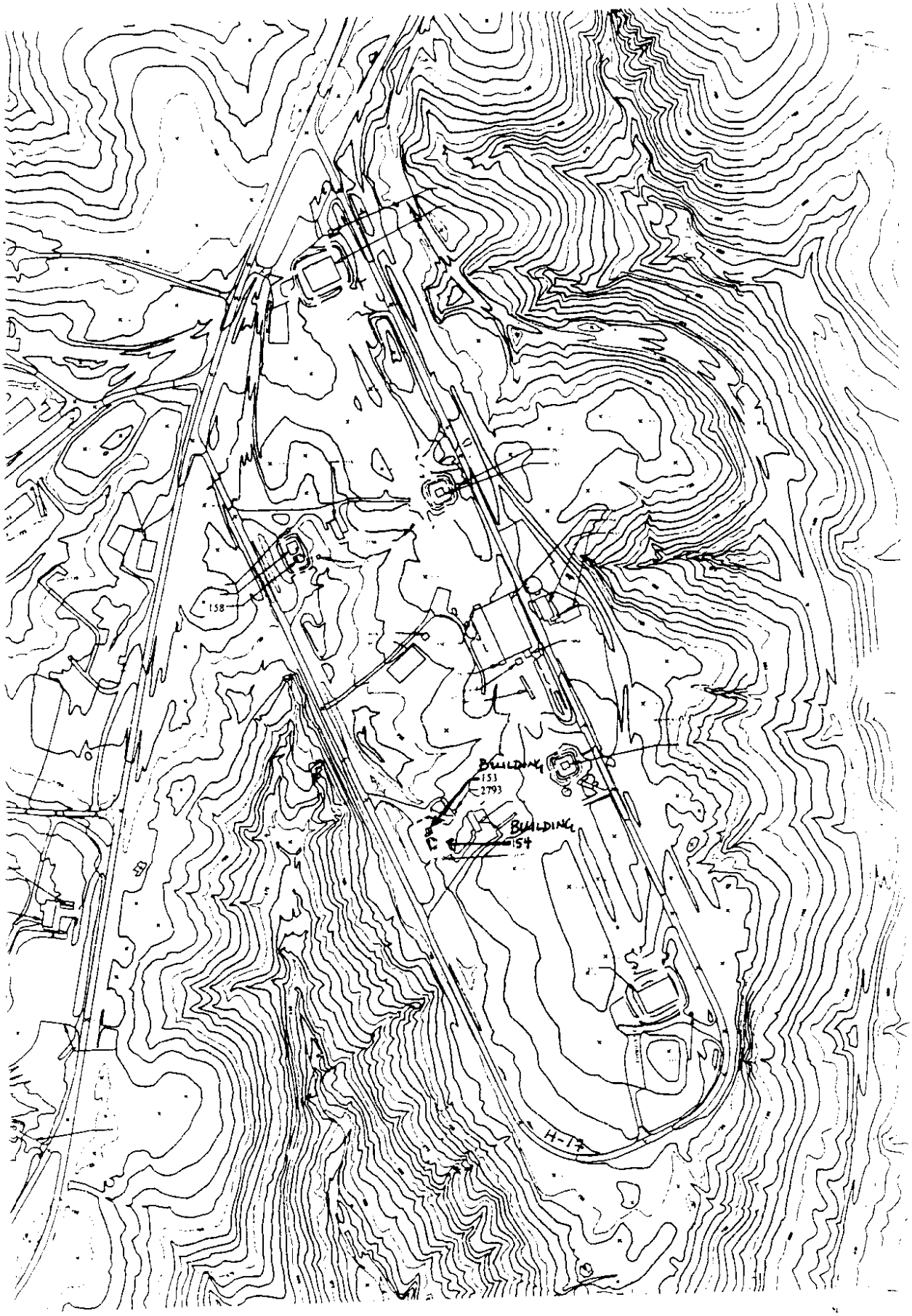


Figure 1-2
Vicinity Map of NSWC Crane, Indiana



02/11 Dye Burial Ground
03/10 Ammunition Burning Ground Area
10/15 Rockeye
12/14 Mine Fill A
13/14 Mine Fill B
14/00 Sanitary Landfill and Lithium Batterey
16/16 Cast High Explosives Fill/Incineration complex
17/04 PCB Capacitor Burial/Pole Yard
23/00 Battery Shop
24/00 Sludge Drying Bed A
24/00 Sludge Drying Bed B

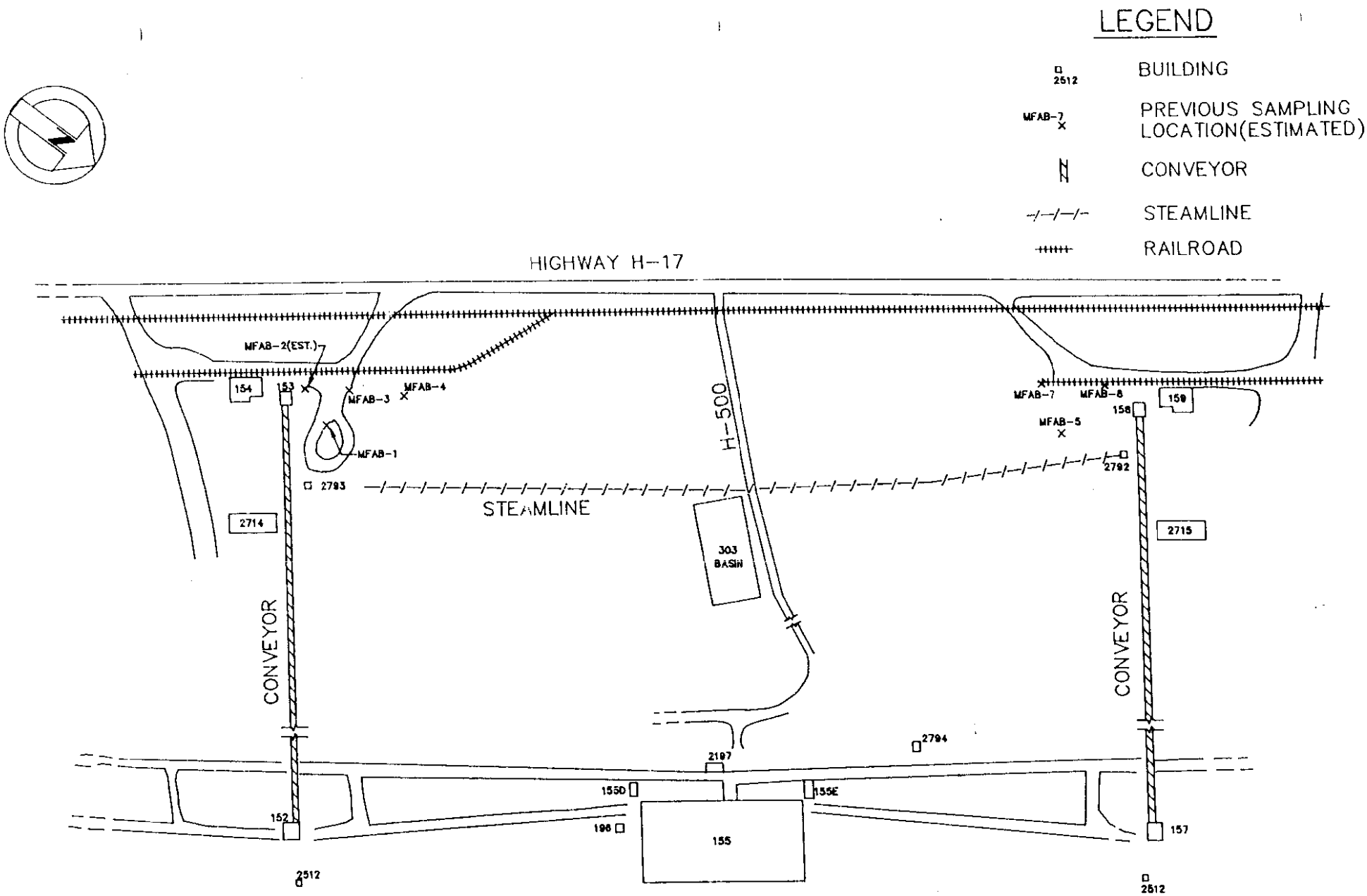
FIGURE 1-3
Location of Solid Waste Management Units



300 0 300 600 Feet

FIGURE 1-4

MINE FILL A



LAYOUT OF CENTRAL MINE FILL A

(NOT TO SCALE)

MORRISON KNUDSEN CORP

Drawing Figure 1-5 Rev.

SECTION 2
PROJECT ORGANIZATION AND RESPONSIBILITY

2.1 PROJECT ORGANIZATION CHART

Figure 2-1 of this plan shows the project organization chart indicating individual assignments for the remediation activities at NSWC Crane. All participants are directly subject to the requirements of this QAPP. The responsibilities of individual positions are briefly described in the following sections.

2.2 MANAGEMENT RESPONSIBILITIES

U.S. EPA RCRA Project Coordinator/State Project Manager

The RCRA Project Coordinator (RPC) has the overall responsibility for all EPA-related aspects of the RCRA interim measures cleanup, including approval of project objectives. The State Project Manager has overall responsibility for all State-related phases of the RCRA Interim Measures Cleanup with oversight by the U.S. EPA RPC.

Southern Division Project Manager

The Southern Division Project Manager is responsible for implementing the project, and has the authority to commit the resources necessary to meet project objectives and requirements. The Southern Division Project Manager's primary function is to ensure that technical, financial, and scheduling objectives are achieved successfully. The Southern Division Project Manager will coordinate directly with the U.S. EPA Region 5 RPC and will provide the major point of contact and control for matters concerning the project. The Southern Division Project Manager will:

- Define project objectives and develop a detailed work plan schedule;
- Establish project policy and procedures to address the specific needs of the project as a whole, as well as the objectives of each task;

- Acquire and apply technical and corporate resources as needed to ensure performance within budget and schedule constraints;
- Orient all field leaders and support staff concerning the project's special considerations;
- Monitor and direct the field leaders;
- Develop and meet ongoing project and/or task staffing requirements, including mechanisms to review and evaluate each task product;
- Review the work performed on each task to ensure its quality, responsiveness, and timeliness;
- Review and analyze overall task performance with respect to planned requirements and authorizations;
- Approve all reports (deliverables) before their submission to U.S. EPA Region 5;
- Ultimately be responsible for the preparation and quality of interim and final reports; and
- Represent the project team at meetings and public hearings.

MK Program Management Office

The Program Management Office (PMO) in Charleston, South Carolina has overall responsibility for all cleanup measure activities at all sites in the Southern Division of the Naval Facilities Engineering Command under Contract No. N62467-93-D-1106. The PMO is the primary point of contact for Southern Division.

2.3 QUALITY ASSURANCE RESPONSIBILITIES

MK Program QA Manager

The MK Program QA Manager will remain independent of direct job involvement and day-to-day operations, and have direct access to corporate executive staff as necessary, to resolve any QA dispute. The Program QA Manager is responsible for auditing the implementation of the QA program in conformance with the demands of specific investigations, MK's policies, and US EPA requirements in accordance with this QAPP. Specific functions and duties include:

- Providing QA audit on various phases of the field operations;
- Reviewing and approving of QA plans and procedures;
- Providing QA technical assistance to project staff; ~~and~~
- Reporting on the adequacy, status, and effectiveness of the QA program on a regular basis to the program manager and executive vice president for technical operations**; and**
- Reviewing analytical laboratory qualifications.

MK Site Quality Control Supervisor

The MK Site Quality Control Supervisor (SQCS) will provide the necessary guidance to the project staff on quality-related matters. The SQCS will provide quality assurance reviews of project documents, field procedures, laboratory quality assurance plans, project documentation and will perform project audits. Duties of the SQCS include identifying quality problems, initiating corrective actions and verifying the implementation of the corrective actions.

U.S. EPA Region 5 Quality Assurance Program Plan (QAPP) Coordinator

EPA Region 5 QAPP Coordinator has the responsibility to review and approve all Quality Assurance Project Plans (QAPPs). Additional U.S. EPA responsibilities for the project include.

- Conducting external Performance and System Audits of the off-site analytical Laboratory; and

- Reviewing and evaluating field and laboratory procedures.

2.4 LABORATORY RESPONSIBILITIES

MK Project Chemist

The MK Project Chemist will report to the MK Project Manager and will be responsible for:

- Review of analytical laboratory qualifications;

- Coordinating laboratory analyses requirements with field personnel;

- Review of preliminary analytical data;

- Maintenance of final evidence files;

- Review of analytical reports;

- Approving final analytical reports prior to submission to Southern Division; and

- Overseeing data verification and data validation procedures.

Laboratory Project Manager

The Laboratory Project Manager will report directly to the MK Project Chemist and will be responsible for the following:

- Providing technical support for analytical method procedures and discrepancies;

- Ensuring all resources of the laboratory are available on an as-required basis; and

- Reviewing final analytical reports.

Laboratory Operations Manager

The Laboratory Operations Manager will report to the Laboratory Project Manager and will be responsible for:

- Coordinating laboratory analyses;
- Supervising in-house chain-of-custody;
- Scheduling sample analyses;
- Overseeing data review, **verification & validation;**
- Overseeing preparation of analytical reports; and
- Approving final analytical reports prior to submission to MK.

Laboratory Quality Assurance Officer

The Laboratory QA Officer has the overall responsibility for data after it leaves the laboratory. The Laboratory QA Officer will be independent of the project staff but will communicate data issues through the Laboratory Project Manager. In addition, the Laboratory QA Officer will:

- Oversee laboratory quality assurance;
- Oversee QA/QC documentation;
- Conduct detailed review and verification of analytical data in reports prior to submission to MK;
- Determine whether to implement laboratory corrective actions, if required;
- Define appropriate laboratory QA procedures; and
- Prepare laboratory Standard Operation Procedures.

Laboratory Sample Custodian

The Laboratory Sample Custodian will report to the Laboratory Operations Manager. Responsibilities of the Laboratory Sample Custodian will include:

- Receiving and inspecting the incoming sample containers;
- Recording the condition of the incoming sample containers;
- Signing appropriate documents;
- Verifying chain-of-custody and its correctness;
- Notifying Laboratory Manager and Laboratory Supervisor of sample receipt and inspection;
- Assigning a unique laboratory identification number and customer number, and entering each into the sample receiving log;
- With the help of the laboratory manager, initiating transfer of the samples to appropriate lab sections; and
- Controlling and monitoring access/storage of samples and extracts.

Final responsibility for project quality rests with MK's Project Manager. Independent quality assurance will be provided by the Laboratory Project Manager and Laboratory QA Officer prior to release of all data to MK.

Laboratory Technical Staff

The Laboratory technical staff will be responsible for sample analysis and identification of corrective actions. The staff will report directly to the Laboratory Operations Manager.

2.5 FIELD RESPONSIBILITIES

Site Project Manager

The MK Project Manager (MK PM) will have overall management responsibilities for technical, financial and scheduling matters. The PM will report to the designated Southern Division Project Manager for all project related issues and to the MK PMO for project oversight, management direction, and resolution of any company related matters. The MK PM will follow the project developments and notify Southern Division of any problems encountered in the field which may effect data quality or the project schedule.

The MK PM has overall responsibility for ensuring that the project meets U.S. EPA's objectives and MK QA/QC standards. The MK PM will provide assistance to Southern Division in terms of distributing the QAPP to all those parties connected with the project. The MK PM will report directly to Southern Division and is responsible for technical quality control and project oversight.

Site QC Supervisor

The primary role of the Site QC Supervisor will be to assist in supervising the day-to-day performance of field activities of the field technical staff (i.e. QC Inspector) and specific subcontractors (i.e. geotechnical laboratory and surveyor). The Site QC Supervisor will ensure that the data quality objectives established for the project are met through implementation of field procedures and project schedule. The Site QC Supervisor will also be responsible to the PM for coordination with all support service groups including procurement, project controls and quality control.

Site Safety and Health Officer

The Site Safety and Health Officer (SSHO) is responsible for the implementation of the Site Safety and Health Plan and the day-to-day control of health and safety activities. This individual will provide the necessary guidance to the project staff so they can safely perform their functions in accordance with corporate, state, and federal requirements. The SSHO will report directly to the PM and the MK PMO Safety and Health Manager.

Contract Administration

The Contract Administrator has primary responsibility to ensure contract requirements are met, and to document and implement change orders to the contract. The Contract Administrator will work with the Superintendent and will report directly to the Project Manager.

Field Engineer

The Field Engineer will be responsible for gathering and analyzing field data, and preparing daily reports and data evaluations. The Field Engineer will work closely with operation personnel both at Mine Fill A and the Biofacility to ensure tasks are completed with care and objectives are met. The Field Engineer will report to the Superintendent and ultimately to the Project Manager.

Superintendent

The Superintendent is primarily responsible for the coordination and control of all field activities to ensure that all tasks in the Work Plan and Operational Plan are completed. The Superintendent will also coordinate off-site waste disposal activities and provide daily reports to the Project Manager on the status of field work.

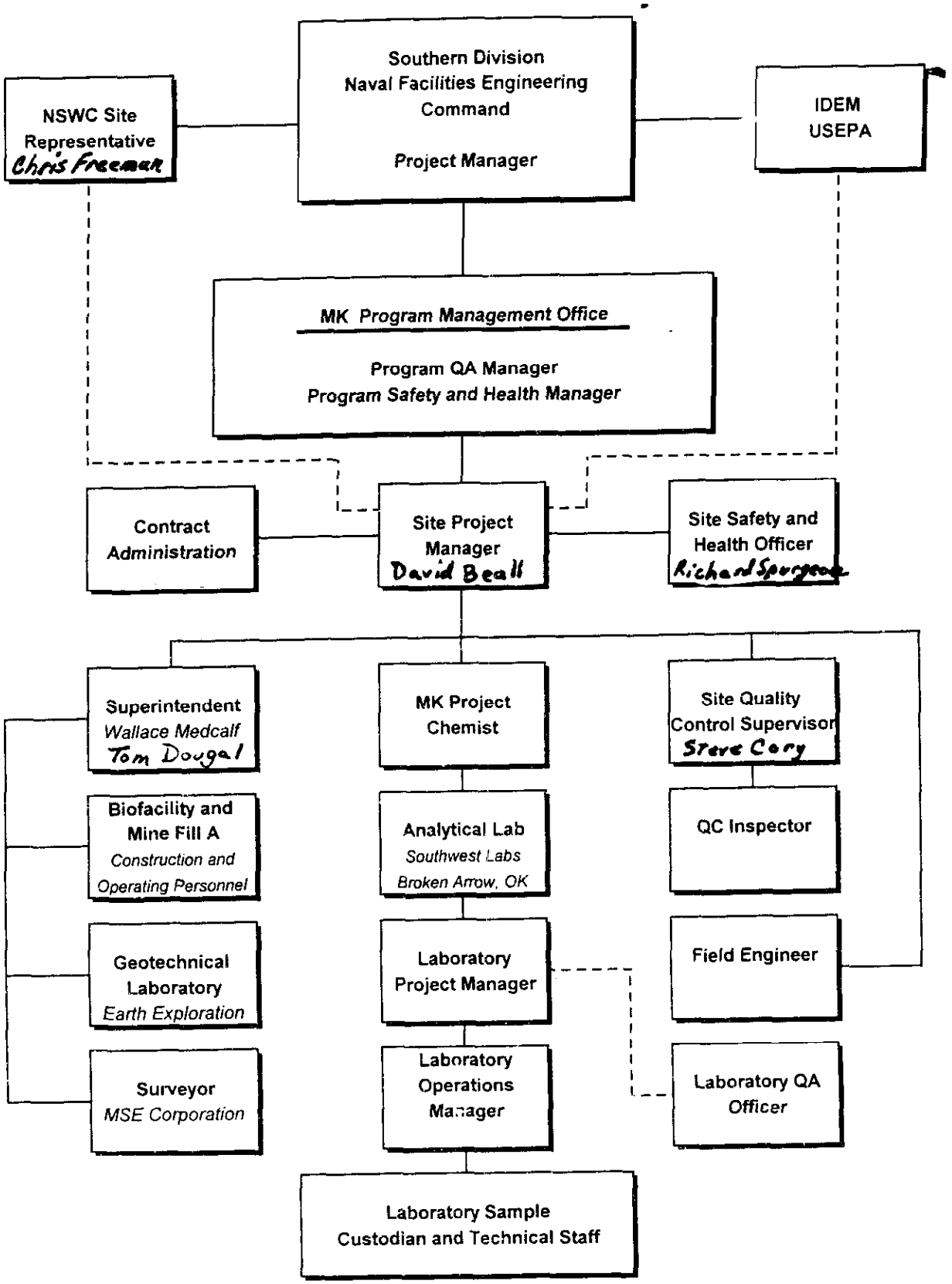


FIGURE 2-1. ORGANIZATION CHART

SECTION 3
QUALITY ASSURANCE OBJECTIVES FOR MEASUREMENT DATA

The overall QA objective for this project is to develop and implement procedures for field sampling, chain-of-custody, laboratory analysis, and reporting that will provide results which are legally defensible in a court of law. Specific procedures for sampling, chain-of-custody, laboratory instrument calibration, laboratory analysis, reporting of data, internal quality control, audits, preventive maintenance of field equipment, and corrective action are described or referenced in other sections of this QAPP. Laboratory reporting and/or detection limits for each method in Table 1-3 are provided in Appendix B. General quality control limits for accuracy, precision and completeness are provided in Tables 3-1 through 3-4.

3.1 PRECISION

3.1.1 Definition

Precision is a measure of the reproducibility of measurements under a given set of conditions. Precision is usually stated in terms of relative standard deviation (RSD) or relative percent difference (RPD). Results of QC samples can be used to calculate the precision of the analytical or sampling process. Field duplicate and replicate samples will be used to determine the precision of the sampling process. Laboratory duplicates, matrix spikes and matrix spike duplicates will be used to determine the precision of the analytical process.

3.1.2 Field Precision Objectives

Field duplicate data will be evaluated to determine potential variability introduced by soil heterogeneity and sampling technique. This evaluation will be performed during data verification and summarized in the data assessment. One duplicate sample will be analyzed for every 10 samples to qualitatively evaluate precision.

3.1.3 Laboratory Precision Objectives

Precision in the laboratory is assessed through the calculation of RPD and RSD for three or more replicate samples. The equations to be used for precision in this project can be found in Section 12. Precision control limits are established by SW-846 methods. Limits for confirmation soil sample analyses are summarized in Tables 3-1 through 3-4.

3.2 ACCURACY

3.2.1 Definition

Accuracy is a measure of the bias in a measurement system, and defined as the closeness of the reported value to the true value. Potential sources of error include the sampling process, field contamination, preservation, handling, sample matrix, sample preparation and analytical techniques. The accuracy of a measurement system will be assessed by evaluating the results of quality control samples such as matrix spikes, analytical surrogates and the use of field blanks, trip blanks and equipment rinsate blanks.

3.2.2 Field Accuracy Objectives

For laboratory data evaluation, field blanks, trip blanks and equipment rinsate blank data will be evaluated to identify potential sources of error introduced during sampling. This evaluation will be performed during data validation and summarized in the data assessment. Accuracy is also assessed through adherence to all sample handling, preservation and holding times.

3.2.3 Laboratory Accuracy Objectives

Laboratory accuracy is assessed through the analysis of standard matrix spikes, sample matrix spikes (MS) or standard reference materials (SRM) and the determination of percent recoveries. The equation to be used for accuracy in this project can be found in Section 12. The accuracy control limits are also summarized in Tables 3-1 through 3-4, and may vary based on laboratory performance.

3.3 COMPLETENESS

3.3.1 Definition

Completeness is a measure of the amount of valid data obtained from an analytical data set compared to the amount that was expected to be obtained under normal sampling and analytical conditions.

3.3.2 Laboratory Completeness Objectives

Laboratory completeness is a measure of the amount of valid measurements obtained from all measurements taken in the project. The equation for completeness is presented in Section 12. The laboratory completeness goal for Level C data is 90 percent, which is considered to be attainable using SW-846 and other EPA-approved analytical methods. This completeness objective was used as part of Phase II contaminant soil remediation at the Umatilla site (Final RAMP, October 4, 1994).

3.4 REPRESENTATIVENESS

3.4.1 Definition

Representativeness expresses the degree to which data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition.

3.4.2 Measures to Ensure Representativeness of Field Data

Representativeness is a qualitative parameter which is controlled by the proper design of the sampling program. Representativeness is addressed by describing sampling techniques and the rationale used to select sampling locations. It will be assessed through the use of samples collected in a manner such that they are equally representative of a given point in space or time. Representativeness will be satisfied by ensuring that this QAPP is followed and that proper sampling techniques and sample preservation procedures are followed.

3.4.3 Measures to Ensure Representativeness of Laboratory Data

Representativeness in the laboratory is ensured by using the proper analytical procedures, meeting sample holding times and analyzing and assessing field duplicate samples. The Pilot Scale sampling network was designed to provide data representative of site conditions. During development of this network, consideration was given to past waste disposal practices, existing analytical data, physical setting and processes, and constraints inherent to the RCRA program.

3.5 COMPARABILITY

3.5.1 Definition

Comparability is a qualitative parameter expressing the confidence with which one data set can be compared with another.

3.5.2 Measures to Ensure Comparability of Field Data

The comparability goal is achieved by using standard techniques to collect and analyze representative samples and reporting analytical data in appropriate units. Comparability will be satisfied by ensuring that the QAPP procedures are followed and that proper sampling techniques are used.

3.5.3 Measures to Ensure Comparability of Laboratory Data

Planned analytical data will be comparable when similar sampling and analytical methods are used and documented per the QAPP. Comparability is also dependent on similar QA objectives.

3.6 LEVEL OF QUALITY CONTROL EFFORT

Field blank, trip blank, method blank, sample duplicate, standard reference materials (SRM), standards matrix spike and sample matrix spike samples will be analyzed to assess the quality of the data resulting from the field sampling and analytical programs.

Field and trip blanks consisting of organic-free, ultra-pure water will be submitted to the analytical laboratories to provide the means to assess the quality of the data resulting from the field sampling program. Trip blanks are used to assess the potential for contamination of samples due to contaminant migration during sample shipment and storage. Sampling equipment rinsates are collected to ensure sampling equipment is clean and that the potential for cross-contamination has been minimized.

Two types of field blanks will be collected, "A" and "B." Field Blank A samples will be collected from each source of tap water used for equipment decontamination for each event. Only one sample per tap water source per event will be collected. Field blank B samples will be collected by pouring organic-free, ultra-pure water into sample containers at the sample collection site. These samples will be analyzed to check for procedural contamination at the facility which may cause sample contamination. The

frequency of Field Blank B will be 1 per 20 regular samples collected. This frequency may be reduced, depending on evaluation of field blank data.

Method blank samples are generated within the laboratory and used to assess contamination resulting from laboratory procedures. Duplicate samples are analyzed to check for sampling and analytical reproducibility. Matrix spikes provide information about the effect of the sample matrix on the extraction or digestion and measurement methodology. All matrix spikes are performed in duplicate for organic methods and are hereinafter referred to as matrix spike/ matrix spike duplicate (MS/MSD) samples. One MS/MSD will be collected for every 20 or fewer investigative samples. MS/MSD samples are designated/collected for all analytical methods.

MS/MSD samples are investigative samples. Soil MS/MSD samples require no extra volume for VOCs or extractable organics. However, aqueous MS/MSD samples must be collected at triple the volume for VOCs and double the volume for extractable organics. One MS/MSD sample will be collected/designated for every 20 or fewer investigative samples per sample matrix (*i.e.*, ground water, soil).

Field duplicates will be collected at a frequency of one sample per 10 regular samples collected, depending on the data use and as dictated in Table 1-3. The general level of the QC effort will be one field duplicate for every 10 or fewer investigative samples **per sampling interval for compost sampling** One volatile organic analysis (VOA) trip blank, consisting of distilled deionized ultra pure water, will be included along with each shipment of aqueous VOA samples.

Compost must be regarded as an unusual matrix. The laboratory is prepared to conduct serial dilutions and post digest spikes, and MSA as necessary if interference problems are noted in accordance with SW-846, Method 7000A, section 8.0 and method 6010A, section 8.5.

TABLE 3-1
QA OBJECTIVES FOR
PRECISION, ACCURACY AND COMPLETENESS
VOLATILE ORGANIC COMPOUNDS (VOCs) - Method 8240/SOP MS300

Data Quality Parameter	Matrix	Method of Determination	Frequency	QC Limits
Precision	Water	Laboratory MS/MSD	One per 20 samples (one per analytical batch)	RPD = 0-30
	Water	Laboratory Control Sample/Duplicate	One per 20 samples (one per analytical batch)	RPD = 0-30
	Soil	Laboratory MS/MSD	One per 20 samples (one per analytical batch)	RPD = 0-40
	Soil	Laboratory Control Sample/Duplicate	One per 20 samples (one per analytical batch)	RPD = 0-40
Accuracy	Water	Surrogate	All samples and standards	See Appendix F, Page 43 (Table 4)
	Water	Laboratory Control Sample/Duplicate	One per 20 samples (one per analytical batch)	See Appendix F, Page 44 (Table 6)
	Water	Laboratory MS/MSD	One per 20 samples (one per analytical batch)	See Appendix F, Page 43 (Table 5)
	Soil	Surrogate	All samples and standards	See Appendix F, Page 43 (Table 4)
	Soil	Laboratory Control Sample/Duplicate	One per 20 samples (one per analytical batch)	See Appendix F, Page 44 (Table 6)
	Soil	Laboratory MS/MSD	One per 20 samples (one per analytical batch)	See Appendix F, Page 43 (Table 5)
Completeness	Water, Soil	Valid Samples	All Samples	90 percent

Notes: MS/MSD = matrix spike/ matrix spike duplicate; RPD = relative percent difference

TABLE 3-2
QA OBJECTIVES FOR
PRECISION, ACCURACY AND COMPLETENESS
EXPLOSIVES (Method 8330)

Data Quality Parameter	Matrix	Method of Determination	Frequency	QC Limits
Precision	Water	Laboratory MS/MSD	One per 20 samples (one per analytical batch)	See Appendix F, Page 74 (Table 6)
	Water	Laboratory Control Sample/Duplicate	One per 20 samples (one per analytical batch)	RPD = 0 - 30
	Soil	Laboratory MS/MSD	One per 20 samples (one per analytical batch)	See Appendix F, Page 74 (Table 6)
	Soil	Laboratory Control Sample/Duplicate	One per 20 samples (one per analytical batch)	RPD = 0-40
Accuracy	Water	Surrogate	All samples and standards	60-140% using 3,4-DNT
	Water	Laboratory Control Sample/Duplicate	One per 20 samples (one per analytical batch)	See Appendix F, Page 74 (Table 5)
	Water	Laboratory MS/MSD	One per 20 samples (one per analytical batch)	See Appendix F, Page 74 (Table 6)
	Soil	Surrogate	All samples and standards	60-140% using 3,4-DNT
	Soil	Laboratory Control Sample/Duplicate	One per 20 samples (one per analytical batch)	See Appendix F, Page 74 (Table 5)
	Soil	Laboratory MS/MSD	One per 20 samples (one per analytical batch)	See Appendix F, Page 74 (Table 6)
Completeness	Water, Soil	Valid Samples	All Samples	90 percent

Notes: MS/MSD = matrix spike/ matrix spike duplicate; RPD = relative percent difference

TABLE 3-3
QA OBJECTIVES FOR
PRECISION, ACCURACY AND COMPLETENESS
Metals: Arsenic, Barium, Cadmium, Chromium, Lead, Mercury, Selenium, Silver

Data Quality Parameter	Matrix	Method of Determination	Frequency	QC Limits
Precision	Water, Soil	Laboratory Duplicate	One per 20 samples (one per analytical batch)	RPD = 0-20
Accuracy	Water, Soil	Laboratory MS/MSD	One per 20 samples (one per analytical batch)	**Recovery = 80-120**
	Water	Laboratory Control Sample/Duplicate	One per 20 samples (one per analytical batch)	Recovery = 80-120
	Soil	Laboratory Control Sample/Duplicate	One per 20 samples (one per analytical batch)	Recovery = 80-120
Completeness	Water, Soil	Valid Samples	All Samples	90 percent

Notes:
MS/MSD = matrix spike/ matrix spike duplicate; RPD = relative percent difference

TABLE 3-4
QA OBJECTIVES FOR
PRECISION, ACCURACY AND COMPLETENESS
Miscellaneous Parameters (All SOPs are provided in Appendix F)

Parameter	SOP	Section (in SOP)	Appendix Page	Section Page
TOC - soil	IN045S	8	F-118	3
TOC - water	IN045W	9	F-127	6
TKN	IN170	8	F-132	2
TSS	IN275	8	F-232	3
COD	IN090D, IN090C	10, 10	F-238, F-243	4,4
BOD	IN025	8	F-247	3
Oil and Grease	IN200, IN205	9	F-254	5
Nitrate	IN030B	10	F-263	5
Ammonia-N	IN020	9	F-269	4

SECTION 4
SAMPLING PROCEDURES

Sampling procedures will be used to collect data to meet the objectives of five data collection tasks:

- Initial Excavation Site Characterization (Section 4.1)
- Equipment Wash Water/Decontamination Water Sampling (Section 4.2)
- Storm Water Retention Pond Sampling (Section 4.3)
- Process Sampling (Section 4.4)
- Excavation Sampling (Section 4.5)

These tasks and associated objectives are described in Section 1.4.

QA/QC samples are described in Section 4.6. Decontamination procedures are provided in Section 4.7. Sample packaging and shipping information is provided in Section 4.8. A field equipment list and field test kit data sheets are provided in Appendix A.

Each of the sampling tasks listed above will be completed during the Pilot Scale, as applicable. Mine Fill A soils will be excavated and treated. It is anticipated that only 200 cubic yards will be excavated from the site for treatment. Additional excavation at Mine Fill A will be completed during Full Scale Operations.

Confirmation sampling in the excavation(s) for the purpose of closure will not be completed during the Pilot Scale phase. The 200 cubic yard excavation will be sampled for explosives, site-specific VOCs and metals (barium, cadmium, total chromium, and lead) prior to being temporarily backfilled. This data will assist in delineating further excavation at Mine Fill A.

4.1 INITIAL EXCAVATION SITE CHARACTERIZATION PROCEDURES

As defined in Section 1.4, this task has three specific objectives:

Objective 1: Identify areas for Pilot Scale excavation. Analyze the soil, primarily in the vicinity of Building 153 or Building 158 (backup in case Building 153 cannot be used during the Pilot Scale) for explosives. Additional excavation at Mine Fill A will be completed during Full Scale Operations. This data will also be used as "entry " or "startup" concentrations for composting.

Objective 2: Determine if site-specific waste products are present. Site specific VOCs include methylene chloride, acetone, methyl ethyl ketone, methyl isobutyl ketone, toluene and xylene. These constituents will serve as indicators of the presence of paint wastes. Depending on VOC results, additional testing for naphtha, methanol, isopropanol and ethanol may be required. Site specific metals include barium, cadmium, chromium and lead.

Objective 3: Determine if soil is hazardous with respect to metals and TCLP list VOCs. Soil will not be accepted into the Biofacility for treatment if it is hazardous with respect to these constituents.

These three objectives will be met by collecting soil samples in a general area where high levels of explosives have been previously detected (Figure 1-5). Associated soil sample data, collected in 1985, are summarized in Table 1-1. Soil samples will be collected and analyzed for explosives; site-specific VOCs and metals; TCLP-metals; and TCLP list VOCs. This data will assist in identifying areas for Pilot Scale excavation. This data will not be used to define the extent of contamination.

Note that in this section and in Table 4-1, "VOCs analysis" indicates the soil will be tested for site-specific VOCs and TCLP list VOCs. Only one sample will be collected for analysis of both sets of VOCs.

Sampling Areas

Four sampling areas have been designated north and northeast of Building 153 (Figure 4-1). These areas encompass an earthen berm around Building 153 which is potentially contaminated with explosives, based on previous sampling results (Table 1-1). Additional sampling areas, north and east of the turn-around road will be gridded using a 20-foot spacing.

Grid layout and boundaries will be affected by the presence of buildings, utilities, etc. Grid borders, including those of sampling areas 1-4, will be flagged in the field and areas will be chosen which will be accessible by heavy equipment (i.e. individual grid blocks will not be placed in the midst of heavy utilities or surface structures). Grid blocks are not expected to be perfectly square (i.e. those in the berm), but should roughly represent a 20-foot by 20-foot area. Assuming two feet of soil will be excavated, each 20-foot by 20-foot area or "grid block" will represent approximately 30 cubic yards of soil.

Soil Sampling

Soil samples will be collected from each grid block or sampling area using the procedure provided in Table 4-1, which is highlighted as follows:

For each grid block:

Volatile Organics Sampling

-At a minimum, one sample for VOCs analysis will be collected from the center of the grid block using a drive sampler fitted with two brass sleeves (one sleeve for VOCs, the second sleeve for metals/explosives grab sample). **A drive sampler, fitted with a brass sleeve, will be used to collect VOCs samples. Once the sleeve is removed from the sampler, each end will be covered with teflon film, capped and taped. These steps should minimize loss of VOCs.**

-The soil will be examined, at each grab sample location, to determine if unusual odors, stains or other indicators are present (i.e. high PID readings) of organic contamination. Based on these field observations, additional VOC samples may be collected.

-VOCs samples will be collected from the 12- to 18-inch depth only, unless otherwise indicated by field observation.

-Grab samples for metals/explosives will be held separately.

Metals/Explosives Sampling

-Four additional grab samples will be collected from each corner of the grid block, as shown in Figure 4-2, for the 6- to 12-inch depth and 2- to 3-foot depth, using a drive sampler fitted with brass sleeves.

-For each depth:

-The four grab samples plus the one from the center of the grid block will be composited for explosives and metals analyses. The grabs will be combined to form one composite sample as follows:

Place each grab sample into an aluminum pie pan or cookie sheet, after removing vegetation and coarse fractions. Break up the soil using gloved hands, and remove large rocks (the soil may also be sieved). Allow the soil to air dry. Cover the pie pan with a second pie pan and shake and swirl vigorously to disperse and homogenize the soil. Cone and quarter the soil in the pie pan. Collect an equal portion from each quarter and composite to fill an 8-ounce jar for explosives analysis.

This process will provide a more representative sample as explosives contamination is known to vary over short distances (Crockett and others, 1996).

-Combine the remaining soil together for metals analysis (barium, total chromium, lead and cadmium) and TCLP-metals analysis. One 8-ounce jar will be required for each analysis.

As a result, for each grid block, a minimum of two composite samples will be collected for metals (barium, total chromium, lead, cadmium), TCLP-metals and explosives. A minimum of one sample will be collected for VOC analysis from the center of the grid block, from a depth of 12 to 18 inches.

Marking Grid Blocks for Excavation

Explosives data will be used to mark grid blocks or sampling areas as requiring excavation. If a grid block or sampling area shows contamination (> 30 mg/kg TNT or RDX) to a depth of two feet, the grid block or area will be marked for two feet of excavation. If a grid block or area shows contamination at the six-inch depth only, the block or area will be marked for one foot of excavation. If a grid block shows no contamination at six inches nor at the two-foot depth, additional grid blocks will be sampled. Sampling will continue until enough soil is "marked" for excavation (200 cubic yards minimum for Pilot Scale).

4.2 EQUIPMENT WASH WATER/DECONTAMINATiON WATER SAMPLING

Decontamination water generated at the staging area at Mine Fill A and at the Biofacility will be collected and stored in a tank. This water will be used in the process only in the startup phase. Once the treatment process is underway, only potable water will be added to enhance moisture content in the compost piles. Any solids which collect in the sumps at the Biofacility will also be added to the compost during the initial phase only.

Decontamination water stored in a tank will be sampled for explosives if used in the treatment process, or will be sampled for explosives, **RCRA metals** and POTW requirements if shipped to an on-site treatment facility. Sampling procedures are provided in Table 4-2.

The samples will be labeled and stored in a cooler with ice and kept at approximately 4 degrees Celsius. Sample documentation, packaging, and shipping procedures will be followed as described in Section 4.8 and Section 5.0, Custody Procedures.

Disposable bailers or disposable pump tubing will be used to sample tanks, eliminating the need for decontamination. Used disposable bailers will be disposed with used PPE.

4.3 STORM WATER RETENTION POND SAMPLING

Surface runoff or storm water present on the Biofacility asphalt-paved areas will be diverted to two ponds, #1 and #2. The water in the retention ponds will be sampled to determine if NPDES discharge requirements are met. Sampling procedures are provided in Table 4-3. Samples will be tested for NPDES parameters listed in Table 4-8.

The samples will be labeled and stored in a cooler with ice and kept at approximately 4 degrees Celsius. Sample documentation, packaging, and shipping procedures will be followed as described in Section 4.8 and Section 5.0, Custody Procedures. Disposable bailers or disposable pump tubing will be used to sample the retention pond water, eliminating the need for decontamination. Any used disposable bailers will be disposed with used PPE.

4.4 PROCESS SAMPLING

Amendments will be sampled prior to addition of soil excavated from Mine Fill A, and analyzed for TOC and TKN to determine initial levels of organic carbon and nitrogen.

During the Pilot Scale, approximately eight compost piles, consisting of a mixture of soil and amendments, will be tested. This process is anticipated to take a minimum of 28 ~~days~~ **30 days for clean soil and 60 days for contaminated soil after that**, as dictated in the Pilot Scale Operational Plan. Each pile will be 25-30 feet long, 7 feet high and 20 feet wide. Sampling locations are displayed in Figure 4-3. Cross-section A, B, C and D will be sampled throughout the process for various parameters to be analyzed by an off-site laboratory. The schedule for sampling is as follows:

Windrow/Compost Sampling Schedule (off-site analyses) - Pilot Scale				
Parameter	**Analytical Method**	**Number of Samples per Sampling Event**	**Volume Required per sample**	**Schedule**
Explosives (full list)	8330	3 per cross-section (12 total)	8 ounces	Day ~~0, 7, 14, 21 and 28~~ **0, 5, 10, 20, 30, 40, and 60**
TCLP-metals	1311, 6010, 7470	3 per cross-section (12 total)	8 ounces	Day 0 and ~~28~~ **60**
Metals (barium, cadmium, total chromium, lead)	6010	3 per cross-section (12 total)	8 ounces	Day 0 and ~~28~~ **60**
TOC	9060	3 per cross-section (12 total)	8 ounces (both analyses may be done using 8 ounces of soil)	Day ~~0, 7, 14, 21 and 28~~ **0, 5, 10, 20, 30, 40, and 60**
TKN	EPA 351.3	3 per cross-section (12 total)		Day ~~0, 7, 14, 21 and 28~~ **0, 5, 10, 20, 30, 40, and 60**

Once process samples have shown that explosive concentrations have been consistently reduced over time for one or more compost piles or mixtures, a confirmation sample will be collected. Confirmation samples are anticipated to be collected on Day ~~28~~ **60** of processing. However, should processing be extended, confirmation samples will be completed at the end of the time extension. Confirmation samples will be collected using procedures in Table 4-4.

Treated compost which does not meet the process goals for TNT and RDX using all mixtures may also require off-site disposal, especially if it cannot be placed in the on-site landfill. Soil and/or treated compost which requires off-site disposal will be stockpiled and sampled at a frequency to be determined by the Project Manager and the Navy. This task will be implemented during Full Scale Operations.

4.4.1 Other Process Parameters

Field testing equipment will consist of pH, oxygen and temperature direct reading probes. Oxygen and temperature will be measured by pushing the individual probes into the center of each sampling sector of the compost piles and allowing enough time for an accurate reading, per manufacturer recommendation. Compost pH will be determined by measuring the pH level in a solution of deionized water and a compost sample as described in Section 7.2 of the Pilot Scale Operational Plan. Soil moisture will be measured by weighing the sample, drying it, reweighing it and calculating the percent moisture. Frequency of pH, oxygen, temperature and soil moisture measurements are described in Section 8 of the Pilot Scale Operational Plan.

4.4.2 Air Monitoring

The concentration of methane gas, a decomposition by-product, will be measured in the air above the compost piles using a Dynamation Gas Detection Instrument, Model Combo 435. All measurements will be recorded in the field log book. Further information on methane monitoring may be found in Section 11 of the Operational Plan and the SSHP.

4.5 EXCAVATION SAMPLING PROCEDURE

Approximately 200 cubic yards will be required for Pilot Scale operations. Once the excavation is completed, the hole will be temporarily backfilled using soil from an approved source at Crane. Prior to backfilling, the excavation will be sampled to determine levels of explosives, site-specific VOCs and site-specific metals, if any, present above initial remedial goals. This data will be collected to guide further excavation at Mine Fill A during Full Scale Operations.

The base and walls of the excavation will be gridded using 20-foot spacings. Samples will be collected from approximately six inches below ground surface, using procedures in Table 4-1. Samples will be analyzed for explosives, site-specific VOCs and site-specific metals only.

4.6 QA/QC SAMPLES

QC samples will be collected to ensure that representative and reproducible data are obtained. The types of QC samples that will be used are discussed below. QC samples apply only to samples submitted for off-site analysis by a laboratory.

4.6.1. Trip Blanks

A trip blank is a QC sample that consists of two vials filled with organic-free water and is prepared in the laboratory, shipped to the sampling site with the sample containers, and returned to the laboratory with the samples to be analyzed for VOCs. One trip blank will accompany each cooler containing VOC samples and will be stored at the laboratory with the samples and analyzed by the laboratory. Trip blanks will be analyzed for VOCs only.

4.6.2 Equipment Rinsate Blanks

Equipment rinsate blanks (or rinsate blanks) are collected to ensure that sampling equipment is clean and that the potential for cross-contamination has been minimized or prevented. Rinsate blanks are collected from the final reagent grade water rinse used in sample equipment decontamination. Rinsate blanks will be collected ~~early in the day on a daily basis~~ and analyzed at a frequency of one **per 10** samples collected, or at a frequency listed with the sampling procedures, whichever is more stringent. A reduction in the frequency of rinsate blanks may be deemed justifiable by the Project Chemist based on review of analytical results which would need to show no discernable evidence of introduction of cross-contamination due to inadequate equipment cleansing.

The rinsate blank is obtained by first decontaminating the sampling device and then pouring reagent grade water over the device. This water is provided by an analytical laboratory at NSWC Crane. The rinsate blank water is collected directly into the sample bottles. The rinsate blanks are analyzed for the same constituents as the related samples. The results will be used to flag or assess the levels of analyses in the samples and evaluate the effectiveness of sampling equipment decontamination.

4.6.3 Field Blanks

Two types of field blanks will be collected during the Pilot Scale operations. The first type (A) is defined as follows:

> "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 regular samples." (NEESA, 1988)

As a result, samples of decontamination water used, including the potable water used during treatment, will be collected at a frequency of 1 per source per event, as shown in Table 4-6.

The second type of field blank (B) to be collected is defined as follows:

> "(A field blank is) is used to determine if any contaminants present at the site may have an effect on sample integrity. (The blank is) prepared by pouring distilled/deionized water into sampling containers at certain sampling locations (typically areas where dust and/or volatile organic contamination may emanate from other sources)." (Hazardous Waste Consultant, 1992)

Field Blank B will be collected at a frequency of 1 per 20 regular samples. Frequency of collection and analysis of these blanks (type A and type B) will be finalized by the Project Chemist.

4.6.4 Field Duplicate Samples

Field duplicate samples will be collected to allow determination of analytical and sampling precision. One duplicate sample per ten samples will be collected and submitted for the same analysis as the regular samples. The duplicate sample will be collected simultaneously with the true sample. The sample will be collected with the same sampling equipment at the same location as the regular sample, but it will be labeled with the next consecutive sample number.

4.6.5 Matrix Spike Samples

Matrix spike/matrix spike duplicates (MS/MSD) samples will be used as further QC checks. These samples will be spiked at the laboratory. The samples will be collected and prepared at the frequency of one MS and MSD for every 20 field samples (including trip blanks, field blanks, and duplicates). The samples will allow accuracy to be determined by the recovery rates of compounds (the matrix spike and/or surrogate spike compounds defined in the analytical methods). Precision will also be assessed by comparison of matrix spike duplicate recoveries. The purpose of these laboratory spikes is to monitor any possible matrix effects specific to samples collected from the site. The addition of known concentrations of compounds/ constituents to the sample also monitors extraction/digestion efficiency.

MS/MSD samples will be collected to provide sufficient volume per QC and analytical laboratory requirements. The laboratory will select aliquots that are as homogeneous as possible with respect to one another to avoid precision problems related to sample homogeneity.

4.7 DECONTAMINATION PROCEDURES

4.7.1 Sample Bottles

Sample bottles will be provided by the analytical laboratory. These bottles will arrive at the site in sealed boxes or coolers. Certification of cleanliness will accompany each box of bottles. The exception to this would be if any bottles were pre-preserved by the lab.

If certification is not included or cleanliness of the containers is suspect, USEPA approved cleaning procedures will be carefully conducted by the analytical laboratory. The procedures are as follows:

Metals: Detergent wash, tap water rinse, 1:1 HNO_3 rinse, tap water rinse, 1:1 HCL rinse, final reagent grade water rinse.

Volatiles (40 ml glass vials and septa): Non-phosphate detergent wash, tap water rinse, reagent grade water wash, dry (glassware only) in a muffle furnace at 105 degrees Celsius for one hour.

Explosives: Non-phosphate detergent wash, tap water rinse, deionized water rinse, methanol rinse, final reagent grade water rinse.

4.7.2 Sample Equipment Decontamination

Decontamination of construction and field equipment will take place in a designated area using procedures described in Section 6.2 of the Pilot Scale Operational Plan. All non-disposable sampling equipment, including stainless steel trowels and spoons, will be decontaminated according to the following procedure and sequence, depending on the contaminants involved:

1) Manual scrub with Alconox and tap water wash.
2) Rinse with potable water.
3) 10% nitric acid rinse (for metals only).

4) Rinse with reagent grade water.
~~5) Reagent grade water rinse.~~
~~6)~~ **5)** Air dry.

Once dry, sampling equipment will be stored in plastic bags to prevent contamination.

Decontamination water from equipment and vehicle decontamination activities will be stored in a dedicated tank. Decontamination water and water collected in sumps will either be recycled as compost process water or transported to a wastewater treatment facility (WWTF) at Rockeye or Mine Fill A. Water shipped to the WWTF will be sampled and analyzed per waste water treatment facility permit requirements.

4.8 SAMPLE PACKAGING AND SHIPPING

Immediately after collection, samples will be properly labeled, tagged and properly preserved. Table 4-7 lists the proper container, volume requirements, and preservation needed for the sampling effort. Samples requiring refrigeration for preservation will be immediately placed in coolers packed with ice or ice packs. Proper chain-of-custody documentation will be maintained as discussed in Section 5.0 - Custody Procedures.

Packaging and shipping procedures will vary depending upon sample media, potential contaminant concentration, preservation technique, and sample container. General procedures are provided in Table 4-5. A waterproof metal or equivalent strength ice chest or cooler is suitable for packaging and shipping samples not considered "Dangerous Goods" by the U.S. Department of Transportation (USDOT). The person packaging the samples is responsible for ensuring that the cooler is in suitable condition for shipping. In the event a concern arises regarding whether a sample shipment is a USDOT "Dangerous Good" or is potentially in violation of international transportation regulations, the Project Engineer or Project Manager will be consulted.

Sample packaging should ensure that the chance of breakage is minimized and that, in the event a sample container breaks or leaks, it will not impact the integrity of other samples in the shipment. Also, samples should be protected from ice melt. Each sample container should be placed in a Ziploc™-type bag, and wrapped in packing material such as bubble wrap or comparable shock-absorbing materials when shipping glass containers to avoid breakage. Adequate ice, contained in double Ziploc™-type bags, must be included with each cooler shipment so that the contents are maintained at 4 (± 2) degrees Celsius until receipt at the laboratory. If necessary, a temperature blank will be shipped with each cooler.

In addition to an adhesive label, sample tags will be tied around the jars, as possible. The tags will include the same information recorded on the label.

A chain-of-custody record, protected from moisture by enclosure in a Ziploc™-type bag, must accompany each cooler shipped. The cooler lid should be secured using packing tape around the outside of the cooler. Custody seals will be signed, dated and affixed to the front, side and back of the cooler. The seals should be placed in such a way that tampering with the cooler lid will result in a ripped seal.

TABLE 4-1. SOIL SAMPLE COLLECTION (TASK 1 AND TASK 5)
Objectives: Task 1: -Identify areas for Pilot Scale excavation. -Measure "entry " or "startup" concentrations of explosives in soil to be treated. -Determine if soil is hazardous with respect to metals and VOCs. Task 5: -Determine levels of explosives and metals still remaining following Pilot Scale excavation
Equipment: -small stainless steel trowel, stainless steel hand shovel, stainless steel spoons -large shovel -flagging and pins -measuring tape -sample containers -aluminum pie pans -PID -drive sampler -brass sleeves and endcaps -teflon film -plastic sheets -stainless steel bowls

TABLE 4-1.
SOIL SAMPLE COLLECTION (TASK 1 AND TASK 5)

Methods:

Placing the Sampling Grid

Task 1: Initial Excavation Site Characterization

1. *Layout a grid using 20-foot spacings west of the steam line and north of the conveyor, in the* vicinity of Building 153. Each 20 x 20-foot grid block may or may not be square, due to the presence of utilities, buildings or roads (see example Figure 4-2).
2. Label these blocks or sampling areas, beginning with "1," as shown in Figure 4-1. Note that sampling areas 1-4 have already been designated.

Task 5: Post-Excavation Sampling

1. *Layout a grid using 20-foot spacings along the base of the excavation(s). Place pin flags at each* node.
2. For the sidewalls, place pin flags every 20 feet, along each wall. Place the flags midway between ground surface and the excavation base. If a sidewall is less than one foot in height, do not place flags and do not collect samples from the wall.

TABLE 4-1.
SOIL SAMPLE COLLECTION (TASK 1 AND TASK 5)

Method (continued):

Sample Collection - 20-foot x 20-foot Grid Blocks (Task 1)

NOTE: 2-inch diameter, 6-inch long sleeves will be used in the drive sampler.

1. Follow these steps when sampling each grid block or sampling area:
   a. Place flags near each corner and at the center (see Figure 4-2 for an example).
   b. At the center of the grid block:
      -Clear surface vegetation and debris in a 6-inch by 6-inch area.
      -Using a stainless steel trowel and spoon, remove the upper 6 inches of soil.
      -Using a drive sampler fitted with two brass sleeves, collect soil from the 6-inch depth to 12-inch depth; and from the 12-inch depth to the 18-inch depth.
      -Label the sleeve from the 12-18 inch depth as the volatile organics sample. Cover both ends of the sleeve with teflon film and cap. Store the sleeve in a cooler with bagged ice.
      -Take the other sleeve and push the contents into a stainless steel bowl labeled "metals/explosives grabs - 6-12 inches."
      -Remove soil from the boring down to 2 feet.
      -Using a drive sampler fitted with two sleeves, collect soil from the 2-foot to 3-foot range. Push the contents of shallow sleeve (2-2.5 feet) into a second stainless steel bowl labeled "metals/explosives grabs - 2-3 feet," break up the soil, and cover with a plastic sheet. Wait a few minutes, lift the plastic, and screen the headspace of the bowl using a PID.
         -If readings are above background levels, cover both ends of the deeper sleeve (2.5-3 feet) with teflon film and cap. Submit the capped sleeve for VOCs analysis, as authorized by the MK Project Manager.
         -If PID readings are not > background, place the contents of the deeper sleeve into the bowl.
   c. At each of the four corners:
      -Clear surface vegetation and debris in a 6-inch by 6-inch area.
      -Using a stainless steel trowel and spoon, remove the upper 6 inches of soil.
      -Using a drive sampler fitted with two brass sleeves, collect soil from the 6-inch depth to 12-inch depth; and from the 12-inch depth to the 18-inch depth.
      -Push the contents of the 6-12 inch sleeve into the designated bowl. PID screen and evaluate the contents of this sleeve to determine if the second sleeve (12 to 18 inch depth) should be submitted for VOCs analysis.
      -Remove soil from the boring down to 2 feet.
      -Using a drive sampler fitted with two sleeves, collect soil from the 2-foot to 3-foot range. Push the contents of both sleeves into the stainless steel bowl labeled "metals/explosives grabs - 2-3 feet."
   d. At a minimum, one bowl of soil will result from the 6-12 inch depth and one bowl of soil will result from the 2-3 foot depth. A minimum of one sample, collected from the center of the grid block, will be tested for VOCs.

TABLE 4-1.
SOIL SAMPLE COLLECTION (TASK 1 AND TASK 5)

Method (continued):

NOTE: Note PID measurements in the field logbook. If unusual odors and stains are discovered and/or PID measurements are above background levels, collect a sample for VOCs analysis. Analysis of additional VOCs samples will be approved by the Project Manager or Project Chemist.

e. Use the following steps for forming a composite sample for each depth:
-Place each grab sample into a large aluminum pan, after removing vegetation.
-Break up the soil using gloved hands, remove large rocks (the soil may also be sieved), and allow soil to air dry.
-Cover the pie pan with a second pie pan and shake and swirl vigorously to disperse and homogenize the soil.
-Cone and quarter the soil in the pie pan.
-Collect equal portions from each quarter to fill one 8-ounce jar for explosives analysis.
-Fill the remaining jars (2 8-ounce jars) with soil for metals analysis. One jar will be labeled for TCLP-metals (Task 1 only). The second jar will be labeled for total metals (Ba, Cd, Cr, Pb only) (Task 1 and 5).

2. Record general sampling information including a description of the sampling location (including a sketch), sample I.D.s, date, time and observations (i.e. presence of stains, grain size, color, percent moisture)

3. If soil samples cannot be collected due to high clay content or high % of cobbles or gravel, move the affected soil boring to an adjacent location.

Sample Collection - 20 x 20-foot Grid Blocks (Task 5)

Follow the same procedure as for Task 1, however do not collect sleeves or samples from the 2-3 foot depth. Samples will only be analyzed for site-specific VOCs, site-specific metals and explosives (full Method 8330 list).

Sample Collection - Sidewalls (Task 5 only)

Collect a sample from each flagged location along each sidewall using the procedure from Step 1b. Submit one sleeve for site-specific VOCs analysis from 12-18 inches below ground surface. Submit one sleeve from 6-12 inches for explosives and metals (barium, cadmium, total chromium and lead).

TABLE 4-1.
SOIL SAMPLE COLLECTION (TASK 1 AND TASK 5)

QA/QC:

One field duplicate will be collected for every 10 regular samples collected. Field duplicate locations will be marked in the field using different colored flags. Additional volume for the field duplicate (and MS/MSD) will be drawn from the composite mixture for metals and explosives. For the VOCs field duplicate, a sleeve of soil will be collected from the 18-24 inch depth, at the center of the grid block. For Task 1, the duplicate will be analyzed for metals (barium, cadmium, total chromium, lead), TCLP-metals, explosives, site-specific VOCs (see Table 1-4 or 4-7 for list of VOCs), and TCLP list VOCs. For Task 5, the duplicate will be analyzed for explosives, site-specific VOCs and site-specific metals ONLY.

Field equipment will be decontaminated using procedures in Section 4.7. One equipment rinsate will be collected for every 10 regular samples collected. Sampling parameters are listed in Table 4-6.

One field blank (type B) will be collected for every 20 regular samples collected to determine if any contaminants were introduced during sampling. An additional field blank (type A) will be collected of the water used in decontamination. Sampling parameters are listed in Table 4-6.

One trip blank will be submitted with the cooler containing the VOCs samples. Each trip blank will be tested for VOCs only.

TABLE 4-2.
WASTE WATER STORAGE TANK SAMPLING PROCEDURE

Objective: To collect representative samples and obtain data necessary to properly dispose of wastewater generated during site activities.

Equipment:
-stainless steel bucket or ladle
-disposable Teflon or stainless steel bailer
-slow rate pump and disposable tubing (may be used for tank sampling if tank is too large to access with a bailer)
-fishing line, cord and reel

Method:

Waste water generated during decontamination activities will be pumped into holding tanks. Following solids settling, a minimum of one water sample will be collected from each tank. It is anticipated that only explosives data are required. However, a procedure for VOCs sampling is included as a contingency.

1. If the tank is not fitted with a valve, a submersible pump will be lowered midway between the water surface and tank bottom. Care will be taken not to disturb the settled solids at the tank bottom.

2. If VOCs are required, collect volume for this analysis first. Lower the pump slowly into the water to avoid degassing. Pump the water from the tank at a low rate (200 mL/min or less).
   a. Slowly fill the volatile organic analysis (VOA) vials, taking care not to force out the preservative.
   b. Slightly overfill vial to form a meniscus.
   c. Tightly cap the vial and invert the bottle.
   d. Tap the sides of the vial to see if air bubbles are trapped in the sample.
   e. If air bubbles are present, place the vial in an upright position and open the cap. Repeat steps "a" through "d". If air bubbles are still present, discard sample and redo all steps.

   A disposable Teflon or decontaminated stainless steel bailer may be used in place of a pump and tubing. Slowly lower the bailer beneath the water surface and fill. Attach a VOA vial "bottom-emptier" device to the bottom of the bailer. NOTE; the bailer will begin to drain immediately. Be sure that the bottom fill device tube has been placed in the vial prior to attachment to the bailer. Fill the vial until full. Follow steps a through e to properly seal the vials.

TABLE 4-2.
WASTE WATER STORAGE TANK SAMPLING PROCEDURE

Method (continued):

3. Remaining sample containers may be filled using pump and tubing or directly from a bailer. Using a fresh bailer volume, fill the remaining containers to nearly full (usually to the lip of the container).

4. A sample can be obtained through a bottom valve if no other access point is available. The sample may be rather turbid due to high solids content at the bottom of the tank. Consult the Project Manager to determine if the samples will be filtered for metals in this case. Also determine if the sludge/solids at the base of the holding tank will be sampled as well.

Documentation and Shipping
Documentation procedures are provided in Section 5.0. Packaging and shipping instructions are provided in Table 4-5.

QA/QC:
It is anticipated that 1-2 samples of wastewater will be collected. At the Project Chemist's discretion, one field duplicate will be collected and analyzed for the same parameters as the regular samples.

If non-disposable equipment is used for sampling (i.e stainless steel bailer), one rinsate blank will be collected and analyzed for the same parameters as the regular samples.

One trip blank will be submitted with the cooler containing any VOCs samples. Each trip blank will be tested for VOCs only.

TABLE 4-3. STORM WATER RETENTION POND SAMPLING PROCEDURE

Objective: To determine if NPDES requirements have been met for on-site disposal.

Equipment:
-Teflon or polyethylene disposable **bottom-filling** bailers ~~or stainless steel bailers~~
-fishing line and reel
~~-slow rate pump and tubing/hose~~ **container on a rod**

Method:

During bioremediation activities, surface water, such as incidental storm runoff from the site, will be diverted to storm water retention ponds #1 and #2. This water will be sampled to determine if disposal criteria or NPDES requirements have been met (Table 4-8). The sampling procedure for the storm water retention ponds is provided below.

1. Collect a water sample using a sample collection device such as a disposable **bottom filling** bailer or ~~pump~~ **or surface water sampling device**. Fill the bottle directly from the bailer or ~~pump tubing~~ **grab sample container**. If the capacity of the bottle exceeds the capacity of the bailer, begin filling all the bottles from each bailer sample until all the bottles are filled. Lower the bailer to the same depth each time if more than one bailer volume is needed. Take care not to disturb bottom sediments in the pond(s).

2. Document the approximate location and depth the water sample was collected.

3. Clean the exterior of the sample container, and ensure that the container is tightly sealed before applying the sample label.

Documentation and Shipping
Documentation procedures are provided in Section 5.0. Packaging and shipping instructions are provided in Table 4-5.

QA/QC:

It is anticipated that several samples of pond water will be collected. At the Project Chemist's discretion, one field duplicate will be collected and analyzed for the same parameters as the regular samples.

If non-disposable equipment is used for sampling (i.e stainless steel bailer **is substituted**), one equipment rinsate will be collected and analyzed for the same parameters as the regular samples.

TABLE 4-4. COMPOST SAMPLING PROCEDURE

Objective: To support the operations at the Biofacility and monitor the effectiveness of soil remediation using composting.

Equipment:
-pH monitors
-temperature and oxygen probes
-portable oven (moisture content) and scale
-~~hand auger, sample bucket~~ **stainless steel push tube with sleeves or split spoons** and attachments or drive sampler
-PID
-stainless steel spoons, bowl and trowels
-riffle splitter
-No. 4 Wiley Mill, or equivalent

Method:

Consult Section 7.2 of the Operational Plan for methods and devices used to measure moisture content, temperature, oxygen and pH. Use the following steps to collect samples of compost for off-site analysis. Sample locations are provided in Figure 4-3.

1. Place temporary flags at each cross section to be sampled.
2. Take the sample marked "1" in Figure 4-3 first. Using the decontaminated ~~hand auger~~ **push tube** and attachments, ~~bore~~ **push** down approximately three feet and collect a sample. Retrieve enough volume to fill all required sample containers (see Section 4.4).
3. Place the contents of the ~~bucket~~ **tube** in a stainless steel bowl. Allow compost to air dry.
4. Macerate (soften) air-dried compost in No. 4 Wiley mill. Split the sample using a riffle splitter to fill containers.
5. Collect the samples marked "2" and "3" using a ~~hand auger~~ **push tube**. Make sure the ~~auger~~ **tube**, ~~bucket~~ and attachments are decontaminated prior to sampling the pile and in between sampling locations.
6. Take care not to sample material which has caved into the boring. If necessary, use a long length of PVC pipe to keep the hole open once the initial boring has been made.
7. Record all observations, including test results and off-site sample information (sample I.D., date, time, analysis), in the field log book. All field equipment will be calibrated daily per manufacturer guidelines. Record calibration data in the field logbook.
8. Samples for off-site analysis will be collected at the frequency summarized in Section 4.4.

Documentation and Shipping
Documentation procedures are provided in Section 5.0. Packaging and shipping instructions are provided in Table 4-5.

TABLE 4-4. COMPOST SAMPLING PROCEDURE
QA/QC: One field duplicate will be collected per compost pile, per sampling event, and analyzed for the same parameters as the regular samples (explosives, metals (barium, cadmium, total chromium, lead), TCLP-metals, TKN, TOC). Field equipment will be decontaminated using procedures in Section 4.7. One equipment rinsate will be collected per 20 samples collected and analyzed for the same parameters as the regular samples (explosives, metals (barium, cadmium, total chromium, lead), TKN, TOC). One field blank (type B) will be collected at the composting area (1 per 20 samples) to determine if any contaminants were introduced during sampling. An additional field blank (type A) will be collected of the water used in decontamination. Both blanks will be tested for the same parameters as the regular samples (explosives, metals (barium, cadmium, total chromium, lead), TKN, TOC). A third field blank will be collected from the water used in the process, unless the source is the same as the potable water source for decontamination.

TABLE 4-5. PROCEDURE FOR SAMPLE DOCUMENTATION, PACKAGING AND SHIPPING

Objective:
To ensure that samples are properly packaged to prevent or minimize breakage and maintain sample integrity between the field and laboratory, and to maintain proper chain-of-custody protocols.

General:

For waste characterization samples, the Subcontractor shall be responsible for shipping samples to a Subcontractor-procured laboratory. All decontamination and rinse samples will be submitted by the Subcontractor to the Contractor-procured laboratory. The name and address of the Contractor-procured laboratory will be provided prior to work.

One trip blank (consisting of two 40-mL vials) will be submitted with each cooler of volatile organics samples shipped. All trip blanks will be prepared and shipped to the site by the Contractor-procured laboratory.

For each sample shipment, complete sample documentation, including labels, tags, chain-of-custody (COC) forms, etc. and properly package sample containers for shipment to the laboratory in accordance with the following procedures:

Sample Labels and Tags.

After a sample is collected, it shall be temporarily stored in the field in a cooler with 2-3 bags of ice. When sampling is completed, the cooler(s) shall be brought to the support zone. Any empty containers found in the cooler shall be discarded.

All sample containers sent to the laboratory shall be labeled and tagged, as possible. Labels shall be completed in ink. If an error is made on a label, strike out th erroneous item with a single line and initial the strike out.

At a minimum, the following information shall be included on each sample label and tag:

- Sample identification number;
- Analytical method; and
- Date and time of collection.

If enough room is available on the sample label or tag, the following information shall also be included:

- Site name,
- Location of Sample,
- Initials of sample collector, and
- Preservative, if applicable.

TABLE 4-5. PROCEDURE FOR SAMPLE DOCUMENTATION, PACKAGING AND SHIPPING

Chain-of-Custody Form (COC).

Once the samples have been recorded in the field logbook, complete the Chain-of-Custody (COC) form. Prior to filling out the COC form, verify that all samples are accounted for and all volume requirements are met. At least one COC shall be completed for each cooler of samples to be shipped. COC forms will vary slightly depending on the laboratory providing the forms. The Contractor will specify which COC format is to be used for decon and rinse water samples. The following general instructions shall be followed when completing the COC form:

a. If the COC form does not have a unique document number, assign a sequential reference number to the COC form and record this number in the top right corner of the document (i.e. COC #001). Each COC form shall have a unique reference number. Reference numbers, whether already included on the document, or assigned, shall be recorded in the field logbook.

b. Include one line on the COC form for each sample. Fill in the appropriate information in the columns on the form for each sample. Ensure the following information is included for each sample:
   - sample identification number;
   - sample location;
   - date sample was collected;
   - time sample was collected;
   - sample matrix (i.e. water, sludge);
   - size and quantity of containers included; and
   - required analyses.

c. Specify QC samples (i.e. field blank, rinsate blank and trip blank), when collected. DO NOT specify duplicate samples. Trip blank "sample time" must be entered on the COC form. Use the time the cooler was sealed.

d. Ensure the sample time and sample I.D. on the COC form are exactly the same as on the sample container label or tag.

e. When specifying an analysis, write both the analysis and the method number.

f. Enter the turnaround time for the samples in the "Remarks" section of the COC form.

g. Complete the "Relinquished By" information (person, date and time) at the bottom of the COC form. The "Relinquished Time" should be roughly the time the cooler is sealed and given to overnight shipper.

h. Include the overnight shipment airbill number somewhere on the COC form. Record this number in the field logbook.

i. Keep one copy of the COC form for site records and submit the remaining copies to the Laboratory. Seal the Laboratory copies in a gallon size Ziploc™, or equivalent, bag and affix to the underside of the cooler lid using strapping tape.

TABLE 4-5. PROCEDURE FOR SAMPLE DOCUMENTATION, PACKAGING AND SHIPPING

Sample Preparation.

The following tasks will be completed, prior to sample packaging:

a. Remove visible dirt, residue and excess moisture from the sample jars;

b. Check container lids to ensure they are on tight;

c. Place sample jars (glass) in protective bubble wrap and seal using adhesive strip on bubble wrap package or tape.

Sample Packaging.

Package samples carefully to prevent breakage or movement of containers during shipment. The following guidelines shall be used for packaging:

a. Line the base and sides of the cooler with bubble wrap. Coolers designated for decon and rinse water samples will be provided by the Contractor-procured laboratory.

b. Pack the larger containers into the cooler first. Place double-bagged cubed ice or small containers between larger containers then pack the remaining smaller containers. Add additional double-bagged ice to the cooler, distributing as evenly as possible to chill all samples. This is **most important** with samples requiring VOC analysis.

c. Place a trip blank in each cooler containing samples to be analyzed for volatile organic compounds. The trip blank is a regular sample and must be labeled and recorded as such. It shall be analyzed for volatile organics only. When possible, keep all volatile organic analyses samples together in the same cooler to minimize the number of trip blanks required.

d. Don't overload the cooler with samples. Allow enough room for a layer of ice at the top, as well as a final layer of bubble wrap. At least one COC form shall be filled out for each cooler of samples. The samples and ice should be packed tight enough to prevent movement of containers during shipment.

e. Close the cooler and seal closed with custody seals. Use 2-3 custody seals (signed and dated) per cooler (small or large). Place seals on the front, side and back of each cooler where they shall be visible and easily ripped if the cooler is tampered with. Use a thin cover of strapping tape to protect the edges of the seal during shipment.

f. Seal the cooler with strapping tape.

TABLE 4-5. PROCEDURE FOR SAMPLE DOCUMENTATION, PACKAGING AND SHIPPING
Sample Shipping. Ship all samples to the laboratory for next-day delivery. Ensure the air bills have been completely filled out and copied. Place one airbill into each envelope. Affix the envelope to the top of the cooler, if the lid is flat. If the lid is sliding, as with a "playmate" cooler, affix the envelope to the side of the cooler. Do not obstruct address labels provided by the laboratory (if present). **NOTE**: If samples are collected on Friday, the block labeled "Saturday Delivery" on the airbill MUST BE MARKED. Otherwise the samples will arrive at the lab on Monday. Fax the Chain-of-Custody Form to the Contractor Project Chemist or designee [(216) 523-5201] the same day samples are shipped. If short turn-around time is needed, such as 24 or 48-hours, notify the Project Chemist **IMMEDIATELY** by phone. It is preferable to notify the Project Chemist of short turn-around times as soon as it is known that samples will require this.
QA/QC and Documentation: Maintain all generated sample documentation (copies of COCs, field logbooks, etc) in the field during the course of the site work. This documentation may be managed by the Contractor, or if managed by the Subcontractor will be available for review. At the conclusion of site work, provide a copy of all documentation to the Contractor.

TABLE 4-6. SUMMARY OF SAMPLING AND ANALYSIS PROGRAM

Task	Matrix	Parameters	Estimated No. of Samples(a)		QA/QC Samples				
			RS	FD	FBA	FBB	RB	TB	MS/ MSD
1. Initial Excavation Site Characterization	Soil	total VOCs (site-specific and those on the TCLP list), metals (Ba, Cr, Cd, Pb), TCLP-metals, Explosives	14+	2+	1	1+	1+	4+	1+
2. Equipment Wash Water/ Decon Water	Water	Explosives, **RCRA metals**, POTW parameters (as required by NSWC Crane)	3+	1	1	1	0	0	1
3. Storm Water Retention Pond Sampling	Water	NPDES parameters (listed in Table 4-8)	3+	1	1	1	0	0	1
4. Compost (Process) Sampling (includes confirmation sampling)	Compost + soil	Explosives, TOC, TKN (5 events: day ~~0, 7, 14, 21, 28)~~ **0, 5, 10, 20, 30, 40 and 60)**	12 per pile per event	1 per pile per event	1 per event (b)	2 per event	2 per event	0	5 per event
		metals (Ba, Cd, Cr, Pb), TCLP-metals (2 events: day 0, ~~28~~ **60** only)	12 per pile per event	1 per pile per event	1 per event (b)	2 per event	2 per event	0	5 per event
	Amend-ment(s)	TOC, TKN	8	1	0	0	1	0	1
5. Excavation Soil Sampling	Soil	Explosives, site-specific VOCs, metals (Ba, Cd, Cr, Pb)	10+	1+	1	1+	1+	4+	1+

TABLE 4-6. SUMMARY OF SAMPLING AND ANALYSIS PROGRAM

Task	Matrix	Parameters	Estimated No. of Samples(a)		QA/QC Samples				
			RS	FD	FBA	FBB	RB	TB	MS/ MSD

Notes:

FBA = field blank type A (sample of waters used for decontamination, including distilled/deionized water and potable water); type A field blanks will be collected at a frequency of 1 per event per source of tap water for decontamination; analytical frequency will be finalized by the Project Chemist

FBB = field blank type B (water sample prepared during sampling event to determine if contamination introduced at sampling site); type B field blanks will be collected at a frequency of 1 per 20 regular samples; analytical frequency will be finalized by the Project Chemist

RB = rinsate blank or equipment rinsate blank

TB = trip blank (will be prepared by laboratory and tested for VOCs only)

MS/MSD = matrix spike and matrix spike duplicate (collected at a frequency of 1 per 20 samples collected)

RS = regular sample

FD = field duplicate

a. Sample totals are estimated. The numbers may increase or decrease based on field conditions.
b. An additional field blank (A) will be collected of the water used in composting (one time sampling only), unless it is the same water used in decontamination and steam cleaning.

TABLE 4-7. ANALYTICAL METHOD REQUIREMENTS

Analyte	Analytical Method[1]	Sample Container[2]	Preservation	Holding Time
Initial Excavation Site Characterization Analyses (soil), Compost Solids, Soil in Excavation				
Volatile Organic Compounds (VOCs)	8240: methylene chloride, acetone, xylene, toluene, methyl ethyl ketone, methyl isobutyl ketone	1 4-ounce glass jar with Teflon-lined lid or brass sleeve (pack tightly!)	Ice to 4 C	14 days
VOCs (TCLP list)	8240: benzene, carbon tetrachloride, chloroform, chlorobenzene, 1,2-dichloroethane(1,2-DCA), 1,1-dichloroethene (1,1-DCE), methyl ethyl ketone, tetrachloroethene, trichloroethene, vinyl chloride			
Explosives	8330 (full list)	1 8-ounce glass jar with Teflon-lined lid or brass sleeve	Ice to 4 C	14 days; analyze within 40 days after extraction
Total metals (Barium, cadmium, total chromium, lead only)	6010	1 8-ounce glass jar with Teflon-lined lid or brass sleeve	Ice to 4 C	180 days
TCLP metals	1311, 6010, 7470	1 500 mL, plastic	HNO3 to pH < 2; ice to 4 degrees C	mercury; field collection to extraction 28 days; from extraction to analysis 28 days; other metals: 180 days from extraction to analysis

TABLE 4-7. ANALYTICAL METHOD REQUIREMENTS

Analyte	Analytical Method[1]	Sample Container[2]	Preservation	Holding Time
Total organic carbon (TOC)	9060	1 8-ounce glass jar with Teflon-lined lid or brass sleeve	Ice to 4 C	28 days
Total kjeldahl nitrogen (TKN)	EPA 351.3			28 days
Water Analyses (decontamination water, storm water retention pond)				
Explosives	8330 (full list)	2 1-L amber glass jars	Ice to 4 C	7 days; analyzed within 40 days after extraction
total suspended solids (TSS)	EPA 160.2	1 500-mL plastic	Ice to 4 C	7 days
chemical oxygen demand (COD)	EPA 410.1, 410.2	1 250-mL plastic	H2SO4 to pH < 2, Ice to 4 C	28 days
biochemical oxygen demand (5-day BOD)	EPA 405.1	1 1-L amber glass jar	Ice to 4 C	**48 hours**
Oil and Grease	EPA 413.1	2 1-L amber glass jars	HCL to pH < 2; Ice to 4 C	28 days
Nitrate	EPA 300.0	1 500-mL plastic	Ice to 4 C	**48 hours**
Ammonia-N	EPA 350.2	1 500-mL plastic	H2SO4 to pH < 2; Ice to 4 C	28 days
Additional Water Analyses (blanks)				
Volatile Organics	8240: methylene chloride, acetone, xylene, toluene, methyl ethyl ketone, methyl isobutyl ketone, benzene, carbon tetrachloride, chloroform, chlorobenzene, 1,2-DCA, 1,1-DCE, vinyl chloride tetrachloroethene, trichloroethene,	3 40-mL glass vials	HCL to pH < 2; Ice to 4 C	14 days

TABLE 4-7. ANALYTICAL METHOD REQUIREMENTS

Analyte	Analytical Method[1]	Sample Container[2]	Preservation	Holding Time
Explosives	8330 (full list)	2 1-L amber glass jars	Ice to 4 °C	7 days; analyze within 40 days after extraction
RCRA metals	6010	1 500-mL plastic	HNO3 to pH < 2; Ice to 4 °C	180 days
TOC	EPA 415.1	1 500-mL plastic	H2SO4 to pH < 2, Ice to 4 °C	28 days
TKN	EPA 351.3			28 days

Notes:

Note holding times in **bold-face** type.

1. SW-846 methods (latest revision) will be used unless otherwise noted.
2. Container size may vary from lab to lab. Aliquots from several analyses may be obtained from one container, as directed by the laboratory.

TABLE 4-8. NPDES DISCHARGE LIMITATIONS[1]

Parameter	Monthly Limit - Average (mg/L)	Monthly Limit - Maximum (mg/L)
Total Suspended Solids (TSS)	10	15
Chemical Oxygen Demand (COD)	10	15
Biochemical Oxygen Demand (BOD)	10	15
Oil and Grease	26	52
Nitrate	N/A	90
Ammonia-N	3	6
pH (field measured)[2]	between 5.0 and 11.0 units	

Notes:

1. These values were obtained from the NPDES permit no. IN 0021539 (highest levels permitted).
2. Field pH will be measured using an Orion Model pH meter (290). Calibration data and model information will be recorded in the field logbook, including results.

N/A = not available

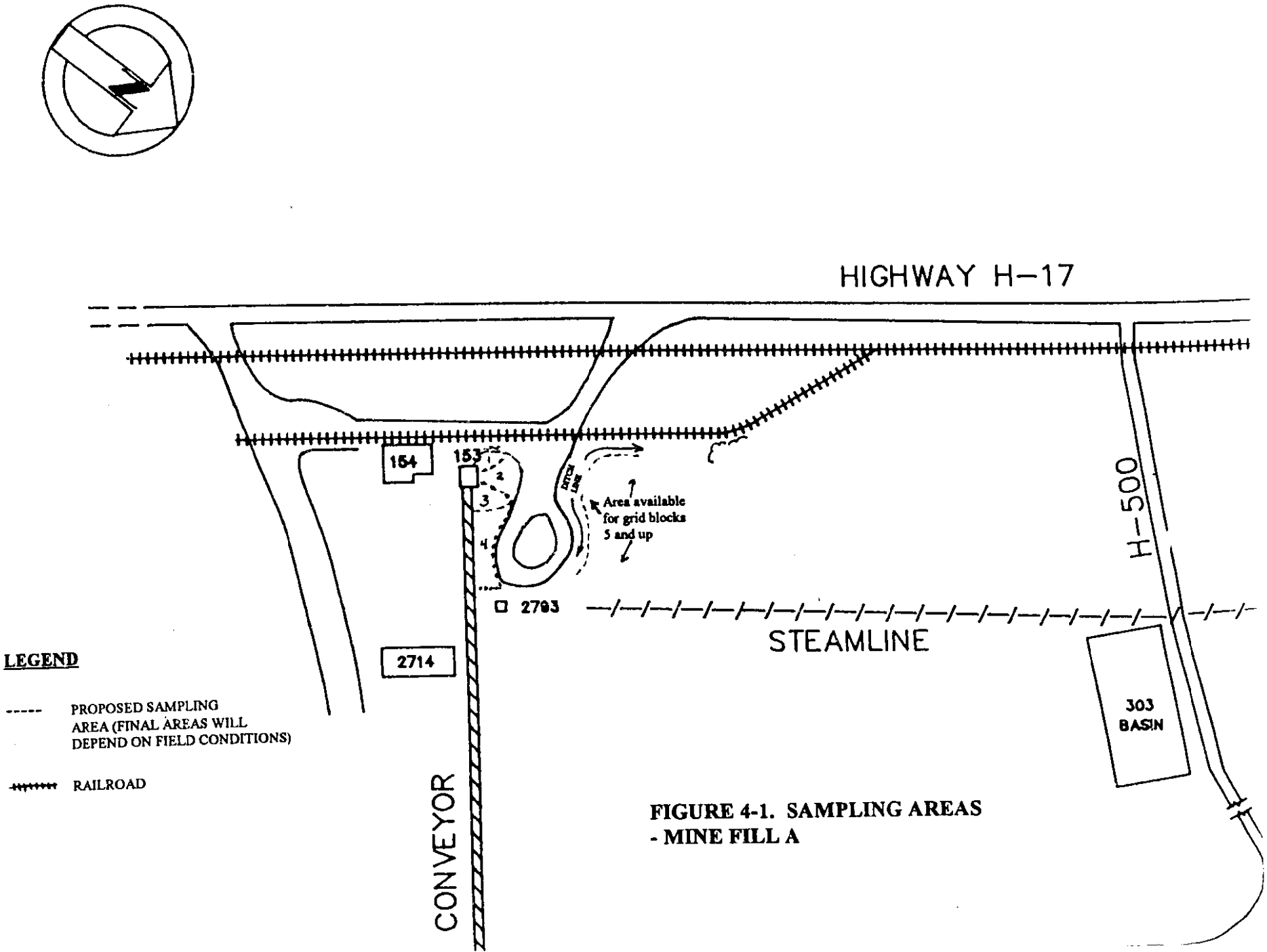


FIGURE 4-1. SAMPLING AREAS - MINE FILL A

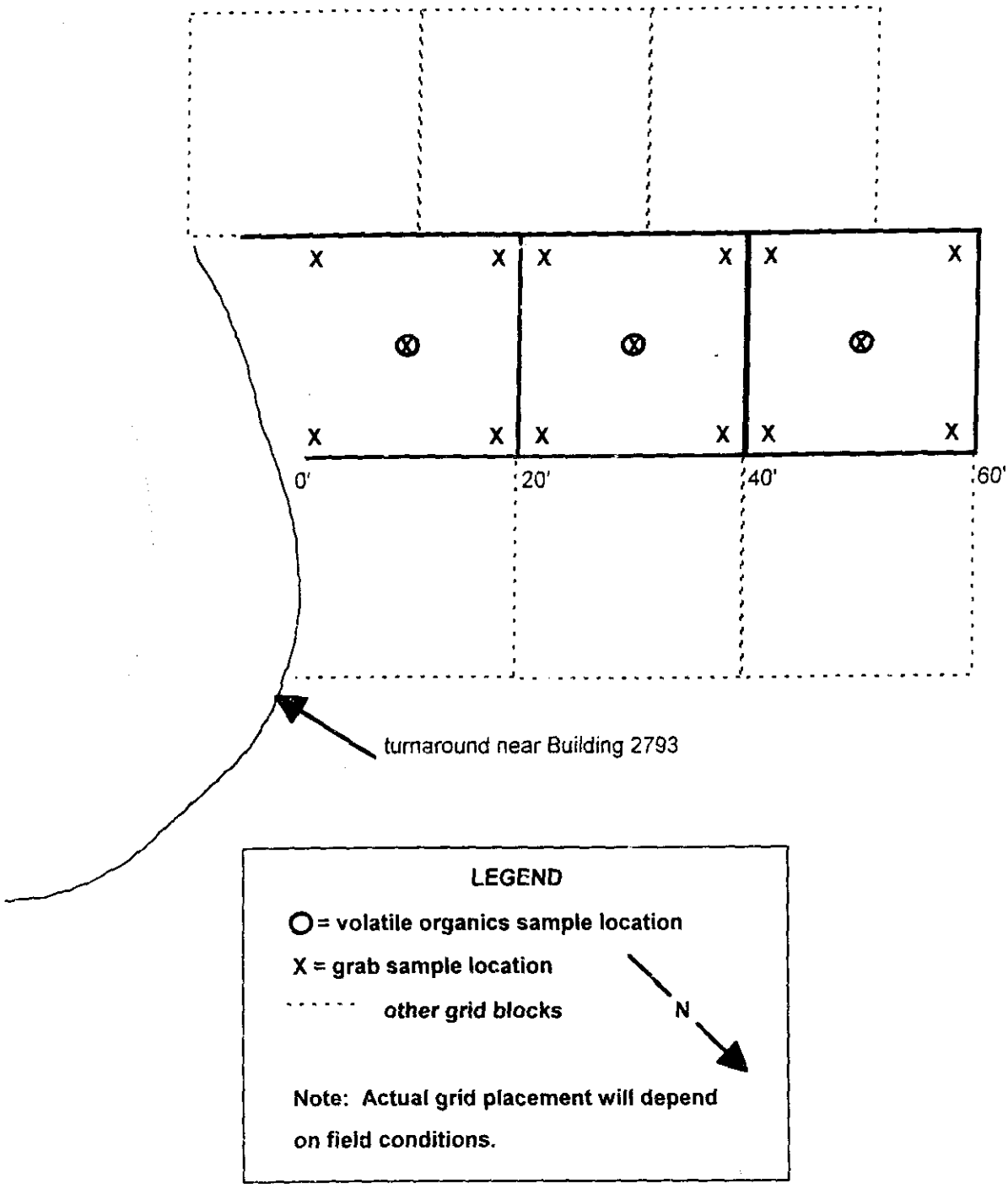
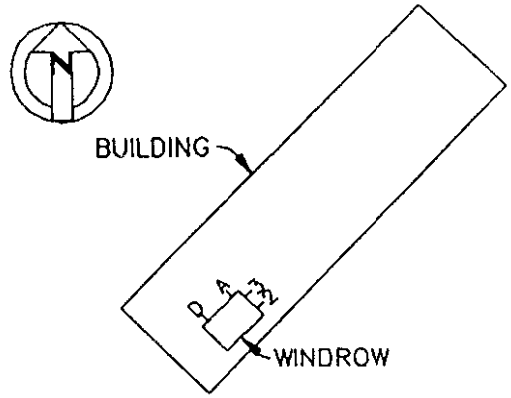
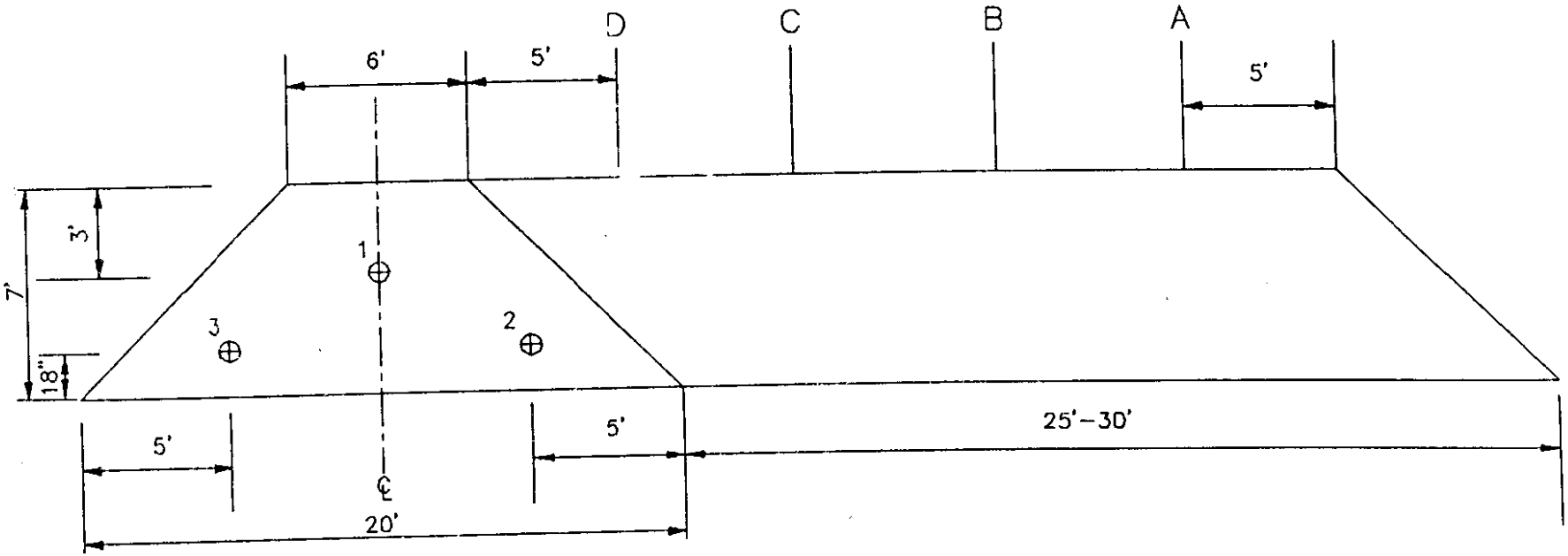


FIGURE 4-2. EXAMPLE GRID LAYOUT - BUILDING 153/2793 VICINITY



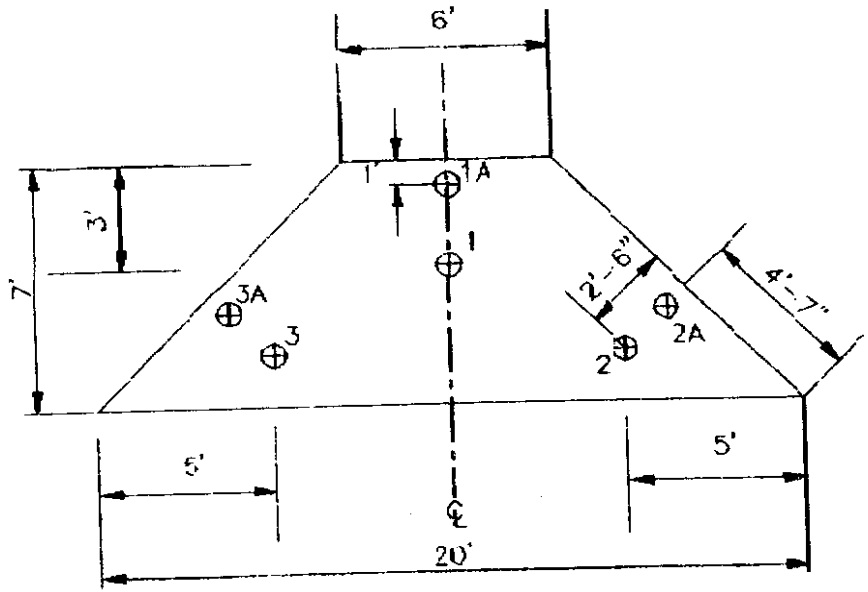
KEY PLAN



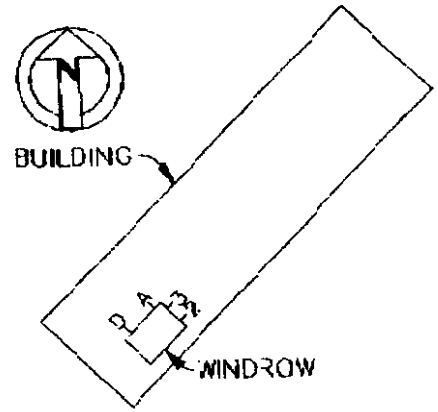
PILOT SCALE
WINDROW SAMPLE LOCATIONS

MORRISON KNUDSEN CORP.	
Drawing FIGURE 4-3 A	Rev. 0
Date 10/10/96	Sheet 1

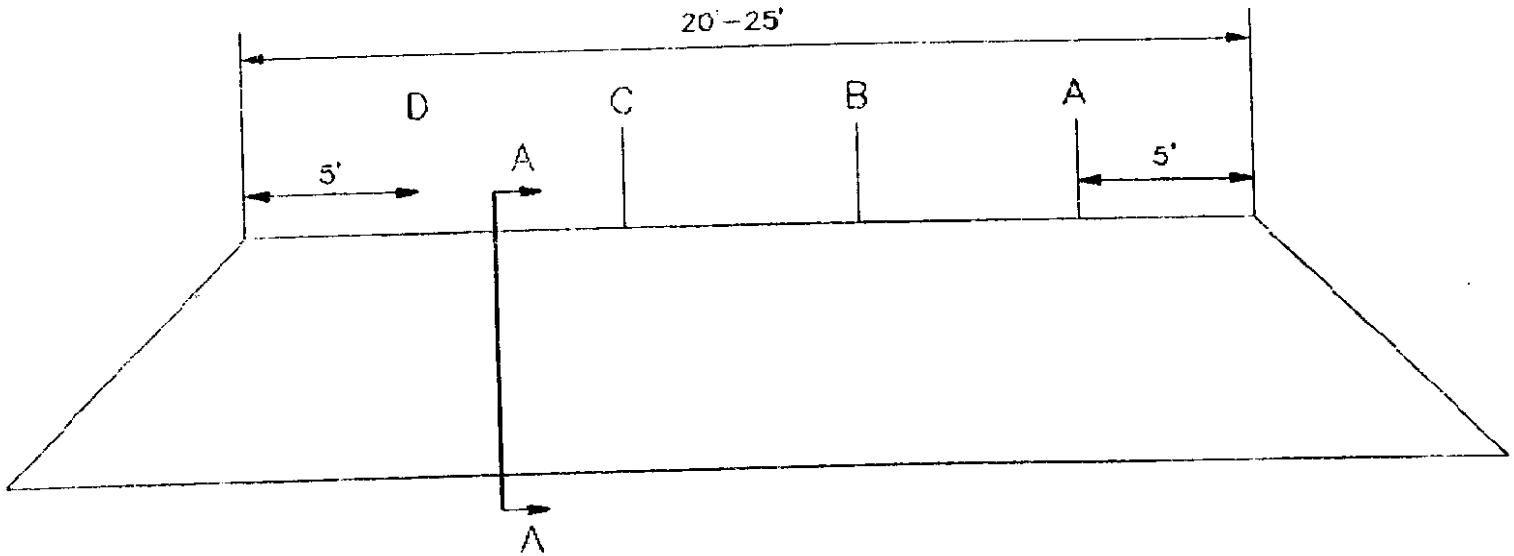
NOTE: ALL SAMPLE NUMBERS FOLLOWED BY AN "A" WILL BE TAKEN AT 1' BELOW THE SURFACE.



SECTION A–A



KEY PLAN



PILOT SCALE
WINDROW SAMPLE LOCATIONS

MORRISON KNUDSEN CORP.	
Drawing FIGURE 4–3	Rev. 1
Date 5/28/97	Sheet 1

DRAWING NAME: F:\JOBS\VORANE\PEGASUS.DWG

SECTION 5
CUSTODY PROCEDURES

Custody is one of several factors which is necessary for the admissibility of environmental data as evidence in a court of law. Custody procedures help to satisfy the two major requirements for admissibility: relevance and authenticity. Sample custody is addressed in three parts: field sample collection, laboratory analysis, and final evidence files. Final evidence files, including all originals of laboratory reports and purge files, are maintained under document control in a secure area.

A sample or evidence file is under a person's custody if:

- The item is in actual possession of a person; or
- The item is in the view of the person after being in actual possession of the person; or
- The item was in actual physical possession but is locked up to prevent tampering; or
- The item is in a designated and identified secure area.

5.1 FIELD CUSTODY PROCEDURES

Field logbooks will provide the means of recording data collecting activities. As such, entries will be described in as much detail as possible so that persons going to the facility could reconstruct a particular situation without reliance on memory.

Field logbooks will be bound field survey books or notebooks. Logbooks will be assigned to field personnel, but will be stored in the document control center when not in use. Each logbook will be identified by the project-specific document number.

The title page of each logbook will contain the following:

- Person or persons to whom the logbook is assigned,
- Logbook number,
- Project name,
- Subject (Sampling, Waste Management, etc.)
- Site/SWMU name,
- Project start date, and
- End date.

Entries into the logbook will contain a variety of information. At the beginning of each entry, the date, start time, weather, names of all sampling team members present, level of personal protection being used, and the signature of the person making the entry will be entered. The names of visitors to the site, field sampling or investigation team personnel and the purpose of their visit will also be recorded in the field logbook.

Measurements made and samples collected will be recorded. All entries will be made in indelible ink, signed, and dated and no erasures will be made. If an incorrect entry is made, the information will be crossed out with a single strike mark which is signed and dated by the sampler. Whenever a sample is collected or a measurement is made, a detailed description of the sampling location shall be recorded. All sample locations will be surveyed, with respect to permanent features. The number of the photographs taken at Mine Fill A or the Biofacility, if any, will also be noted. All equipment used to make measurements will be identified, along with the date and results of calibration.

Samples will be collected following the sampling procedures documented in Section 4.0. The equipment used to collect samples will be noted, along with the time of sampling, sample description, depth at which the sample was collected, volume and number of containers. Sample identification numbers will be assigned prior to sample collection. Field duplicate samples, which will receive an entirely separate sample identification number, will be noted under sample description. A detailed description of assigning sample identification numbers is provided in Section 5.1.2.

The sample packaging and shipment procedures summarized in Section 5.1.4 will ensure that the samples will arrive at the laboratory with the chain of custody intact. Chain-of-custody procedures are summarized in Section 5.1.3. An example of a field custody document is presented in Figure 5-1.

5.1.1 Field Documentation

Field notebooks will be used to document all field activities. Information to be entered in the field notebook includes at a minimum:

- Project name and location
- Name and address/phone number of field contact
- Individuals on site
- Weather conditions
- Sample locations and depths (include sketch)
- Date and time of each activity
- Results of air monitoring and personal protective equipment levels

- Sample identification
- Date and time of sample collection
- Requested analyses, type and number of sample containers, preservative
- Results of field screening measurements
- Type of sample collected (pond water, decontamination water, soil, grab, etc.)

Notes will be written on sequentially numbered pages with indelible ink. Each page will be initialed and dated when full. Corrections to logbook entries will be made by lining through incorrect entries with a single line and initialing and dating the strike out. At the end of the day, any unused space at the bottom of the last page will be "x-ed" out, initialed and dated.

5.1.2 Sample Identification

Samples will be labeled, preserved, and properly packaged for shipment to the analytical laboratory. Information on the sample label will include, at a minimum:

- Sample identification number
- Site Name
- Analytical method **and type of preservative**
- Date and time of sampling

Sample identification numbers will be used to provide a tracking procedure allowing information on a particular sample location to be easily and accurately retrieved. This system also ensures that each sample is unique and not confused with any other sample. The Project Manager will maintain a complete list of sample numbers.

Each sample name will consist of three initials "CRA" (standing for "CRANE"), followed by a series of eight digits describing the sample. A generic sample name would be:

CRA/XXXXXXXX

Sample Site

Pilot Scale samples will be collected at either Mine Fill A or the Biofacility:

- MPS - Mine Fill A Pilot Scale
- BPS - Biofacility

This identifier will be placed directly after "CRA", as follows:

CRA/MPSXXXXX
CRA/BPSXXXXX

Mine Fill A Samples

Samples collected at Mine Fill A will consist of one of the following types:

- ICS - Initial Characterization Soil (sample) (pre-excavation - Task 1)
- PES - Post-Excavation Soil (sample) (Task 5)

This three-letter identifier will be placed after "MPS." The sample number will occupy the last two digits of the sample identification number:

CRA/MPSICS01
CRA/MPSPES01

Biofacility Samples - Non-Compost Samples

Samples collected at the Biofacility will consist of one of the following types:

- RPW - Retention Pond Water (Task 3)
- WWM - Waste Water from Mine Fill A operations (Task 2)
- WWB - Waste Water from Biofacility operations (Task 2)
- WSB - Waste Sump Solids from the Biofacility (Task 2)
- AMS - Amendment Sample (not yet mixed with soil) (Task 4)
- TWS - Treatment Water Sample (water used during treatment to enhance moisture content) (Task 4)

This three-letter identifier will be placed after the term "BPS." The sample number will occupy the last two digits of the sample identification number:

- CRA/BPSRPW01
- CRA/BPSWWM01

Biofacility Samples - Compost Samples (Task 4)

Compost samples collected during Pilot Scale operations will be identified using the following format:

CRA/BPSXXXXX

-The first "X" will represent the windrow number (1-8)
-The second and third "X's" will represent the "Day" sampled (~~00, 07, 14, 21, 28~~ **00, 05, 10, 20, 30, 40, 60**, and so on...)
-The fourth "X" will represent the cross-section letter (A, B, C, or D)
-The fifth and final "X" will represent the sample number within the cross-section (1, 2, or 3)

Thus, an example compost sample identification number is:

CRA/BPS2~~14~~**20**A2, which represents a compost sample collected from pile #2, on Day ~~14~~ **20** of the process, at cross-section A, location #2.

QC Sample Identifiers

The following QC identification symbols will be used for sampling:

- T - Trip Blank
- A - Field Blank A
- B - Field Blank B
- R - Rinsate Blank

When a QC sample is collected, the appropriate letter code will be used:

CRA/BPSTXXXX
CRA/MPSAXXXX

The last four digits will be the date the sample was collected. For example:

CRA/BPSR1010, represents a rinsate blank collected on October 10th, during Pilot Scale operations at the Biofacility.

If more than one type blank is collected in one day, an additional letter (A, B, C and so on...) will be added after the QC code. For example:

CRA/MPSRA1010, represents the first rinsate blank collected on October 10th, during Pilot Scale operations at Mine Fill A.

CRA/MPARB1010, represents the second rinsate blank collected on October 10th during Pilot Scale operations at Mine Fill A.

Field Duplicates

Field duplicate samples will be labeled using the same protocol as regular samples. However, the field duplicate will labeled as the next consecutive sample collected, even though it was collected from the same spot as the regular sample. For example, a soil sample was collected from the excavation area (Task 5). A field duplicate sample was also collected. The regular sample would be labeled as:

CRA/MPSPES08

The field duplicate would be labeled as:

CRA/MPSPES09

For compost pile field duplicates, the last two digits will be replaced with "FD." For example, the regular sample collected from windrow/pile #2, on Day ~~14~~ **20**, from cross-section A, location #2, would be:

CRA/BPS2~~14~~**20**A2

The field duplicate, in order to blind the lab to its location, would be labeled:

CRA/BPS2~~14~~**20**FD

Document all field duplicates with their associated samples and locations in the field logbook.

All information related to the sample matrix, location, and depth shall be recorded in the log book by the sampling team. The Field Engineer is responsible for maintaining a map showing the locations of each sample and keeping a list of which samples are submitted to the laboratory for QC purposes.

The sample number is entered on sample labels and chain-of-custody forms. All sample identification information will be documented in the sampler's field logbook.

Samples will be placed in shipping containers for shipment to the laboratory. Custody seals will be affixed to the shipping containers. Information on the custody seal will include the date when the container was sealed and the signature of the sampler or relinquisher.

5.1.3 Chain of Custody Procedures

To maintain and document sample possession, chain-of-custody (COC) procedures must be implemented. A COC form will be used to record pertinent information for each sample and the individuals responsible for the sample collection, shipment and receipt. **In addition to sample labels, sample tags will be appropriately labelled and attached to sample containers.**

The subcontracted laboratories will not accept samples collected by MK for analysis without a completed COC form. The COC form shall be initiated in the field by the person collecting the sample, for each sample. Each chain-of-custody form will be assigned a unique number. If sampling is conducted at the Biofacility and Mine Fill A, two COC forms, each with a unique COC number, will be used: one for Mine Fill A and one for the Biofacility. Each sample will be assigned a unique identification number, as described in Section 5.1.2, and that number will be entered on the COC form. In order to be complete, project identification, date and time of sample collection, sample location, requested analyses, turnaround time, and any special instructions must be included on the COC, along with each sample identification. Custody will be relinquished by using the signature blocks at the bottom of the custody form. The original COC form will accompany the shipment, and a copy will be retained by the PM or designee. A signed COC form will be obtained from the laboratory custodian after the samples have been received and their condition checked. For samples shipped by commercial carrier, the air bill or bill of lading will serve as an extension of the COC form.

Upon receipt in the laboratory, all samples will proceed through an orderly processing sequence (as defined in the laboratory QA/QC Plan) specifically designed to ensure continuous integrity of both the sample and other information pertinent to the analysis. All samples will be carefully checked and verified for proper COC records, preservation, presence of broken or leaking sample containers, proper label identification, and any associated discrepancies. If any samples arrive leaking or broken, or the custody seal on the shipment coolers is not intact, the Project Manager and Project Chemist will be notified of the problem(s) immediately. If no discrepancies are identified, the sample COC record will be signed, and the samples will subsequently be assigned a unique laboratory identification number by the laboratory

for tracking and filing. The laboratory QA system and the use of an internal COC procedure will ensure that the samples are appropriately tracked from receipt through completion of the analytical process.

5.1.4 Handling and Shipping

All samples will be collected in the appropriate containers for each analysis, as detailed in Table 4-7. All sample labels used on sample containers will include, at a minimum, a sample identification number, the date and time of sample collection, site name, and the analytical method requested. The label will adhere to the container, and the writing on it will be in indelible ink. The label will be secondarily affixed to the container with clear adhesive tape completely covering the label. A waterproof metal or equivalent strength plastic cooler will be used for packaging and shipping samples. Samples will be individually packed with bubble wrap or other cushioning material to prevent breakage during transport. Ice will be placed in the coolers with the samples to maintain shipping temperature at 4 ±2 degrees Celsius. Include a temperature blank with each cooler. The COC will be placed in plastic and affixed to the underside of the cooler lid. Custody seals will be affixed to the front and back of each cooler and covered with clear adhesive tape.

Samples packaging and shipping will vary depending upon sample media, contaminant concentration, preservation technique, and sample container. All samples will be packaged and shipped following all state and federal regulations and will conform with DOT requirements.

5.2 LABORATORY CUSTODY PROCEDURES

Laboratory custody procedures for sample receiving and log-in; sample storage and numbering; tracking during sample preparation and analysis; and storage of data are described in the Laboratory QA Plan. The QA Plan contains standard operating procedures which have been deemed proprietary by the Laboratory. Laboratory custody procedures are provided in Appendix D.

5.3 FINAL EVIDENCE FILES

The final evidence file will be the central repository for all documents which constitute evidence relevant to sampling and analysis activities as described in this QAPP. MK is the custodian of the evidence file and maintains the contents of evidence files for the Interim Measure, including all relevant records, reports, logs, field notebooks, pictures, subcontractor reports and data reviews in a secured, limited access area, under custody of the Morrison Knudsen.

The final evidence file will include at a minimum:

- Field logbooks;
- Field data and data deliverables;
- Photographs;
- Drawings;
- Laboratory data deliverables;
- Data validation reports;
- Data assessment reports;
- Progress reports, QA reports, interim project reports, etc.; and
- All custody documentation (tags, forms, air bills, etc.)

Prior to final disposition, items from the final evidence file will be provided, upon request and approval by the Navy, to the USEPA, for use in risk assessment or other related activities at NSWC Crane.

FIGURE 5-1

CHAIN OF CUSTODY RECORD

MORRISON KNUDSEN CORPORATION
720 Park Blvd., P.O. Box 73
Boise, Idaho 83729
(208) 386-5000

Project No.:

Project Name:

Samplers: (Signature)
Sampler: (Print)

Sample Type	Sampling Point Description	Sample Date	Time	Sample I.D. Number	Split Samples Yes	Split Samples No	Analysis Required	Remarks

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:	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	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:

SECTION 6
CALIBRATION PROCEDURES AND FREQUENCY

This section describes the calibration procedures and the frequency at which these procedures will be performed for both field and laboratory instruments.

6.1 FIELD INSTRUMENT CALIBRATION

Field instruments will be calibrated as prescribed by the owner's manual. Instruments, including the PID, pH meter and temperature/oxygen probe will be calibrated daily, in the morning, prior to use. Calibration data will be documented in the field logbook. A post-calibration may be completed at the end of the day as a check on instrument function. Calibration procedures for field test display instruments and the PID are provided as Appendix E, and are summarized below.

pH Meter Calibration (Horiba Twin B-213, the following procedure will be adjusted as needed to meet the requirements of this device)

The pH meter will be calibrated with standard buffer solutions before being taken to the field. In the field, the meter will be calibrated daily with two buffer solutions before use. The range of the buffer solutions will be at least three or more pH units apart and will bracket the expected pH of the sample being measured.

- Ensure the temperature of the buffer and the sample are the same.
- Connect pH electrode into pH meter and turn on the pH meter.
- Set temperature setting based on the temperature of the buffer; place electrode in first buffer solution.
- After reading has stabilized, adjust "CALIB" (or equivalent) knob to display correct value.
- Repeat procedure for second buffer solution.
- Place pH electrode in the sample and record the pH as displayed.
- Remove pH electrode from sample and rinse off with distilled water.
- Recalibrate the pH meter every time it is turned off and turned back on, or if it starts giving erratic results.

PID (MicroTIP MP-1000) (from user's manual)

MicroTIP must be calibrated daily in order to display concentrations in units equivalent to ppm. First a supply of zero air, which contains no ionizable gases or vapors, is used to set MicroTIP's zero point. Then, span gas, containing a known concentration of a photoionizable gas or vapor, is used to set the sensitivity. Usually clean ambient air will be suitable as zero air. Isobutylene at 100 ppm in air is recommended as span gas.

- Connect the supplied regulator to the span gas cylinder. Hand tighten the fittings. Observe proper handling techniques for all gases.
- Open the gas valve on the gas bag by turning the valve stem fully counterclockwise.
- Attach the nut to the regulator. Hand tighten the fittings.
- Turn the regulator knob counterclockwise about half a turn to start the flow of gas.
- Fill the gas bag about half full and then close the regulator fully clockwise to turn off the flow of gas.
- Disconnect the bag from the adapter and empty it. Flush the bag a few times with the span gas and then fill it.
- Close the gas bag by turning the valve clockwise.
- Press SETUP and select the desired Cal memory with the arrow keys and press ENTER. Press EXIT to return to the normal display.
- Press CAL and enter the desired response factor. Use a table of response factors available in the User's Manual to find the factor for the compound of interest. If the compound is not in the table or you are not looking for one specific compound, enter 1.00. The concentration detected by MicroTIP will be multiplied by the response factor before it is displayed and logged.
- Expose MicroTIP to zero air. Press ENTER and MicroTIP sets its zero point.
- MicroTIP then asks for the span gas concentration. Enter the known span gas concentration and then connect the span gas bag adapter to the inlet.
- Press ENTER and MicroTIP sets its sensitivity.
- When MicroTIP's display reverts to normal, MicroTIP is calibrated and ready for use. Remove the span gas bag from the inlet.

Temperature/Oxygen Probes (Reotemp)

If the monitor/probe has not been used for 15 minutes, it will automatically shut down.

To turn on the monitor, press any key on the front panel. Press the far right hand key until you reach AUTOCAL. Press RUN. The instrument will sample room air and set a reading of 20.9% oxygen automatically.

6.2 LABORATORY INSTRUMENT CALIBRATION

Laboratory instruments will be calibrated at prescribed intervals and/or as part of daily use. Frequency will be based upon the analytical method, type of equipment, inherent stability, and manufacturer's recommendations. Equipment will be calibrated, whenever possible, using reference standards having known relationships to nationally recognized standards or accepted values of physical constants. If national standards do not exist, the basis for calibration will be documented.

The analytical laboratory will be responsible for the maintenance of laboratory instruments and equipment. Instruments, and the measurements made as part of the analytical methodology, will be as specified in the method, without modification. The laboratory's quality assurance (QA) program ensures that only trained personnel perform routine maintenance on all major instruments and that repairs are performed by trained laboratory personnel or service technicians employed by the instrument manufacturer or representative. Instrument maintenance will be appropriately documented through the use of instrument logs which will be included in the laboratory project file.

General calibration information is provided in Section 1 of Appendix F. SOP calibration information is summarized in Table 6-1.

TABLE 6-1. CALIBRATION CRITERIA - LABORATORY METHODS				
Parameter	**SOP Number**	**Table (if applicable)**	**Page Number**	
			Appendix F-	**SOP page number**
Volatile Organic Compounds (Method 8240)	MS300	Table 3 - Tune Criteria	43	12
		Table 8 - Criteria for CCCs	45	14
		Table 9 - Criteria for SPCCs	45	14
		Table 13 - equations for RFs	49	18
Explosives	GC200	Section VII.C.	71	7
Metals	MT913B	Section IX (quality control)	91	4
Mercury	MT310B	Section IV.C	105	6
TOC - soil	IN045S	Section VII.B	117	2
TOC - water	IN045W	Section VII	123	2
TKN, TSS, COD, BOD, ammonia-N	(none required)			
Oil and grease	IN200, IN205	Section VII.B	252	3
Nitrate	IN030B	Section VIII	262	4

SECTION 7
ANALYTICAL PROCEDURES

This section describes the analytical procedures for all samples collected for analysis.

7.1 FIELD ANALYTICAL PROCEDURES

Operation and QC procedures for the PID, pH meter and temperature/oxygen probes are provided in Appendix E.

7.2 LABORATORY ANALYTICAL PROCEDURES

The laboratory will implement project required Standard Operating Procedures (SOPs). These laboratory SOPs for sample preparation, cleanup and analysis are based on the latest SW-846 Revision (U.S. EPA, January 1995) and EPA methods for methods of chemical analysis of water and waste (USEPA, 1983) . These SOPs which provide sufficient details and are specific to this RCRA Corrective Action Interim Measure are provided in Appendix F and summarized in Table 7-1.

The site samples for volatile organic compounds analysis (VOA) may be screened in the laboratory, as described in the VOA SOP and shall be analyzed, either as low or medium level concentration samples, or as a series of dilutions in order to cover the expected concentration range of the site-specific compounds of interest.

The documentation of appropriate method validation for the project target compounds will be submitted in the Pilot Scale Completion Report. It includes the criteria for acceptance, rejection or qualification of data.

7.2.1 List of Project Target Compounds and Laboratory Reporting Limits

A listing of project target compounds, soils cleanup levels, and analytical methods to be used for samples collected during the project are summarized in Tables 1-3, 1-4, and 1-5. Reporting limits are provided in Table 1-4. Other analytical results will be reported using standard method PQLs as reporting limits. Standard method detection limits for each method in Table 1-5 are provided in Appendix B.

7.2.2 List of Associated QC Samples

The analytical methods and laboratory SOPs include a QC section which address the minimum QC requirements for the analysis of specific analyte groups. Compounds to be spiked will be representative of the target compounds of interest. Section 7.1 of the Sampling and Chemical Analysis Quality Assurance Requirements for the Navy Installation Restoration Program (NEESA, 1988) contains a complete listing of the associated QC samples required for this project. This document is provided as Appendix H.

TABLE 7-1. SUMMARY OF SOPs		
Parameter(s)	**SOP Number**	**Analytical Method**
SOIL		
Volatile Organics	MS300	8240
Explosives	GC200	8330
Arsenic	MT913B	6010
Barium	MT913B	6010
Cadmium	MT913B	6010
Chromium	MT913B	6010
Lead	MT913B	6010
Mercury	MT310B	7471
Selenium	MT913B	6010
Silver	MT913B	6010
TCLP - metals	HW700/HW710, MT913B, MT310B	1311, 6010
TOC	IN045S	9060
TKN	IN170	EPA 351.3
WATER		
Volatile Organics	MS300	8240
Explosives	GC200	8330
Arsenic	MT913B	6010
Barium	MT913B	6010
Cadmium	MT913B	6010
Chromium	MT913B	6010
Lead	MT913B	6010
Mercury	MT310B	7470
Selenium	MT913B	6010
Silver	MT913B	6010
TOC	IN045W	EPA 415.1

TABLE 7-1. SUMMARY OF SOPs

Parameter(s)	SOP Number	Analytical Method
TKN	IN170	EPA 351.3
total suspended solids (TSS)	IN275	EPA 160.2
chemical oxygen demand (COD)	IN090D, IN090C	EPA 410.2
Biochemical oxygen demand (5-day BOD)	IN025	EPA 405.1
Oil and Grease	IN200, IN205	EPA 413.1
Nitrate	IN030, IN055, IN135, IN185, IN195, IN255, IN295	EPA 300.0
Ammonia-N	IN020	EPA 350.2

Notes:

1. SOPs are laboratory confidential. SOPs are provided by Southwest Laboratories (Broken Arrow, Oklahoma)

SECTION 8
INTERNAL QUALITY CONTROL CHECKS

8.1 FIELD QUALITY CONTROL CHECKS

Assessment of field sampling precision and bias will be made by collecting field duplicates and field blanks for laboratory analysis. Collection of the samples will be in accordance with applicable procedures in Section 4.0. Recommended QC checks for other field equipment are provided in Appendix E.

8.2 LABORATORY QUALITY CONTROL CHECKS

The laboratory QC program shall ensure the reliability and validity of the analysis performed at the laboratory. All analytical procedures are documented in writing as SOPs, and each SOP includes a QC section which addresses the minimum QC requirements for the procedure. The internal quality control checks might differ slightly for each individual procedure but in general the QC requirements include the following:

- Method blanks;
- Reagent/preparation blanks (applicable to inorganic analysis);
- Instrument blanks;
- Matrix spikes/matrix spike duplicates;
- Surrogate spikes;
- Analytical spikes (Graphite furnace);
- Laboratory duplicates;
- Laboratory control standards;
- Internal standard areas for GC/MS analysis; control limits;
- Mass tuning for GC/MS analysis; and
- Second, dissimilar column confirmation for GC/EC analysis.

A description of the specific QC requirements for this facility investigation and the frequency of audit is described in laboratory SOPs. These documents are provided in Appendix F and are considered laboratory confidential. QC criteria are also included in Section 3 and Section 6.

SECTION 9
LABORATORY DATA REDUCTION, VALIDATION AND REPORTING

All data generated during field activities or by the laboratory shall be reduced and validated prior to submittal to MK. No data shall be disseminated by the laboratory until it has been subjected to these procedures which are summarized in subsections below.

9.1 DATA REDUCTION

9.1.1 Field Data Reduction Procedures

Field data reduction procedures will be minimal in scope compared to those implemented in the laboratory setting. Only direct read instrumentation will be employed in the field. Data will be recorded in field logbooks immediately after measurements are taken. If errors are made, results will be legibly crossed out, initialed and dated by the field sampler, and corrected in a space adjacent to the original (erroneous) entry.

Because the use of field instrumentation such as a mobile gas chromatograph will not be used, there will be no further need for assuring that field data has been reduced properly through the use of formulas or interpretation of raw data printouts.

9.1.2 Laboratory Data Reduction Procedures

The following protocol will be used for laboratory data reduction. All raw analytical data will be recorded in numerically identified laboratory notebooks or equivalent forms. These notebooks will be issued only by the Laboratory QA Manager. Data are recorded in this notebook along with other pertinent information, such as the sample identification number and the sample tag number. Other details will also be recorded in the lab notebook, such as the analytical method used (SOP#), name of analyst, the date of analysis, matrix sampled, reagent concentrations, instrument settings, and the raw data. Each page of the notebook shall be signed and dated by the analyst. Copies of any strip chart printouts (such as gas chromatograms) will be maintained on file. Periodic review of these notebooks by the Lab QA Manager will be completed prior to final data reporting. (Records of notebook entry inspections are maintained by the Lab QA Manager.)

For this project, the equations that will be employed in reducing data are presented in Section 12. Such formulas make pertinent allowances for matrix type. All calculations will be checked by the Organic Section supervisor at the conclusion of each operating

day. Errors will be noted, corrections made, with the original notations crossed out legibly. Analytical results for soil samples shall be calculated and reported on a dry weight basis, and TCLP results will not be matrix spike recovery-corrected.

Quality control data (e.g. laboratory duplicates, surrogates, matrix spikes, and matrix spike duplicates) will be compared to the method acceptance criteria. Data considered to be acceptable will be entered into the laboratory computer system. Data summaries will be sent to the Laboratory QA Manager for review. If approved, data are logged into the project database format. Unacceptable data shall be appropriately qualified in the project report. Case narratives will be prepared which will include information concerning data that fell outside acceptance limits, and any other anomalous conditions encountered during sample analysis. After the Lab QA Manager approves these data, they are considered ready for third party data validation.

9.2 DATA VALIDATION

Data validation procedures shall be performed for both field and laboratory operations as described below:

9.2.1 Procedures Used to Evaluate Field Data

Procedures to evaluate field data for this project primarily include checking for transcription errors and review of field log books, on the part of field crew members. This task will be the responsibility of the SQCS, who will otherwise not participate in making any of the field measurements, or in adding notes, data or other information to the log book.

9.2.2 Procedures to Validate Laboratory Data

All data will be verified by reviewing analytical laboratory data and associated documentation, including Chain-of-Custody records, sample preservation records, analytical holding times, sample data, and reporting requirements. Verification is a preliminary analytical review to ensure that the samples are preserved, shipped, maintained, and analyzed in accordance with established data quality objectives and standard operation procedures. Data will be reviewed for timeliness of the report, chain-of-custody compliance, accurate sample identifications, and compliance to extraction and analytical holding times. Quality control performed by the laboratory, including method blanks, standard and sample matrix spikes, replicate samples, and surrogate spikes, will be evaluated so that the completeness and correctness of the data report can be addressed.

Following verification, 10 percent of the confirmation sample data for the compost samples will be validated according to NEESA data validation guidelines. Qualifiers will be applied to the data based on results of the validation effort. Data validation of laboratory data will follow accepted criteria defined by Section 7.3.2 of NEESA 20.2-047B. Problems or discrepancies will be discussed with the laboratory by the data validator for resolution and/or documentation.

9.3 DATA REPORTING

Data reporting procedures shall be carried out for field and laboratory operations as indicated below:

9.3.1 Field Data Reporting

Field data reporting shall be conducted principally through the transmission of report sheets containing tabulated results of all measurements made in the field, and documentation of all field calibration activities.

9.3.2 Laboratory Data Reporting

The task of reporting laboratory data begins after the validation activity has been concluded. The Laboratory QA Manager must perform a final review of the report summaries and case narratives to determine whether the report meets project requirements. In addition to the record of chain-of-custody, the report format shall consist of the following:

1. Case Narrative:

    a. Date of issuance;

    b. Laboratory analysis performed;

    c. Any deviations from intended analytical strategy;

    d. Laboratory batch number;

    e. Numbers of samples and respective matrices;

f. Quality control procedures utilized and also references to the acceptance criteria;

g. Laboratory report contents;

h. Project name and number;

I. Condition of samples 'as-received' including cooler temperature;

j. Discussion of whether or not sample holding times were met;

k. Discussion of technical problems or other observations which may have created analytical difficulties;

i. Discussion of any laboratory quality control checks which failed to meet project criteria; and

m. Signature of the Laboratory QA Manager.

2. Chemistry Data Package

a. Case narrative for each analyzed batch of samples;

b. Summary page indicating dates of analyses for samples and laboratory quality control checks;

c. Cross referencing of laboratory sample number to project sample identification numbers;

d. Data qualifiers to be used should be adequately described;

e. Sample preparation and analyses for samples ;

f. Sample results;

g. Raw data for sample results and laboratory quality control samples;

h. Results of (dated) initial and continuing calibration checks, and GC/MS tuning results;

i. Matrix spike and matrix spike duplicate recoveries, laboratory control samples, method blank results, calibration check compounds, and system performance check compound results; and

j. Labeled (and dated) chromatograms/spectra of sample results and laboratory quality control checks.

The data package submitted will be a "CLP-like" data package consisting of all the information presented in a CLP data package. A list of deliverables is provided in Table 9-1.

TABLE 9-1. DATA SET DELIVERABLES
(use CLP-forms as listed below)

Contaminants	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 Form 1. Sample results using standard data flags. Raw data must be submitted with data deliverable package.	Form 1, Sample chromatograms and mass spectra
	Surrogate recovery from samples reported on Form 2. For volatiles by GC, the names of surrogates should be changed to reflect the surrogate used.	Form 2
	Matrix spike/spike duplicate data; one spike and spike duplicate per QC lot of samples of similar matrix reported on Form 3.	Form 3, sample chromatograms and mass spectra
	Method blank data reported on Form 4.	Form 4, sample chromatograms and mass spectra
	GC/MS tuning results for volatiles/semi-volatiles.	Form 5

TABLE 9-1. DATA SET DELIVERABLES
(use CLP-forms as listed below)

Contaminants	Method Requirements	Deliverables
	Initial calibration data. Initial calibration data with response factors for GC/HPLC methods.	Form 6, sample chromatograms and mass spectra No form Form 9, sample chromatograms and mass spectra Raw data will be submitted as applicable.
	Continuing calibration GC/MS data For GC and HPLC methods, the response factors and their percent differences from the initial calibration must be reported. Internal Standard Area for volatiles.	Form 7 SLF Form 8
Organics	Second column confirmation shall be done for all GC and HPLC work when compounds are detected above reporting limits.	Form 9 Chromatograms
	Internal standard area for GC/MS analyses shall be supplied.	Form 8
	Instrument Logs	SLF (std. lab form)
	Standard Preparation Logs	SLF
	Sample Preparation Logs	SLF
	Internal COC form	SLF
Metals	Method blank spikes with results and control charts. Run with each batch of samples processed.	Control chart
	Sample results with standard CLP flagging system. Raw data must be submitted with the data deliverable package.	Form 1/Raw Data
	Initial and continuing calibration.	Form 2, Part 1 only, Raw Data
	Blanks 10% frequency	Form 3

TABLE 9-1. DATA SET DELIVERABLES
(use CLP-forms as listed below)

Contaminants	Method Requirements	Deliverables
	Method blank taken through digestion (1 per 20 samples of same matrix).	Form 3
	ICP interference check sample.	Form 4

TABLE 9-1. DATA SET DELIVERABLES
(use CLP-forms as listed below)

Contaminants	Method Requirements	Deliverables
Metals	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 laboratory standards.	Form 5, Part 2 (never used for GFAA work)
	Postdigest spike for GFAA. Duplicates (1 per 20 samples will be split and digested as separate).	Recovery noted on raw data Form 6 samples
	Method blank spike information will be plotted on control chart, one per batch of samples processed.	Control chart
	Standard addition, as required.	Form 8
	Holding times	Form 10
	Instrument Logs	Standard Lab Form (SLF)
	Standard Preparation Logs	SLF
	Sample Preparation Logs	SLF
	Internal COC form	SLF
Wet Chemistry	Method blank spikes with results and control charts. Run with each batch of samples processed.	Report result, control chart
	Method blank 1 per batch	Report result, no format
	Sample results (raw and final data)	Report result, no format
	Matrix spike/spike duplicate or calibration information (Raw data)	Report result if applicable
	Instrument Logs	Standard Lab Form (SLF)
	Standard Preparation Logs	SLF
	Sample Preparation Logs	SLF
	Internal COC form	SLF

TABLE 9-1. DATA SET DELIVERABLES
(use CLP-forms as listed below)

Contaminants	Method Requirements	Deliverables
Wet Chemistry	Calibration check report percent RSD or percent difference from initial calibration	Report percent or percent difference, no format
General Requirements	Method blank spikes with results and control charts. Run with each batch of samples processed.	Report result, control chart
	Internal Chain-of-Custody forms	SLF
	Instrument logs (what samples have been run and under what conditions)	SLF
	Summaries of Extraction Logs	SLF
	Standards Logs	SLF
	Spiking Logs	SLF
	Quantification Reports	SLF
	Percent Moisture Logs	SLF
	Standard Chromatograms	SLF
	Standard Spectra	SLF
	QC Comments (unless already included in the case narrative)	SLF

SECTION 10
PERFORMANCE AND SYSTEM AUDITS

10.1 PERFORMANCE AND SYSTEM AUDITS AND FREQUENCY

To verify compliance with the specific project Quality Assurance Program requirements, MK QA/QC assurance personnel will perform planned and documented surveillances and audits of project activities. These audits will consist of, as appropriate, an evaluation of quality assurance/quality control procedures and the effectiveness of their implementation; an evaluation of work areas and activities; and a review of project documentation. Audits will be performed in accordance with written checklists by trained personnel and, as appropriate, technical specialists. Surveillance results will be formally documented and sent to the PM.

Audits may include, but not be limited to, the following:

- Subcontractor capabilities and performance;
- Field operations and records;
- Laboratory testing and records;
- Equipment calibration and records;
- Identification and control of samples;
- Computer program documentation and verification;
- Transmittal of information; and
- Record control and retention.

Planned audits and surveillances for this project will, as appropriate, cover the field activities and final reports. At regular intervals, ongoing field activities, laboratory activities and office material generated to backup future reports will be examined. Reports will be audited between the draft and final stages. Auditing will be performed in accordance with applicable engineering and quality assurance standards. The audits of field and laboratory activities include two independent parts - internal and external audits. A regular schedule for audits will be finalized for full scale operations.

10.2 FIELD PERFORMANCE AND SYSTEM AUDITS

10.2.1 Internal Field Audits

Internal Field Audit Responsibilities

An individual audit plan will be developed by the MK Program Quality Control Manager (PQCM) to provide a basis for each audit. This plan will identify the audit scope, activities to be audited, audit personnel, any applicable documents, and the schedule. The plan will be consistent with the scope of work, schedule, and requirements.

Internal Field Audit Frequency

These audits will verify that all established procedures are being followed. Internal field audits will be conducted at least once at the beginning of the site sample collection activities and periodically, as needed, throughout the life of the project.

Internal Field Audit Procedures

Field operations surveillance will involve an on-site visit by the MK PQCM or designated representative. Items to be examined may include the availability of appropriate and approved Work Plan and procedures; implementation of approved procedures; calibration and operation of equipment; labeling, packaging, storage and shipping of samples obtained; subcontractor performance; and documentation of deviations from the Work Plan and nonconformance.

The records of field operations will be reviewed to verify that field-related activities were performed in accordance with appropriate project procedures. Items reviewed will include, but not be limited to, the calibration records of field equipment; daily field activity logs; photographs; and data, logs, and checkprints resulting from the field operations. Audit checklists will be developed once project objectives and activities have been approved.

10.2.2 External Field Audits

External Field Audit Responsibilities

External field audits may be conducted by the USEPA Project Coordinator.

External Field Audit Frequency

External field audits may be conducted any time during the field operations. These audits may or may not be announced and are at the discretion of the USEPA.

10.3 LABORATORY PERFORMANCE AND SYSTEMS AUDITS

10.3.1 Internal Laboratory Audits

Internal Lab Audit Responsibilities

The internal laboratory audit may be conducted by the MK PQCM and the Project Chemist, as required.

Internal Lab Audit Frequency

The internal lab system audits will be done on an annual basis or as required by the MK PQCM and the Project Chemist.

Internal Lab Audit Procedures

The auditing of laboratory testing records will include, but not be limited to, the original data presentations prepared by the laboratory staff and laboratory test scheduling records.

A report audit will be performed prior to issuance of a final report. A report audit may examine, as appropriate, the documentation and verification of field and laboratory data; performance, documentation, and verification of analyses; documentation and verification of computer programs; preparation and verification of drawings, logs, and tables; content, consistency, and conclusions of the report; compliance with MK, regulatory, and project requirements; and maintenance and filing of project records. The issuance of the final submittal will be postponed if quality assurance personnel determine that the work does not meet requirements. If the project schedule demands issuance of a report prior to audit, it may be issued "preliminary" or "draft."

During an audit and upon its completion, the auditors will discuss the findings with the individuals audited and cite corrective actions to be initiated. Minor administrative findings, which can be resolved to the satisfaction of the auditors during an audit, are not required to be cited as items requiring corrective action. All findings that are not resolved during the course of the audit and findings affecting the overall quality of the project, regardless of when they are resolved, will be noted on the audit checklists. Audit results will be given to and discussed with the PM who will, along with the SQCS, institute the necessary corrective actions.

10.3.2 External Laboratory Audits

External Lab Audit Responsibilities

An external audit may be conducted by USEPA Region 5 Central Regional Laboratory (CRL).

External Lab Audit Frequency

An external lab audit may be conducted at least once prior to the initiation of the sampling and analysis activities. These audits may or may not be announced and are conducted at the discretion of the USEPA.

Overview of the External Lab Audit Process

External lab audits will include (but not be limited to) review of laboratory analytical procedures, laboratory on-site audits, and/or submission of performance evaluation samples to the laboratory for analysis.

SECTION 11
PREVENTATIVE MAINTENANCE

11.1 FIELD INSTRUMENT PREVENTATIVE MAINTENANCE

Specific preventative maintenance procedures to be followed for field equipment are those recommended by the manufacturer. Field instruments will be checked and calibrated daily before use. Calibration checks will be documented in the field logbook. Critical spare parts will be kept on-site to reduce downtime. Backup instruments and equipment will be available on-site or within one day shipment to avoid delays in the field schedule.

Required maintenance for the PID, pH meters and temperature probes is provided in Appendix E. Frequency of inspection and a list of replacement parts are provided in Table 11-1.

11.2 LABORATORY INSTRUMENT PREVENTATIVE MAINTENANCE

As part of their QA/QC program, a routine preventative maintenance program is conducted by the laboratory to minimize the occurrence of instrument failure and other system malfunctions. Designated laboratory employees shall regularly perform routine scheduled maintenance and repair of all instruments, or coordinate with the vendor for the repair of all instruments. All maintenance that is performed shall be documented in the laboratory's operating record. Laboratory equipment maintenance procedures are summarized in a SOP provided in Appendix F.

TABLE 11-1. SUMMARY OF MAINTENANCE - FIELD EQUIPMENT

Instrument	Maintenance Schedule	Spare Parts
PID (MicroTIP)	1. Calibrate at least once daily, prior to use, and as necessary during use. 2. Check battery and recharge when low. 3. Clean lamp window every 24 hours of operation. 4. Replace dust filter every 240 hours of operation. 5. Replace sample pump every 5000 hours of operation.	Battery charger Spare lamps Spare filter cartridges Span gas (isobutylene) Tedlar bags

TABLE 11-1. SUMMARY OF MAINTENANCE - FIELD EQUIPMENT		
Instrument	**Maintenance Schedule**	**Spare Parts**
pH Meter	1. Calibrate daily, prior to use.	pH buffers Batteries paper towels disposable gloves plastic cups
Temperature/Oxygen Meters (probes)	1. Calibrate if monitor goes off. 2. Check battery and recharge when low.	Battery charger

SECTION 12
SPECIFIC ROUTINE PROCEDURES USED TO ASSESS DATA PRECISION, ACCURACY AND COMPLETENESS

12.1 ACCURACY ASSESSMENT

In order to assure the accuracy of the analytical procedures, an environmental sample is randomly selected from each sample shipment received at the laboratory, and spiked with a known amount of the analyte or analyses to be evaluated. In general, a sample spike should be included in every set of 20 samples tested on each instrument. The spike sample is then analyzed. The increase in concentration of the analyte observed in the spiked sample, due to the addition of a known quantity of the analyte, compared to the reported value of the same analyte in the unspiked sample determines the percent recovery. Control charts are plotted for each commonly analyzed compound and kept on instrument, matrix, and analyte specific bases. The percent recovery for a spiked sample is calculated according to the following formula:

$$\%R = \frac{\text{Measured Amount in Spiked Sample} - \text{Measured Amount in Sample}}{\text{Known Amount Added}} \times 100$$

12.2 PRECISION ASSESSMENT

Spiked samples are prepared by choosing a sample at random from each sample shipment received at the laboratory, dividing the sample into equal aliquots, and then spiking each of the aliquots with a known amount of analyte. The duplicate samples are then included in the analytical sample set. The splitting of the sample allows the analyst to determine the precision of the preparation and analytical techniques associated with the duplicate sample. The relative percent difference (RPD) between the spike and duplicate spike are calculated and plotted. The RPD is calculated according to the following formula.

$$RPD = \frac{|\text{Amount in Spike 1} - \text{Amount in Spike 2}|}{0.5(\text{Amount in Spike 1} + \text{Amount in Spike 2})} \times 100$$

12.3 COMPLETENESS ASSESSMENT

Completeness is the ratio of the number of valid sample results to the total number of samples analyzed with a specific matrix and/or analysis. Following completion of the analytical testing, the percent completeness will be calculated by the following equation:

$$\text{Completeness} = \frac{\text{(number of valid measurements)}}{\text{(number of measurements planned)}} \times 100$$

SECTION 13
CORRECTIVE ACTION

13.1 CHANGE CONTROL

Prior to implementation, changes to original documents, procedures, and specifications will require approval by the parties originally responsible for approval of this document. It is the responsibility of project personnel to appropriately record the change and to make the documentation available to the PM and SQCS. The effect of the change on the project will then be evaluated by the PM and SQCS. A description of the proposed change, along with the results of the evaluation, will be presented to the Navy for approval. Following Navy approval, the change will be documented as a revision to the original document to reflect the work as actually performed.

13.2 FIELD CORRECTIVE ACTION

Nonconforming items and activities are those which do not meet the project requirements, procurement document criteria, or approved work procedures. Nonconformances may be detected and identified by:

- Project Staff - During the performance of field investigation and testing, supervision of subcontractors, and preparation and verification of analyses and design,

- Laboratory Staff - During the preparation for and performance of laboratory testing, calibration of equipment, and quality control activities, and

- Quality Assurance Personnel - During the performance of audits or daily review of field reports or data.

Each nonconformance affecting quality will be documented by the personnel identifying the nonconformance. For this purpose, a standard form (e.g., Nonconformance Report), results of laboratory analysis quality control tests, audit report, internal memorandum, or letter will be used as appropriate. Documentation will include:

- Identification of the individual(s) identifying the nonconformance,

- Cause and description of the nonconformance,

- Method(s) for correcting the nonconformance (corrective action) or description of the variance granted,
- Schedule for completing corrective action, and
- Any required approval signatures.

Documentation will be distributed to the Program Quality Control Manager (PQCM) with copies to the PM and SQCS. It is then the responsibility of the PQCM to approve and initiate resolution for the institution of the necessary corrective action to the nonconformance. Documentation describing the nonconformance and the corrective action implemented will be placed in the project file. Completion of corrective actions for significant nonconformances should be verified by quality assurance personnel as part of future auditing activities.

Any recurring nonconformance should be evaluated by project, laboratory, and/or quality assurance personnel to determine its cause and appropriate changes instituted in project requirements and procedures to prevent future recurrence. When such an evaluation is performed, the results will be documented.

13.3 LABORATORY CORRECTIVE ACTION

Corrective action in the laboratory may occur prior to, during and after initial analyses. A number of conditions such as broken sample containers, multiple phases, low/high pH readings, and potentially high concentration samples may be identified during sample log-in or just prior to analysis. Following consultation with lab analysts and section leaders, it may be necessary for the Laboratory Quality Assurance (QA) Officer to approve the implementation of corrective action. The laboratory standard operating procedures (SOPs) specify some conditions during or after analysis that may automatically trigger corrective action or optional procedures. These conditions may include dilution of samples, additional sample extract cleanup, automatic reinjection/reanalysis when certain quality control criteria are not met, etc.

The bench chemist will identify the need for corrective action. The Laboratory Project Manager, in consultation with the Laboratory supervisor and staff, will approve the required corrective action to be implemented by the laboratory staff. The Laboratory QA Officer will ensure implementation and documentation of the corrective action. If the nonconformance causes project objectives not to be achieved, it will be necessary to inform all levels of project management including the USEPA RPC to concur with the corrective action.

These corrective actions are performed prior to release of the data from the laboratory. The corrective action will be documented in both Laboratory's corrective action log (signed by analyst, section leader and quality control coordinator), and the narrative data report sent from the laboratory to the MK Project Chemist. If corrective action does not rectify the situation, the laboratory will contact the NSWC Project Manager.

13.4 CORRECTIVE ACTION DURING DATA VALIDATION AND DATA ASSESSMENT

The facility may identify the need for corrective action during either the data validation or data assessment. Potential types of corrective action may include resampling by the field team or reinjection/reanalysis of samples by the laboratory.

These actions are dependent upon the ability to mobilize the field team, whether the data to be collected is necessary to meet the required quality assurance objectives (*e.g.*, the holding time for samples is not exceeded, etc.) When the MK Project Chemist identifies a corrective action situation, it is the MK Project Manager who will be responsible for approving the implementation of corrective action, including resampling, during data assessment. All corrective actions of this type will be documented by the SQCS.

SECTION 14
QUALITY ASSURANCE REPORTS (POST-AUDIT)

Following completion of an audit, the auditors will prepare and submit an audit report to the MK Project Manager or, as necessary, the Laboratory Manager. This report will serve to notify management of audit results. The report may also be sent to other individuals contacted during the audit and the management of any affected subcontractor.

The report will be prepared as soon as possible (within 30 days) after the audit and contain, as appropriate:

- Date(s) of the audit
- Identification of audit participants
- Identification of activities audited
- Audit results
- Description of items requiring corrective action and, if possible, the means for correction
- Due date for completion of corrective actions and/or audit response
- Means for audit response (i.e., in writing)
- Impact, if any, on project schedule

If corrective action is required in the audit report, the corrective action will be undertaken and completed on schedule unless sufficient evidence can be provided to prove that the action is unnecessary.

The individuals audited will respond in writing to the audit report. The response will clearly state the corrective action taken or planned. If all corrective actions have not been completed prior to issuance of the audit response, a scheduled date for completion must be provided.

SECTION 15
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APPENDIX A

FIELD DATA SHEETS

Table A-1. Field Equipment List (Pilot Scale)

Category/item	Req'd Qty	Collected
Soil Sampling		
small stainless steel spoons		
large stainless steel spoons		
stainless steel bowls		
flagging and pins		
hand shovel		
hand auger and attachments		
stainless steel trowel		
putty knife		
folding table		
drive sampler		
brass sleeves and endcaps		
teflon tape		
aluminum pie pans (large)		
plastic sheets		
Compost Sampling		
Wiley No. 4 mill		
riffle splitter		
portable oven		
sample scale		
pH monitor		
temperature and oxygen probes		
~~aluminum pie pans (large)~~		
small stainless steel spoons		
large stainless steel spoons		
stainless steel bowls		
flagging and pins		
hand shovel/ large shovel		
~~hand auger~~ and attachments Push tube		
Measuring Sticks		
Decontamination		
distilled water		
reagent grade water (from lab)		
10% nitric acid solution (from lab)		
____ gallon tubs		
brushes		
pipe cleaner brushes		
alconox		
spray bottles		
chemical rinse tub		
Paper Towels		
Sample Shipping		
coolers from lab		
cubed ice		
ziploc bags (quart and gallon-sizes)		
bubble wrap		
strapping tape		
fed ex airbills and envelopes		
temperature blank		

Category/item	Req'd Qty	Collected
Miscellaneous		
sample containers (Table 4-8)		
photoionization detector		
paper towels		
clear plastic cups		
large garbage bags		
ziploc bags (gallon and quart)		
scissors		
tools (pliers, wrenches)		
steel tape/measuring tape (100')		
ruler (in cm, ft)		
stopwatch		
5.5 gallon buckets		
camera and film		
surveyor tape (cloth tape)		
cellular phone/2-way radio		
extra batteries for meters		
Pond/Tank Sampling		
~~disposable or stainless steel bailers~~		
fishing line (30+ pound test)		
cord reel		
~~slow rate pump and tubing~~		
garden hose ____ feet		
flow regulator (valve)		
purge water storage container		
bailer bottom-emptying devices		
extra bottle preservative		
pH paper		
Container on Rod		
Documentation		
field logbook		
sharpies		
sample labels		
custody seals		
sample logbook		
soil logging guidelines		
Quality Assurance Project Plan		
Pilot Scale Operational Plan		
Site Safety and Health Plan		
digging permits		
map of sampling locations (1 per site)		
hot work permit		
field data sheets		
chain-of-custody forms		
clipboard		
sample tags		

Table A-1. Field Equipment List (Pilot Scale)

Category/item	Req'd Qty	Collected
Health and Safety		
(rentals)		
PID		
Isobutylene or other cal gas (PID)		
regulator (ask rental place)/tubing		
Dust Monitor (mini-ram)		
personal air monitor		
(consumables)		
Kim wipes (medium/large)		
Respirators (note size), if required		
Chemical cartridges, if required		
HEPA cartridges, if required		
Cleaner/sanitizer for respirators		
PPE Covering Size:_____		
Type:_____Size:_____		
PPE gloves; Size:_____Type:_____		
PPE foot protection Size:_____		
Foam earplugs		
Safety Glasses		
Silver cloth duct tape		
Personal first aid kit		
Reflective safety vests		
Yellow "Caution" tape		
PID Lamp Cleaner bottles		
Two 10-foot tygon tubing:		
10 feet - 0.25 inch I.D.		
10 feet - 0.125 inch I.D.		
Two 20lb. ABC dry chemical fire		
extinguishers		
Bloodborne pathogen kits		
hardhats		
steel-toed boots		
orange pylons		
workhorses		

Category/item	Req'd Qty	Collected
Health and Safety		
(consumables)		
Fire Extinguisher		
CPR Pocket Masks		
Dual station/eyewash station		
Moist towelettes		
UL listed hazardous locations		
Smoke Alarms		
(support documents)		
copy of MK Safety manual (SouthDiv specific)		
Copy of MK Industrial Hygiene Procedures Manual		
Copy of MK Safety and Health Program Desc. for Haz. Waste Site Operations		
Copy of MK Accident Prev. Plan for SouthDiv Contract		
OSHA posters/ 2 OSHA 200 forms		
Tool Box Talks (orange book)		
EM 85-1-1 Corps Safety Man.		
Signed off SSHP/Work Plan		
MK Accident Data Report Form # 678/91		
MK Supervisor Accident Investigation Report Form # CAS 24/77		
MK Daily Logbook Report from SSHP		
MK Weekly Inspection Checklist forms from SSHP		
PMO project procedures: PHSP 001.1, 002.1, 003.1, 004.1, 005.1		
29 CFR 1910, 29 CFR 1926		

Notes:

1. A checkmark is placed under the column "Collected" once the item is inventoried at the site.
2. The above list should be reviewed carefully prior to work beginning to make sure all necessary items are accounted for.

Table A-1. Field Equipment List (Pilot Scale)

Category/item	Req'd Qty	Collected
Health and Safety		
(rentals)		
PID		
Isobutylene or other cal gas (PID)		
regulator (ask rental place)/tubing		
Dust Monitor (mini-ram)		
personal air monitor		
(consumables)		
Kimwipes (medium/large)		
Respirators (note size), if required		
Chemical cartridges, if required		
HEPA cartridges, if required		
Cleaner/sanitizer for respirators		
PPE Covering Size:_____:		
Type:_____Size:_____:		
PPE gloves; Size:_____Type:_____		
PPE foot protection Size:_____:		
Foam earplugs		
Safety Glasses		
Silver cloth duct tape		
Personal first aid kit		
Reflective safety vests		
Yellow "Caution" tape		
PID Lamp Cleaner bottles		
Two 10-foot tygon tubing:		
10 feet - 0.25 inch I.D.		
10 feet - 0.125 inch I.D.		
Two 20lb. ABC dry chemical fire		
extinguishers		
Bloodborne pathogen kits		
hardhats		
steel-toed boots		
orange pylons		
workhorses		

Category/item	Req'd Qty	Collected
Health and Safety		
(consumables)		
Fire Extinguisher		
CPR Pocket Masks		
Dual station/eyewash station		
Moist towelettes		
UL listed hazardous locations		
Smoke Alarms		
(support documents)		
copy of MK Safety manual (SouthDiv specific)		
Copy of MK Industrial Hygiene Procedures Manual		
Copy of MK Safety and Health Program Desc. for Haz. Waste Site Operations		
Copy of MK Accident Prev. Plan for SouthDiv Contract		
OSHA posters/ 2 OSHA 200 forms		
Tool Box Talks (orange book)		
EM 85-1-1 Corps Safety Man.		
Signed off SSHP/Work Plan		
MK Accident Data Report Form # 678/91		
MK Supervisor Accident Investigation Report Form # CAS 24/77		
MK Daily Logbook Report from SSHP		
MK Weekly Inspection Checklist forms from SSHP		
PMO project procedures: PHSP 001.1, 002.1, 003.1, 004.1, 005.1		
29 CFR 1910, 29 CFR 1926		

Notes:

1. A checkmark is placed under the column "Collected" once the item is inventoried at the site.
2. The above list should be reviewed carefully prior to work beginning to make sure all necessary items are accounted for.

TNT SOIL TEST KIT WORKSHEET

$Abs_{background}$ _____ $Abs_{control}$ _____

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)

A-3

RDX SOIL TEST KIT WORKSHEET

SAMPLE #	ABSORBANCE	RDX CONC., PPM $\frac{RDX_{abs} - 0.014}{0.0225}$

1) Abs "background"_____ 2) Abs "control"_____ 3) Abs "Nitrate/Nitrite"_____

A-4

APPENDIX B

LABORATORY REPORTING LIMITS FOR BIOFACILITY PROJECT ANALYTICAL METHODS

SOUT..WEST LABORATORY OF OKLAHOMA, INC.

	Volatiles
Test Code	MS300
Method	SW846 8240, 3rd Edition, Nov. 1986
Matrix	Soil-Water
Sample Volume	5 g - 5 mL
Initial Calibration	20-50-100-150-200 ppb ,%RSD=30% for CCC compounds, SPCC=RFF 0.3, Bromoform=RF 0.25
Continuing Calibration	50 ppb, %D = 25% for CCC Compounds

COMPOUND		CAS NUMBER	PQL's		MDL's	
			WATER	SOIL	WATER	SOIL
			ug/L	ug/kg	ug/L	ug/kg
1,1,1-Trichloroethane		+71-55-6	5	5	0.6	2.6
1,1,2,2-Tetrachloroethane	**	+79-34-5	5	5	1.0	2.9
1,1,2-Trichloroethane		79-00-5	5	5	0.8	2.7
1,1-Dichloroethane	**	+75-34-3	5	5	0.8	1.3
1,1-Dichloroethene	*	+75-35-4	5	5	1.0	1.4
1,2-Dichloroethane		107-06-2	5	5	0.9	1.7
1,2-Dichloroethene (Total)		+540-59-0	5	5	1.9	2.8
1,2-Dichloropropane	*	+78-87-5	5	5	0.6	2.2
2-Butanone		+78-93-3	10	10	1.3	5.2
2-Chloroethylvinyl Ether		+110-75-8	10	10	1.5	3.3
2-Hexanone		+591-78-6	10	10	0.8	3.8
4-Methyl-2-Pentanone		+108-10-1	10	10	0.7	4.3
Acetone		67-64-1	10	10	2.2	6.8
Benzene		+71-43-2	5	5	0.8	1.1
Bromodichloromethane		+75-27-4	5	5	0.7	2.4
Bromoform	**	+75-25-2	5	5	0.8	3.3
Bromomethane		+74-83-9	10	10	0.8	2.2
Carbon Disulfide		75-15-0	5	5	3.8	1.8
Carbon Tetrachloride		+56-23-5	5	5	0.7	3.1
Chlorobenzene	**	+108-90-7	5	5	0.9	1.8
Chloroethane		75-00-3	10	10	1.4	2.9
Chloroform	*	+67-66-3	5	5	0.8	1.4
Chloromethane	**	+74-87-3	10	10	1.0	2.9
Dibromochloromethane		+124-48-1	5	5	0.8	3.1
Ethylbenzene	*	+100-41-4	5	5	1.1	1.4
Methylene Chloride		75-09-2	5	5	1.5	1.3
Styrene		+100-42-5	5	5	1.0	1.5
Tetrachloroethene		+127-18-4	5	5	0.7	2.0
Toluene	*	+108-88-3	5	5	0.8	1.2
Trichloroethene		79-01-6	5	5	0.6	2.2
Vinyl Chloride	*	75-01-4	10	10	1.3	2.7
Vinyl acetate		108-05-4	10	10	0.8	7.5
Xylenes(total)		+1330-20-	5	5	3.3	3.6
cis-1,3-Dichloropropene		10061-01-	5	5	0.9	1.8
trans-1,3-Dichloropropene		10061-02-	5	5	1.0	2.1

* CCC **SPCC

All the above volatile compounds are referenced from PQL Table II, Rev. 0, Sept., 1986

SOUTHWEST LABORATORY OF OKLAHOMA, INC.

	Explosives
Test Code	GC200
Method	SW846 8330, 3rd Edition, Nov. 1992
Matrix	Water-Soil
Extract Volume	1 ml - 2 g
Initial Calibration	3 replicates of a 3 point calibration, %RSD = 20
Continuing Calibration	single point calibration, %D = 15
Instrument/analysis date (soils)	inst 12 – 2/94
Instrument/analysis date (water)	inst 12 – 2/94

COMPOUND	CAS NUMBER	PQL's		MDL's	
		WATER	SOIL	WATER	SOIL
		ug/L	ug/Kg	ug/L	ug/Kg
HMX	+2691-41-0	13.0	2200	3.5	265.0
RDX	+121-82-4	14.0	1000	2.9	182.0
TNB	+99-35-4	7.3	250	1.1	50.0
TETRYL	+479-45-8	10.0	650	3.1	82.0
DNB	+99-65-0	4.0	250	0.7	30.0
TNT	+118-96-7	6.4	250	1.2	43.0
NB	+98-95-3	7.0	260	1.3	65.0
26DNT	+606-20-2	9.4	260	3.1	63.0
24DNT	+121-14-2	5.7	250	1.6	41.0
2NT	+88-72-2	12.0	250	2.7	75.0
4NT	+99-99-0	8.0	250	2.0	55.0
3NT	99-08-1	7.9	250	2.1	48.0

MDL = 3.14 (for 99 percent confidence; from the "Student's t Value" table) times the standard deviation of seven replicates of a spiked sample matrix analyzed using the pertinent calibration. Reference: Federal Register, July 1982.

SOUTHWEST LABORATORY OF OKLAHOMA, INC

Metals reporting limits by ICP

Method	SW846 Third Edition, Nov. 1986, Method 6010
Matrix	Water-Soil
Extract Volume	100mL - 1g
Initial Calibration	0-1000 ug/L -varies
Continuing Calibration	1/2 high std

COMPOUND	CAS NUMBER	Test Services List	MDLS* WATER ug/L	MDLS* SOIL mg/kg
Aluminum	+7429-90-5	MT005	30	3.0
Antimony	+7440-36-0	MT025	30	3.0
Arsenic	+7440-38-2	MT055	100	10.0
Barium	+7440-39-3	MT065	5	0.5
Beryllium	+7440-41-7	MT085	1	0.1
Boron	+7440-42-8	MT115	50	5.0
Cadmium	+7440-43-9	MT125	4	0.4
Calcium	+7440-70-2	MT145	250	25.0
Chromium	+7440-47-3	MT165	5	0.5
Cobalt	+7440-48-4	MT185	5	0.5
Copper	+7440-50-8	MT205	10	1.0
Iron	+7439-89-6	MT225	20	2.0
Lead	+7439-92-1	MT245	40	4.0
Magnesium	+7439-95-4	MT265	250	25.0
Manganese	+7439-96-5	MT285	5	0.5
Molybdenum	+7439-98-7	MT325	15	1.5
Nickel	7440-02-0	MT345	10	1.0
Potassium	7440-09-7	MT365	1500	150.0
Selenium	+7782-49-2	MT385	100	10.0
Silicon	+7440-21-3	MT445	70	7.0
Silver	+7440-22-4	MT405	5	0.5
Sodium	+7440-23-5	MT425	500	50.0
Thallium	+7440-28-0	MT455	75	7.5
Tin	+7440-31-5	MT465	50	5.0
Titanium	+7440-32-6	MT485	10	1.0
Vanadium	+7440-62-2	MT505	5	0.5
Zinc	+7440-66-6	MT525	10	1.0

*DATA BASED ON WATER MDL'S <01/05/95> & SOIL MDL'S <03/22/95>

SOUTHWEST LABORATORY OF OKLAHOMA, INC.

Metals reporting limits by ICP

TEST CODE	MT800
Method	SW846 Third Edition, Nov. 1986, Method 6010
Matrix	Water-Soil
Extract Volume	100mL - 1g
Initial Calibration	0-1000 ug/L -varies
Continuing Calibration	1/2 high std

COMPOUND	CAS NUMBER	Test Services List	MDLS* WATER ug/L	MDLS* SOIL mg/kg
Antimony	+7440-36-0	MT025	30	3.0
Beryllium	+7440-41-7	MT085	1	0.1
Cadmium	+7440-43-9	MT125	4	0.4
Chromium	+7440-47-3	MT165	5	0.5
Copper	+7440-50-8	MT205	10	1.0
Nickel	7440-02-0	MT345	10	1.0
Silver	+7440-22-4	MT405	5	0.5
Zinc	+7440-66-6	MT525	10	1.0

*DATA BASED ON WATER MDL'S <01/05/95> & SOIL MDL'S <03/22/95>

Mercury by Cold Vapors

Test Code	MT310
Method	SW846 Third Edition, Nov. 1986
Matrix	Water-Soil
Extract Volume	100mL - 0.2g
Initial Calibration	0 - 10.0 ug/L
Continuing Calibration	1/2 HIGH STD

COMPOUND	CAS NUMBER	PRACT. QUANT. LIMITS WATER ug/L	PRACT. QUANT. LIMITS SOIL mg/kg	MDLS* WATER ug/L	MDLS* SOIL mg/kg	Test Services List	Method
Mercury	+7439-97-6			0.15		MT	7470
Mercury	+7439-97-6				0.075	MT	7471

* DATA BASED ON MDL'S ON <04/17/95>

Metals reporting limits by Low Level ICP

TEST CODE	MT803
Method	SW846 Third Edition, Nov. 1986, Method 6010
Matrix	Water-Soil
Extract Volume	100mL - 1g
Initial Calibration	0-500ug/L - varies
Continuing Calibration	1/2 high std

COMPOUND	CAS NUMBER	Test Services List	MDLS* WATER ug/L	MDLS* SOIL mg/kg
Arsenic	+7440-38-2	MT053	5	0.5
Lead	+7439-92-1	MT243	3	0.3
Selenium	+7782-49-2	MT383	5	0.5
Thallium	+7440-28-0	MT453	5	0.5

*DATA BASED ON WATER MDL'S <01/05/95> - SOIL MDL'S ARE TO UPDATED

SOUTHWEST LABORATORY OF OKLAHOMA, INC.

Metals by Graphite Furnace

Matrix	Water-Soil
Extract Volume	100mL - 1g
Initial Calibration	0-100 ug/L - varies
Continuing Calibration	1/2 high std

COMPOUND	CAS NUMBER	Method	Test Services List	MDLS WATER ug/L	MDLS SOIL mg/kg
Antimony	+7440-36-0	7041	MT028	3.0	0.3
Arsenic	+7440-38-2	7060	MT058	2.0	0.2
Cadmium	+7440-43-9	7131	MT128	0.5	0.1
Chromium	+7440-47-3	7191	MT168	1.0	0.2
Lead	+7439-92-1	7421	MT248	2.0	0.2
Selenium	+7782-49-2	7740	MT388	3.0	0.3
Silver	+7440-22-4	7761	MT408	0.5	0.05
Thallium	+7440-28-0	7841	MT458	2.0	0.3

* WATERS 03/21/95 <> SOILS 03/20/95

SOUTHWEST LABORATORY OF OKLAHOMA, INC.

Metals Toxicity by TCLP/EP Toxicity

Test Code	MT940
Method	SW846 Third Edition, Nov. 1986, Method 6010
Matrix	Water
Extract Volume	HG - 10mL ; ICP - 50mL
Initial Calibration	HG 0-10 ug/L; ICP 0-1000 ug/L varies
Continuing Calibration	1/2 high std

COMPOUND	CAS NUMBER	MDL * WATER mg/L	Test Services List	Individual Toxicity
Arsenic	+7440-38-2	0.100	MT844	MT054
Barium	+7440-39-3	0.005	MT844	MT064
Cadmium	+7440-43-9	0.004	MT844	MT124
Chromium	+7440-47-3	0.005	MT844	MT164
Lead	+7439-92-1	0.040	MT844	MT244
Mercury - CVAA	+7439-97-6	0.0015	MT314	MT314
Selenium	+7782-49-2	0.100	MT844	MT334
Silver	+7440-22-4	0.005	MT844	MT404

* DATA BASED ON WATER MDL'S FOR HG <04/17/95> & ICP <01/05/95>

SOUTHWEST LABORATORY OF OKLAHOMA, INC.

Micellaneous Inorganic Analyses

Test Code — Methods various

Method

Matrix — Water-Soil

Extract Volume

Initial Calibration

Continuing Calibration

COMPOUND	METHOD	Test Services List	PRACT. QUANT. LIMITS WATER mg/L	SOIL mg/Kg
Acidity	SM402	IN005	1.0	-
Alkalinity	EPA310.1	IN010	20.0	-
Ammonia (ISE)	EPA350.3	IN015	0.1	1.0
Ammonia (Dist)	EPA350.2	IN020	1.0	10.0
Bromide (IC)	EPA300.0	IN030	0.2	1.0
Chemical Oxygen Demand	EPA410.4	IN090	10.0	100.0
Chloride (IC)	EPA300.0	IN055	0.2	1.0
Chloride (by titration)	EPA325.3	IN050	0.1	1.0
Chlorine (Residual)	EPA330.4	IN070	0.2	-
Chlorine (free)	SM408D	IN075	0.1	-
Color	SM204A	IN100	-	-
Corrosivity pH (@25 C)	SW846-9045	HW200	-	-
Cyanide (Total)	SW846-9010	IN120	10.0	0.5
Cyanide (Amenable)	SW846-9010	IN122	0.0	0.5
Density	SM210B	IN128	-	-
Fluoride (IC)	EPA300.0	IN135	0.2	1.0
Fluoride (ISE)	EPA340.2	IN137	0.1	-
Hardness (As CaCO3)	EPA130.2	IN140	1.0	-
Hexavalent Chromium	SW846-7196	MT169	0.05	0.25
Iodine	EPA300.0	IN150	0.2	1.0
Nitrate (Color)	EPA353.2	IN180	0.1	1.0
Nitrate (IC)	EPA300.0	IN185	0.2	0.2
Nitrite (Color)	EPA354.1	IN190	0.1	0.4
Nitrite (IC)	EPA300.0	IN195	0.2	0.4
Oil & Grease	EPA413.1	IN200	0.5	10.0
Oil & Grease by IR	EPA413.2	IN205	0.5	10.0
Ortho Phosphorus (Color)	EPA365.2	IN250	0.1	10.0
Ortho Phosphorus (IC)	EPA300.0	IN255	0.2	1.0
Paint Filter (Filtrate)		HW500	-	-
Percent Moisture	GRAY 105c	IN650	-	
pH Value (@ 25 C)	EPA150.1	IN220	-	-
Phenols (Total)	EPA420.2	IN230	0.1	0.5
Phosphorus (Total)	EPA365.2	IN240	0.1	10.0
Reactivity Cyanide	SW 7.3.4.2	IN125	0.1	0.1
Reactivity Sulfide	SW 7.3.4.2	IN305	0.0	1.0
Silica	EPA370.1	IN260	0.2	1.0
Sodium Absorption Ratio	CALCULATION	IN620	-	-
Specific Conductance (@ –C	EPA120.1	IN080	-	-
Specific Gravity	SM213E	IN139	-	-
Sulfate (Gravimetric)	SM426B	IN290	0.2	1.0
Sulfate (IC)	EPA300.0	IN295	0.2	1.0
Sulfate (Turbidimetric)	EPA375.4	IN297	1.0	1.0
Sulfide	EPA376.1	IN300	1.0	1.0

Sulfite		EPA377.1	IN307	2.0	2.0
Sulfur		ASTM D-1552	OS630	-	-
Surfactants (MBAS)		SM512B	IN160	0.1	-
Total Dissolved Solids		EPA160.1	IN270	10.0	-
Total Kjeldahl Nitrogen		EPA351.3	IN170	1.0	10.0
Total Organic Carbon		EPA415.1	IN045	0.4	100.0
Total Organic Halogens		SW9020	IN060	0.0	10.0
Total Petroleum Hydrocarbon		SM503E	IN212	0.5	10.0
Total Residue		EPA160.3	IN280	10.0	-
Total Settleable Solids		EPA160.5	IN287	-	-
Total Suspended Solids		EPA160.2	IN275	10.0	-
Total Volatile Solids		EPA160.4	IN285	10.0	-
Turbidity		EPA180.1	IN310	0.1	-
Volatile Suspended Solids		SM209D	IN286	1.0	-
Water Content (Dean Startk)		ASTM E-123	IN655	-	-

APPENDIX C

(RESERVED)

APPENDIX D

LABORATORY SAMPLE CUSTODY PROCEDURES

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

Table of Contents

I. CHAIN-OF-CUSTODY 1
A. Summary 1
II. SAMPLE LABEL 1
A. Description-Field Sample Label 1
III. CHAIN-OF-CUSTODY RECORD-EXTERNAL COC 1
A. Description 1
B. Procedure 2
IV. TRANSFER OF CUSTODY AND SHIPMENT-EXTERNAL 2
A. Procedure 2
V. LABORATORY CUSTODY PROCEDURES-INTERNAL 2
A. Procedure 2
B. Sample Tracking 3

FIG. 3.1-A Example of Sample Bottle Label (top) & Sample Tag (right). 5
FIG. 3.1-B Example Custody Seal for Sample Containers/Coolers 5
FIG. 3.2 Example of Chain of Custody Record 6
FIG. 3.3 Chain of Custody Sample Tracking Flow Diagram 7
FIG. 3.5 Client/Laboratory Communication System 8
FIG. 3.6 Sample Request Form/Analysis Request Form 9
FIG. 3.7 LIMS Generated Sample Worksheet 10
FIG. 3.8 EPA-CLP Sample Log-In Sheet 11
FIG. 3.9 Internal Chain of Custody 12
FIG. 3.10 Digestion Record 13
FIG. 3.11 Sample Distillation Record 14
FIG. 3.12 Sample Extraction Log 15
FIG. 3.13 Volatiles GC/MS Run Log 16
FIG. 3.14 Sample Archive Record Sheet 17

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 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 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.

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 TIME | CLIENT CONTACT | PHONE NUMBER

P.O. NUMBER/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)

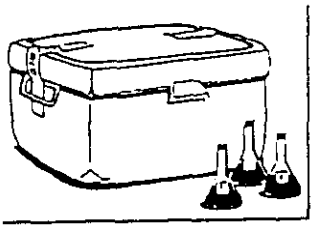
RELINQUISHED BY (Signature)	DATE	TIME	RECEIVED BY (Signature)	RELINQUISHED BY (Signature)	DATE	TIME	RECEIVED FOR LABORATORY BY (Signature)

RELINQUISHED BY (Signature)	DATE	TIME	RECEIVED BY (Signature)	REMARKS:

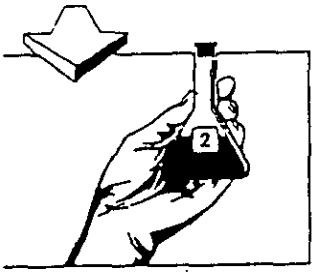
SR012-1192-031

FIG. 3.2 Example of Chain of Custody Record

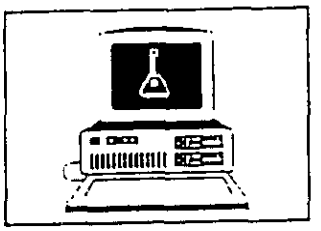
FIG. 3.3 Chain of Custody Sample Tracking Flow Diagram



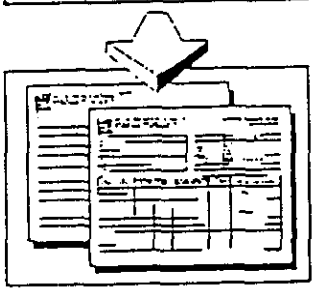
Sample Receipt



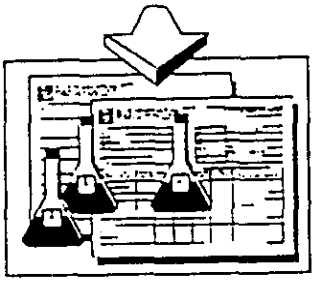
Sample Inspection
(Primary & Secondary Containers)
Radiation screening, RAD Samples Only



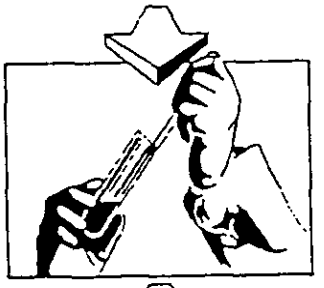
Sample Log-In
(Assigned Unique ID Number)



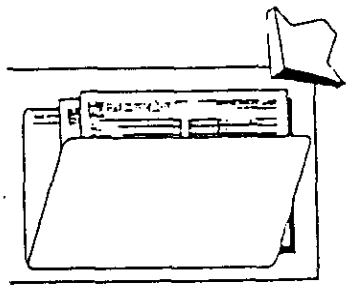
Initiate Internal Chain-of Custody Documentation



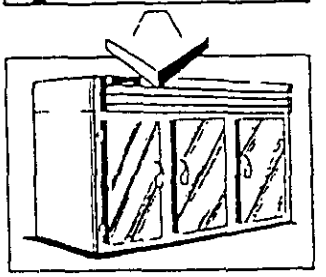
Sample Relinquished to Appropriate Laboratory Technician. (Sample maintained in locked storage)



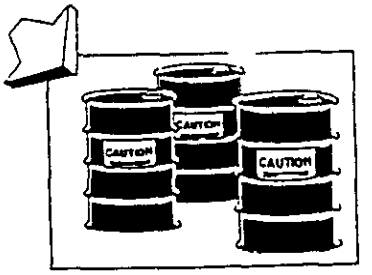
Sample Preparation and Analysis
(Sample maintained in locked storage)



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

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):

➤ 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

D-10

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: 21032

SAMPLE LOG-IN RECORD

SAMPLE #	DATE IN	DESCRIPTION	SDG	MA	NC	TEST	FRI	DUE	CONTAINER	DESCRIPTION	RESULTS	ANALYST	DATE/TIME
16075.01	10/25/93	FT919 10A/GC		W	18	GC815	4	11/14/93	MNOPQR	PEST CLP OLM016			
										EXTRACTION		MT	10/26/93
						MS315	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#21032)		W	6	GC815	4	11/14/93	EF	PEST CLP OLM016			
										EXTRACTION		MT	10/26/93
						MS315	4	11/14/93	AB	VOA-CLP OLM01.8			
						MS515	4	11/14/93	CD	BNA CLP OLM01.8			
										EXTRACTION		TB	10/25/93
16075.03	10/25/93	FT921 (CASE#21032)		W	6	GC815	4	11/14/93	EF	PEST CLP OLM016			
										EXTRACTION		MT	10/26/93
						MS315	4	11/14/93	AB	VOA-CLP OLM01.8			
						MS515	4	11/14/93	CD	BNA CLP OLM01.8			
										EXTRACTION		TB	10/25/93
16075.04	10/25/93	FT922 (CASE#21032)		W	6	GC815	4	11/14/93	EF	PEST CLP OLM016			
										EXTRACTION		MT	10/26/93
						MS315	4	11/14/93	AB	VOA-CLP OLM01.8			
						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

D-12

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: ____________

	CORRESPONDING		
EPA SAMPLE #	SAMPLE TAG #	ASSIGNED LAB #	REMARKS: CONDITION OF SAMPLE SHIPMENT, ETC.

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: ____________ ____________

* Contact SMO and attach record of resolution

Received By: ______________________

Date: ______________________

Logbook No: ______________________

Logbook Page No: ______________________

FORM DC-1

3/90

FIG. 3.8 EPA-CLP Sample Log-In Sheet

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	GCB17	4	10/17/95	CD	PEST CLP OLM03			
										EXTRACTION			
						MS517	4	10/17/95	AB	VOA-CLP OLM03.			
						MS517	4	10/17/95	CD	BNA CLP OLM03.			
										EXTRACTION			
23643.02	09/27/95				0	GCB17	4	10/17/95	CD	PEST CLP OLM03			
										EXTRACTION			
						MS517	4	10/17/95	AB	VOA-CLP OLM03.			
						MS517	4	10/17/95	CD	BNA CLP OLM03.			
										EXTRACTION			
23643.03	09/27/95				0	GCB17	4	10/17/95	GHIJ	PEST CLP OLM03			
										EXTRACTION			
						MS517	4	10/17/95	ABCDEF	VOA-CLP OLM03.			
						MS517	4	10/17/95	GHIJ	BNA CLP OLM03.			
										EXTRACTION			
23643.04	09/27/95				0	GCB17	4	10/17/95	CD	PEST CLP OLM03			
										EXTRACTION			
						MS517	4	10/17/95	AB	VOA-CLP OLM03.			
						MS517	4	10/17/95	CD	BNA CLP OLM03.			
										EXTRACTION			
23643.05	09/27/95				0	MS517	4	10/17/95	AB	VOA-CLP OLM03.			

FIG. 3.9 Internal Chain of Custody

SWLO / AATS Sample Digestion Record

Date : 04/05/1994 Batch: 04059411 Analyst: TS
Type : MT600 ACID DIGESTION - ICP/AA Client: CLP-PRI MEH-II 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	940308000275		50 ML	50 ML	CO/CL	CO/CL		<2	
17824.01MS	940308000275		50 ML	50 ML	CO/CL	CO/CL		<2	0.5 ML W-1,W-2,B5
17824.01MSD	940308000275		50 ML	50 ML	CO/CL	CO/CL		<2	0.5 ML W-1,W-2,B5
17824.02	940309405370		100 ML	100 ML	CO/CL	CO/CL		<2	
17824.03	940309405373		100 ML	100 ML	CO/CL	CO/CL		<2	
17824.04	940309405376		100 ML	100 ML	CO/CL	CO/CL		<2	
17836.01	940309000282		100 ML	100 ML	CO/CL	CO/CL		<2	
17836.02	940309000283		100 ML	100 ML	CO/CL	CO/CL		<2	
17836.02MS	940309000283		100 ML	100 ML	CO/CL	CO/CL		<2	1.0 ML W-1,W-2,B5
17836.02MSD	940309000283		100 ML	100 ML	CO/CL	CO/CL		<2	1.0 ML W-1,W-2,B5
17836.03	940310000284		100 ML	100 ML	CO/CL	CO/CL		<2	
17836.04	940311405412		100 ML	100 ML	CO/CL	CO/CL		<2	
17836.05	940311405413		100 ML	100 ML	CO/CL	CO/CL		<2	
17836.06	940311405415		100 ML	100 ML	CO/CL	CO/CL		<2	
17836.07	940310405394		100 ML	100 ML	CO/CL	CO/CL		<2	
17836.08	940311405414		100 ML	100 ML	CO/CL	CO/CL		<2	
17860.01	940314000285		50 ML	50 ML	CO/CL	CO/CL		<2	
17860.01MS	940314000285		50 ML	50 ML	CO/CL	CO/CL		<2	0.5 ML W-1,W-2,B5
17860.01MSD	940314000285		50 ML	50 ML	CO/CL	CO/CL		<2	0.5 ML W-1,W-2,B5
17860.02	940314000286		100 ML	100 ML	CO/CL	CO/CL		<2	
17860.03	940314000287		100 ML	100 ML	CO/CL	CO/CL		<2	

FIG. 3.10 Digestion Record

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 (Spiking Solution)

SOUTHWEST LABORATORY OF OKLAHOMA, INC. • 1700 W. ALBANY • BROKEN ARROW, OKLAHOMA 74012 • OFFICE (918) 251-2858 • FAX (918) 251-2599 (IN018-0293-01)

FIG. 3.11 Sample Distillation Record

D-16

METHOD	SV	PEST + PCB	OTHER
CONT. EX			
SCP #			
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 DIV	% MOISTURE	DRY WT (GM)	AMOUNT EXTRACTED	EXTRACTED VOLUME	GPC VOLUME	GPC FLUTE #	FLORISIL VOL CONC	FLORISIL # OR CONC	VOL SV - P	FIN PROCESS VOLUME	SCREEN T/F	VOL SV - P	SOXH SV - P	PCB DIV FOR EVALUATION	ACID DIV CONTAMINATION	COMMENTS
				%	g	g * mL	mL	mL	mL	mL	mL	mL	mL		mL	mL			

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):
MISC:

COMMENTS

33050

FIG. 3.12 Sample Extraction Log

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

FIG. 3.13 Volatiles GC/MS Run Log

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-21D	W	04/03/91
5447.11	RS&A	MW 18/B	W	04/03/91
5447.12	RS&A	MW-3D	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-18B	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

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

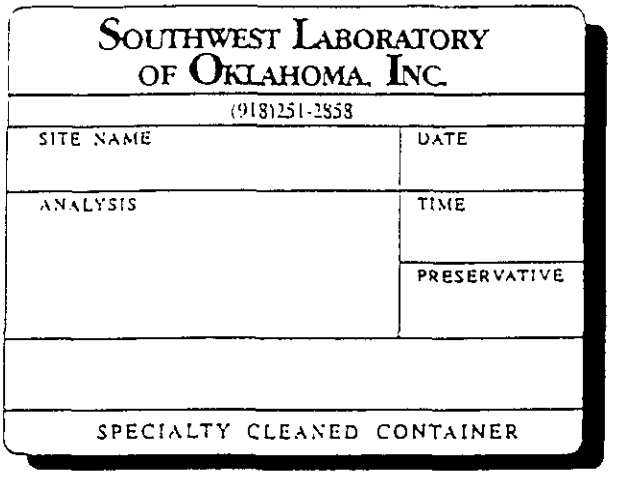
A label is attached to all sample containers at the time of collection. The label is written in indelible ink and contains the following information:

- 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.



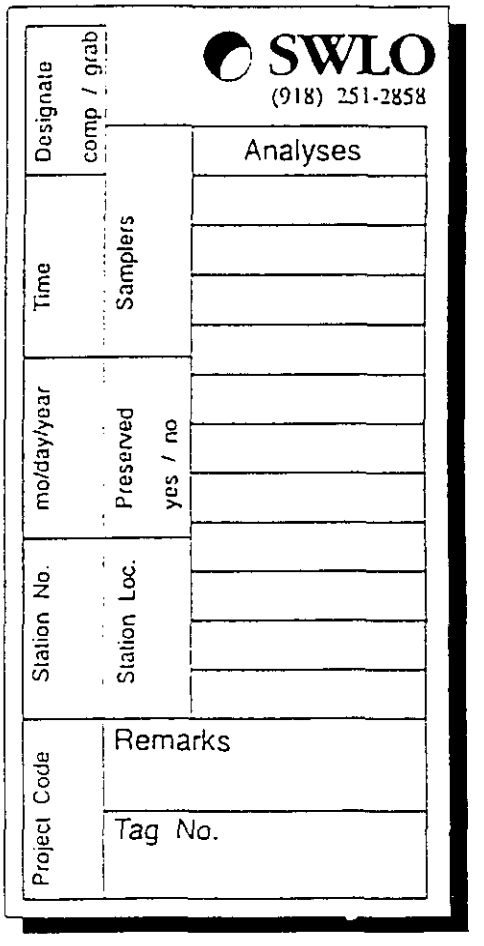


FIG. 3.1 Example of Sample Bottle Label & Sample Tag.

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

CHAIN OF CUSTODY RECORD

SOUTHWEST LABORATORY OF OKLAHOMA, INC.

SAMPLING FIRM | CLIENT CONTACT | PHONE NUMBER

AFE/PROPOSAL NUMBER | PROJECT NAME

SAMPLER (Signature)

ANALYTICAL TESTS REQUESTED

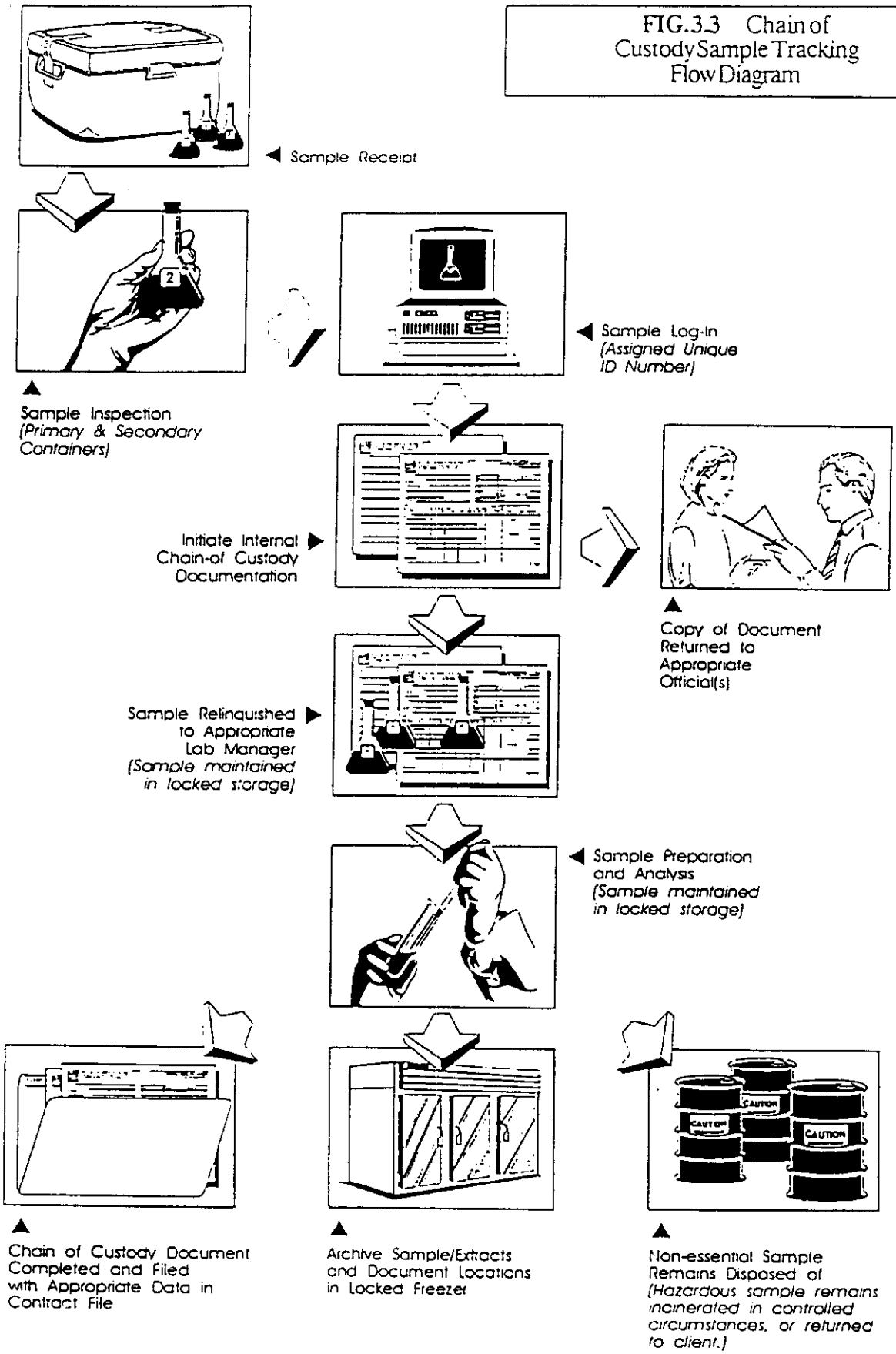
SAMPLE NO	DATE	TIME	COMP. GRAB	LOCATION	MATRIX	NUMBER OF CONTAINERS							REMARKS

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:			

SR012-1192-01

FIG. 3.2 Example of Chain of Custody Record

FIG.3.3 Chain of Custody Sample Tracking Flow Diagram



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)

- 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.

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 followir.

- 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.

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.

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

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 [PJ001-0492-01]

FIG.3.6 Client/Laboratory Communication System

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

SOUTHWEST LABORATORY OF OKLAHOMA, INC.
1700 WEST ALBANY • BROKEN ARROW, OK 74012-1421
OFFICE (918) 251-2858 • 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

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: ____________

EPA SAMPLE #	CORRESPONDING SAMPLE TAG #	CORRESPONDING ASSIGNED LAB #	REMARKS: CONDITION OF SAMPLE SHIPMENT, ETC.

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: ________ ________

* Contact SMO and attach record of resolution

Received By: ________ ________

Date: ________

Logbook No: ________

Logbook Page No: ________

FORM DC-1

3/90

FIG.3.8 (cont.) EPA-CLP Sample Log-In Sheet

SOUTHWEST LABORATORY OF OKLAHOMA, INC.
1700 W. ALBANY SUITE C
BROKEN ARROW, OK 74012 1421

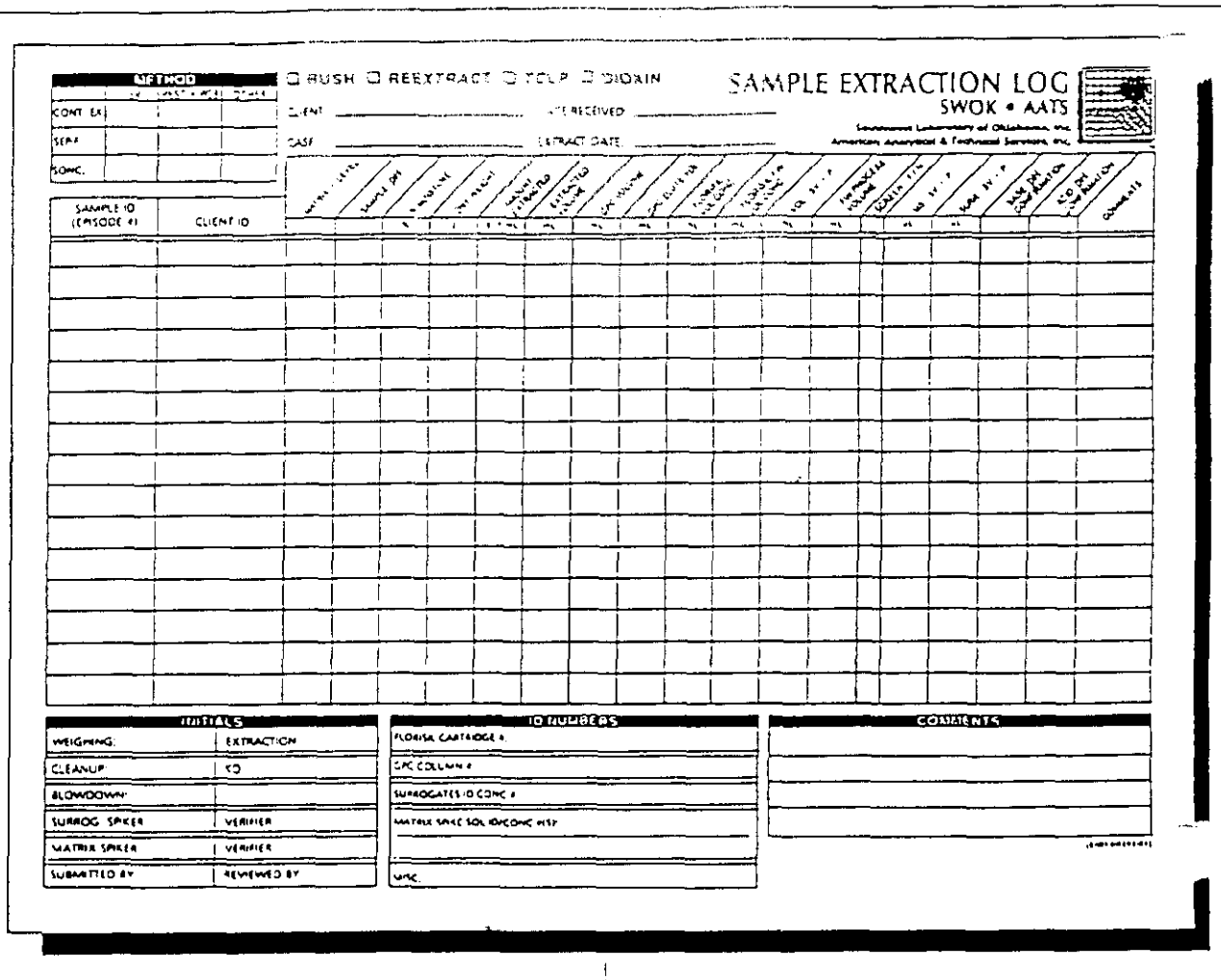
Date: 10/27/93
Batch#: 16075
Client: EPA
Project: [illegible]

SAMPLE LOG-IN RECORD

SAMPLE #	DATE IN	DESCRIPTION	SDG	MA	NO	TEST	PRI	DUE	CONTAINER	DESCRIPTION	RESULTS	ANALYST	DATE/TIME
16075.01	10/25/93	FT919 10A/OC		W	10	GC815	4	11/14/93	MNOFOR	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 OLM01.3			
										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



☐ 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 _____

METHOD			
CONT. EX.			
SEPF			
SONIC.			

SAMPLE ID (EPISODE #)	CLIENT ID	METHOD - LEVEL	SAMPLE DATE	% MOISTURE	DRY WEIGHT	SAMPLE EXTRACTED	EXTRACTED VOLUME	GPC VOLUME	GPC ELUTE VOL	FLORISIL VOLUME	FLORISIL ELUTE VOL	VOL. BY	PRE PROCESS VOLUME	SOLVENT TYPE	VOL. BY	FINAL BY	BASE DIL CONC/DILUTION	ACID DIL CONC/DILUTION	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:
MISC.

COMMENTS

FIG. 3.10 Internal SampleTracking (Extraction Log)

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

SWL/AATS • 1700 West Albany • Broken Arrow, Ok 74012 |VGM008-0492-01|

FIG.3.11 Internal Sample Tracking (Volatiles GC/MS Run Log)

[illegible] / AA75 [illegible] Digestion Record

Date : [illegible] Batch: [illegible] Analyst: [illegible]
Type : [illegible] Client: [illegible] Matrix: [illegible]

LAB ID	Client ID	Typ	Initial Vol	Final Vol	Color Before	Color After	Texture	SM	Comments
PBW		LB	100 ML	100 ML					
LCSW		BS	100 ML	100 ML					1.0 ML W-1.W-2.85
17824.01	940308000275		50 ML	50 ML	CO/CL	CO/CL		+2	
17824.01MS	940308000275		50 ML	50 ML	CO/CL	CO/CL		+2	0.5 ML W-1.W-2.85
17824.01MSD	940308000275		50 ML	50 ML	CO/CL	CO/CL		+2	0.5 ML W-1.W-2.85
17824.02	940309805370		100 ML	100 ML	CO/CL	CO/CL		+2	
17824.03	940309805372		100 ML	100 ML	CO/CL	CO/CL		+2	
17824.04	940309805375		100 ML	100 ML	CO/CL	CO/CL		+2	
17826.01	940309000262		100 ML	100 ML	CO/CL	CO/CL		+2	
17826.02	940309000263		100 ML	100 ML	CO/CL	CO/CL		+2	
17826.02MS	940309000263		100 ML	100 ML	CO/CL	CO/CL		+2	1.0 ML W-1.W-2.85
17826.02MSD	940309000263		100 ML	100 ML	CO/CL	CO/CL		+2	1.0 ML W-1.W-2.85
17826.03	940310000564		100 ML	100 ML	CO/CL	CO/CL		+2	
17826.04	940311805412		100 ML	100 ML	CO/CL	CO/CL		+2	
17826.05	940311805413		100 ML	100 ML	CO/CL	CO/CL		+2	
17826.06	940311805415		100 ML	100 ML	CO/CL	CO/CL		+2	
17826.07	940310805394		100 ML	100 ML	CO/CL	CO/CL		+2	
17826.08	940311805414		100 ML	100 ML	CO/CL	CO/CL		+2	
17860.01	940314000285		50 ML	50 ML	CO/CL	CO/CL		+2	
17860.01MS	940314000285		50 ML	50 ML	CO/CL	CO/CL		+2	0.5 ML W-1.W-2.85
17860.01MSD	940314000285		50 ML	50 ML	CO/CL	CO/CL		+2	0.5 ML W-1.W-2.85
17860.02	940314000286		100 ML	100 ML	CO/CL	CO/CL		+2	
17860.03	940314000287		100 ML	100 ML	CO/CL	CO/CL		+2	

FIG. 3.12 Internal SampleTracking (Sample Digestion Record)

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 (Gm/mL)	Final Vol (mL)	Description (Water color, Clarity, Soil color, Texture Artefacts)	pH	Sulfides Present (Y or N)	Chlorine Present (Y or N)	Comments (Sodium Solution)

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FIG. 3.13 Internal SampleTracking (Sample Distillation Record)

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-7B	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-20	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

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

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

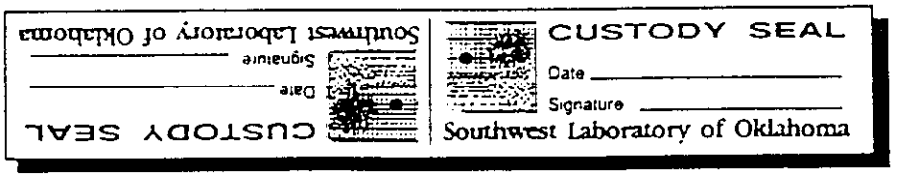
When it becomes necessary to make corrections to any form of documentation (e.g., sample tags, COC forms, daily logbooks), the obsolete information is crossed out with a single line and the changes are made and initialed by the person making the change.

3.9 SAMPLE DISPOSAL

Sample Disposal

1. Upon completion of the analysis, an archive 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



FREEZER TEMPERATURE RECORD

OA/OC 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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[OA01-0992-02]

Figure 3.16 Freezer Temperature Record

APPENDIX E

PROCEDURES FOR FIELD INSTRUMENTS

MICROTIP

MP-1000

User's Manual

PHOTOVAC

MICROTIP

Notice

Use of Serial Devices with MicroTIP

When using a serial device, such as a printer or a computer, with MicroTIP you must turn MicroTIP off before connecting the printer cable to the I/O port.

Operation of serial devices with MicroTIP is described in Sections 2.15, 2.16, 3.1, 3.2, 3.3 (only in EX-4000) and 5.3.

Part No. 600676

MICROTIP MP-1000

Hand Held Air Monitor/Photoionization Detector

User's Manual

PHOTOVAC

Photovac Monitoring Instruments
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Deer Park, New York
11729, USA
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Fax (516)254-4284

Photovac Incorporated
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Part No. 395024

E-3

Table of Contents

Chapter 1 Introduction

1.1 Unpacking 1
1.2 Recharging the Battery 1

Chapter 2 Operation

2.1 Overview 3
2.2 Tutorial Session 4
2.3 DISPLAY 5
2.4 LIGHT 5
2.5 BATT 6
2.6 MAX 6
2.7 CLEAR 7
2.8 EVENT 7
2.9 EXIT 8
2.10 SETUP 8
2.11 AUDIO 9
2.12 ALARM 10
2.13 PLAY 10
2.14 CAL 11
2.15 PRINT 13
2.16 GRAPH 16
2.17 High Sensitivity Operation 19
2.18 Response Factors For Gases and Vapors 20

Chapter 3 Accessories and Other Devices

3.1 Printer 22
3.2 Computer 23
3.3 Chart Recorder 25
3.4 Sample Bag 25
3.5 Three Meter (9.8') Sample Line 26
3.6 Shoulder Strap 26
3.7 Headset 26
3.8 Replacement Detector Lamps 26

Chapter 4 Routine Maintenance

4.1 Charging the Battery 32
4.2 Cleaning the Lamp Window 32
4.3 Replacing the Detector UV Lamp 34
4.4 Replacing the Inlet Filter 34
4.5 Replacing the Sample Pump 35

Chapter 5 Troubleshooting
5.1 If MicroTIP Draws In Liquid 37
5.2 Instrument Status and Fault Displays 37
5.3 Establishing Computer Communications 40

Chapter 6 Technical Description
6.1 Overview 42
6.2 Photoionization Detector 43
6.3 Calibration and Recording 44

Chapter 7 Specifications 46

Chapter 8 Warranty 48

Appendices
Appendix A - Installing AC Plug 49
Appendix B - Span Gas Supplier 49

Index 50

List of Figures

Figure 1 MicroTIP Layout 2
Figure 2 Normal Display 3
Figure 3 Printed Output 15
Figure 4 Graphed Output 18
Figure 5 Printer Cable Electrical Connection 22
Figure 6 Pump Placement 35
Figure 7 Block Diagram 42
Figure 8 Photoionization Detector 44

List of Tables

Table 1 TUTOR Displays 4
Table 2 Response Factors 21
Table 3 Printer Cable Pinouts 23
Table 4 Response Factors with 9.5 eV Lamp 28
Table 5 Instrument Status 38
Table 6 Pin Definition 40

E-5

WARNING

THIS EQUIPMENT GENERATES, USES AND CAN RADIATE RADIO FREQUENCY ENERGY AND IF NOT INSTALLED AND USED IN ACCORDANCE WITH THE INSTRUCTION MANUAL, MAY CAUSE INTERFERENCE TO RADIO COMMUNICATIONS. IT HAS BEEN TESTED AND FOUND TO COMPLY WITH THE LIMITS FOR DOC STANDARD C108.8 AND FOR A CLASS A COMPUTING DEVICE PURSUANT TO SUBPART J OF PART 15 OF FCC RULES, WHICH ARE DESIGNED TO PROVIDE REASONABLE PROTECTION AGAINST SUCH INTERFERENCE WHEN OPERATED IN A COMMERCIAL ENVIRONMENT. OPERATION OF THIS EQUIPMENT IN A RESIDENTIAL AREA IS LIKELY TO CAUSE INTERFERENCE IN WHICH CASE THE USER AT HIS OWN EXPENSE WILL BE REQUIRED TO TAKE WHATEVER MEASURES MAY BE REQUIRED TO CORRECT THE INTERFERENCE.

MicroTIP User's Manual Supplement

MicroTIP has been revised as follows since this User's Manual was produced:

The Battery Charger Input Voltage is printed on the charger. The voltage selector switch has been eliminated.

The Battery Charger Red LED will glow brightly when a discharged battery is connected. The LED will glow dimly when the battery is fully charged.

PHOTOVAC 600756-02

Chapter 1 Introduction

1.1 UNPACKING

Included with the instrument you will find the following standard accessories:

- User's Manual
- 120/230 Volt Battery Charger
- 17 cm (6.8") Sample Line
- 5 Spare Filter Cartridges
- Shoulder Strap

Remove MicroTIP™ and accessories from the shipping box and examine them for any physical damage. Inform Photovac immediately if MicroTIP or the accessories are damaged.

Instruments being used outside the United States and Canada must have a new plug installed on the battery charger. See Appendix A.

1.2 RECHARGING THE BATTERY

Before beginning operation of MicroTIP, the battery pack must be charged.

Note: Use only MicroTIP battery charger Photovac Part No. 395016 for 110V or 395017 for 220V. Using another MicroTIP battery charger will result in damage to the MP-1000 battery pack or charger.

1. Ensure MicroTIP is off by pressing the front of the power switch. See Figure 1.

2. Set the voltage selector switch on the bottom of the battery charger to the appropriate AC line voltage.

3. Press the release button on the bottom of MicroTIP and remove the battery pack by sliding it backwards. See Figure 1.

4. Plug the charger into the battery pack and then into an AC outlet. Allow the battery pack to charge until the red LED on the battery charger flashes slowly.

If the battery pack is fully discharged this will take about 8 hours.

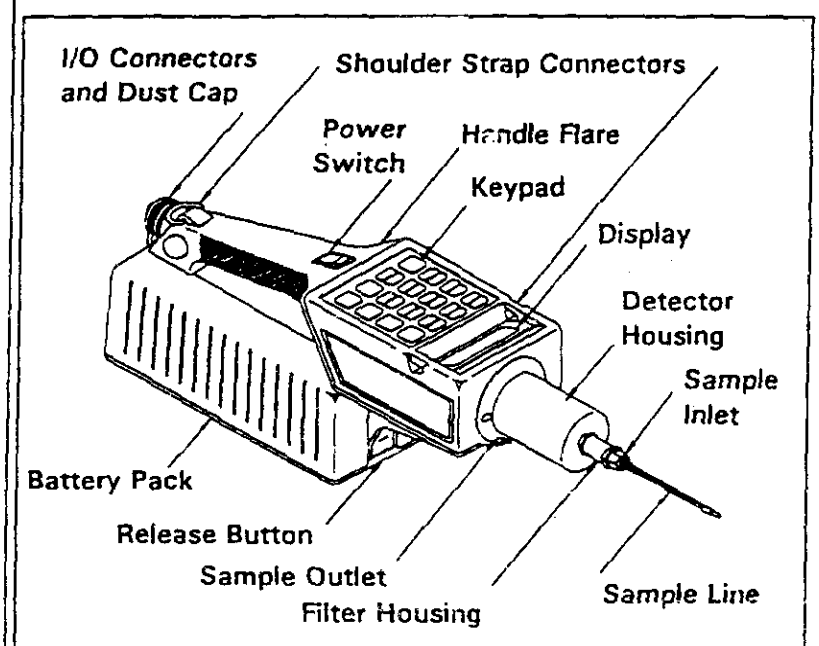


Figure 1 MicroTIP Layout

5. After charging remove the charger, first from the wall outlet then from the battery pack, and slide the battery pack back onto MicroTIP.

The instrument is now fully charged and ready for use.

Chapter 2 Operation

2.1 OVERVIEW

MicroTIP measures the concentration of airborne photoionizable gases and vapors and automatically displays and records these concentrations.

MicroTIP does not distinguish between individual pollutants. The reading displayed represents the total concentration of all photoionizable chemicals present in the sample.

```
Warming up now,
  please wait...
```

Turn the instrument on by pressing the back of the power switch. The pump will start and the message "Warming up now, please wait" will be displayed. Within three minutes the following information will appear on the display: instrument status, current detected concentration, Event number (if the datalogger is on), time, and date.

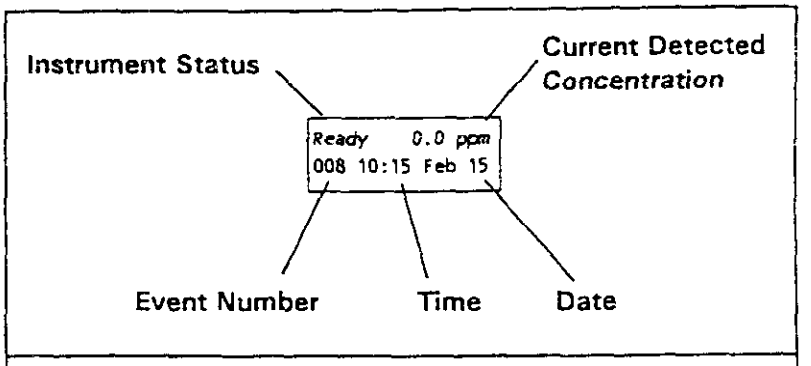


Figure 2 Normal Display

MicroTIP operates automatically. The user reads concentrations directly from MicroTIP's display. The display updates itself each half second.

The Minimum, Maximum, and Average concentrations measured in each 15-second period are automatically recorded in MicroTIP's datalogging memory if the datalogger is turned on. MicroTIP's memory holds 12 hours of concentration data.

Concentration data can be played back from memory on MicroTIP's display, sent to a printer in either tabular or graphical format or sent to a computer in tabular format.

Data are recorded by date, time, and by a sequential Event number. Data are played back by the user specifying a start and a stop Event number.

The keypad is used to set up and calibrate MicroTIP, and allows the user to manipulate the concentrations measured and recorded by MicroTIP in various ways. MicroTIP has 16 clearly labelled keys for direct numeric entry and for using MicroTIP's functions.

All information entered from the keypad and stored in MicroTIP's memory is retained when MicroTIP is switched off. The clock and calendar continue to operate and do not need to be reset the next time MicroTIP is used.

2.2 TUTORIAL SESSION

To assist the user in learning the key functions, MicroTIP has a built-in tutorial session which displays a two-line description of the function of each key. Pressing MicroTIP's TUTOR key begins a tutorial session and pressing EXIT after any key ends the session. While in the tutorial session keypresses have no effect on MicroTIP's operation.

Press the TUTOR key and begin a tutorial session. Press each key and read the display. The tutorial display for each key is given in Table 1.

BATT	Shows battery V normally 6-8.5V	ALARM	Shows set-point for conc alarm
DISPLAY	Displays conc as graph or numeral	MAX	Displays highest conc measured
LIGHT	Shows intensity of detector lamp	SETUP	Sets date time & options for keys
AUDIO	Selects alarm or tone or no audio	EVENT	Sets options for recording data
PRINT	Prints table of recorded data	TUTOR	Press a key then read explanation

Table 1 TUTOR Displays

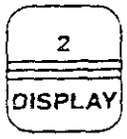
GRAPH	Prints graph of recorded data	CLEAR	Erases the last number pressed
PLAY	Replays recorded data on display	EXIT	Cancels key with no more changes
CAL	Calibrates with zero & span gas	ENTER	Confirms display then continues

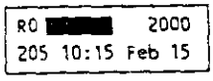
Table 1 - continued

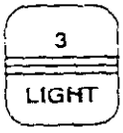
If there are no options to the function then the key acts immediately. If there are options, then the current option is displayed on the lower line. The user is prompted to display the other options by pressing the up arrow or down arrow key. Pressing ENTER confirms that the displayed option is correct. If the function requires numeric input then the current value is displayed on the lower line. The user can change it on the display by pressing the numeric keys. Pressing ENTER confirms that the displayed value is correct.

Some functions have multiple steps for options and/or numeric inputs. These are arranged so that the most frequently changed inputs are displayed first. Once the desired changes have been made the user can bypass the rest of the steps by pressing EXIT.

Each key function is described in more detail in the following sections. Leave MicroTIP on and try each key in turn.







2.3 DISPLAY

If a numerical display is shown, pressing DISPLAY will change it to a bar graph. If the bar graph is shown, pressing DISPLAY changes it to a numerical display. The bar graph range is selected with the SETUP key.

2.4 LIGHT

Pressing the LIGHT key switches the backlighting on to high intensity. The current intensity of the detector UV lamp is also displayed when the LIGHT key is pressed. Pressing the

LIGHT key again decreases the backlighting intensity. Pressing the key for the third time turns the backlighting off. The brighter backlighting consumes more power and is recommended for use only in very dark locations.

The displayed lamp intensity is the current visible intensity of the UV detector lamp. Lamp intensity is typically in the range 1000 to 4000 with a maximum of 4095.

2.5 BATT

Pressing the BATT key displays the current battery level. The battery voltage will be shown for 15 seconds and then the display reverts to normal. The normal operating voltage range is 6 to 8.5 volts.

```
Battery level, V
     8.3
```

When LoBat is displayed there is approximately 10 minutes of operation left. If operation is continued with a low battery pack another message will appear indicating that the batteries are critically low. MicroTIP will now turn off the detector lamp. To continue normal operation replace the discharged battery pack with a fully charged one. Recharge the discharged battery pack for at least 8 hours as soon as it is convenient. This reduces deep discharging of the battery pack.

```
BATTERY PACK
  CRITICALLY LOW
```

Note: Deep discharging of the battery pack will result in loss of recorded data, setup parameters and may cause damage to the battery pack.

2.6 MAX

Press the MAX key. The maximum concentration, the Event during which it was encountered, the time and the date of the occurrence will be displayed. This is shown for 15 seconds and then the display reverts to normal.

```
Max        112 ppm
240 10:12 Feb 15
```

Press MAX then press CLEAR twice to reset the Max register. "Max Cleared" will be displayed with the current date and time. After 15 seconds the display reverts to normal.

```
Press CLEAR
  to reset Max
```

```
Max cleared
240 10:12 Feb 15
```

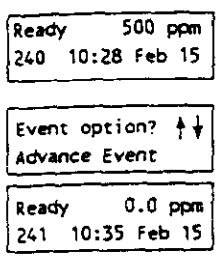
Recording of real time data is not interrupted when the MAX key is pressed or when the Max register is cleared.

2.7 CLEAR

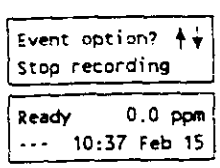
CLEAR erases the last numerical entry. If a number is entered in error press CLEAR to erase the entry and re-enter the correct number. CLEAR used in conjunction with the MAX key resets the Max register.

2.8 EVENT

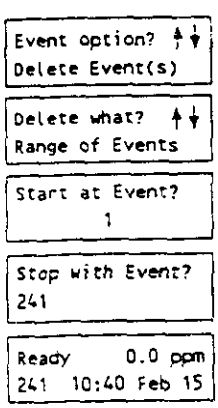
Events may be used to identify a particular sample or sampling location in memory. Recorded data are played, printed and removed from the datalogger by specifying a start and stop Event number.



The EVENT key controls MicroTIP's datalogger. Press the EVENT key to see the Event options. Press the arrow keys until the "Advance Event" option is displayed. To increment the current Event number press ENTER.



Move to the next Event option by pressing the arrow keys. The datalogger can be turned off by selecting the Stop Recording option and pressing ENTER. MicroTIP will continue to display the current detected concentration but no data will be recorded. There will be no event number shown on the normal display. To begin recording again, press EVENT and then ENTER. The Event number is automatically incremented by one when the datalogger or the instrument is turned on.



Press the arrow keys to view the next option. The user can delete the current Event, all recorded Events or a range of Events. Press ENTER then use the arrow keys to select the desired option and press ENTER again. If a range of Events is to be deleted, MicroTIP will ask for the start and stop Event numbers.

To reset the Event counter to 001, select the "Delete all Events" option and press ENTER. After Event 255, MicroTIP resets the Event counter to zero automatically.

Note: Deleted information cannot be recovered. It is a good idea to play back or print the contents of the datalogger before deleting any information.

MicroTIP only stores the last Event number seen in a 15 second period. To assign an Event number to a sample, the Event number should be advanced only once every 15 seconds. If the Event number is advanced more than once in a 15 second period, lower Event numbers will not be stored.

MicroTIP can record continuously for a period of 12 hours. After this time it begins to overwrite existing data one Event at a time. For example: 6 Events of 2 hours each are recorded. Event #7 will overwrite event #1 if it is 2 hours or less in length. If Event #7 is greater than 2 hours it will completely overwrite Event #2 as well. Let's say Event #7 is 3 hours, then Events #3, #4, #5, #6 and #7 are now in the datalogger.

If it is necessary to retain a copy of recorded data, the data should be printed or stored in a computer at least once every 12 hours of operation. This will prevent loss of information when events are overwritten.

2.9 EXIT

The EXIT key terminates all MicroTIP functions except DISPLAY. When EXIT is pressed the display reverts to normal. Most functions exit automatically if no key is pressed for 15 seconds.

When EXIT is pressed during printing or graphing, MicroTIP stops sending information to the printer. The printer will continue to print until its buffer is empty.

2.10 SETUP

The SETUP key allows MicroTIP to be set up for a specific application. The current date and time are also set with the SETUP key.

Press SETUP and step through the options. Press ENTER to accept the displayed data or enter a numerical value using the keypad and then press ENTER. If no values are entered the display reverts to normal. To set up the instrument:

Range 0-? ppm ↑↓
200

1. Press SETUP.

2. The first option sets the full scale range for the bar graph display, the graph output, the audio output, and the 1 volt analog output. Use the up and down arrow keys to select the 20, 200 or 2000 parts per million (ppm) range and press ENTER. This does not affect MicroTIP's numeric display and datalogging

Cal memory ? ↑↓
1

3. MicroTIP has 10 Cal Memories for regular operation and one for High Sensitivity operation. Only one Cal Memory can be used at a time. Select Cal Memory 1 with the up and down arrow keys and press ENTER.

Note: It does not matter which Cal Memory is selected, MicroTIP's response is not specific to any one compound. The reading displayed represents the total concentration of all photoionizable compounds in the sample.

4. Next enter the correct values for the current time. Press ENTER after each value.

5. Enter the numerical values for the day, month and year. Again press ENTER after each selection.

4
AUDIO

2.11 AUDIO

To connect the headset (Photovac Part No. 395030):

1. Remove the dust cap from the I/O connector and plug in the headset.

2. Press the AUDIO key and use the arrow keys to select one of three options for audio output and press ENTER.

Audio output? ↑↓
Off

Audio output? ↑↓
Audio on Alarm

Audio output? ↑↓
Continuous Audio

Audio output can be turned off altogether, set so there is audio output during an alarm condition only, or a continuous audio signal with the tone frequency being proportional to the detected concentration. The volume is controlled by a knob on the headset.

9
ALARM

2.12 ALARM

The ALARM key displays the current alarm level and allows a new alarm level to be entered.

```
Alarm at ? ppm
    100.00
```

1. Press ALARM to display the current alarm level. If this value is correct, press EXIT to return to the normal display.

2. If a new value is to be set, enter the value, and press ENTER.

When an alarm condition is detected the instrument status changes to "Alarm" and an audio signal is heard through the headset (only if "Audio on Alarm" is selected) and remains on until the alarm condition has passed or until it is turned off with the AUDIO key.

7
PLAY

2.13 PLAY

The PLAY key plays back previously recorded data. If either "Audio on Alarm" or "Continuous Audio" is selected the playback audio output (not the real time output) is heard through the headset. To enable playback audio output, press AUDIO and select the desired output before pressing the PLAY key.

```
ENTER to Play
   * for Options
```

1. Press PLAY. Two options are available. Pressing ENTER begins playback where it was last stopped. Press the SETUP (*) key to set the playback options.

```
Play from Event ?
      10
```

2. Enter the start Event. If this Event is not available MicroTIP begins at the next higher Event. An Event may not be available if the Event number was advanced more than once in 15 seconds, or if the selected Event has been deleted or overwritten.

```
Min Avg Max ? ↑↓
Avg
```

3. Next select which value is to be displayed, either the Minimum, the Average, or the Maximum, with the arrow keys and press ENTER.

4. The data can be played back in either numerical or graphical display by pressing the DISPLAY key.

When MicroTIP is playing back recorded data it is also measuring and recording real time concentrations even though the instrument status is "Play". If, during playback, an instrument status with a priority higher than that of "Play" is encountered in real time operation it will be displayed, but MicroTIP will continue to play back.

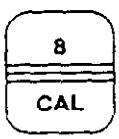
```
Play      100 ppm
010>10:20 Feb 15
```

```
Play      100 ppm
012<10:22 Feb 15
```

The playback speed can be increased or decreased by pressing the arrow keys. Information can also be viewed in the opposite direction, again by pressing the arrow keys. A forward arrow (>) appears in the display if data are being played forward or a backward arrow (<) if the data are being played in reverse.

Press ENTER to freeze the display at any time and use the arrow keys to resume playback. Press EXIT to return to the normal display.

The PLAY function provides a speed search to find the desired start and stop Event numbers for printing or graphing.



2.14 CAL

MicroTIP must be calibrated in order to display concentration in units equivalent to ppm. First a supply of zero air, which contains no ionizable gases or vapors, is used to set MicroTIP's zero point. Then, span gas, containing a known concentration of a photoionizable gas or vapor, is used to set the sensitivity.

Usually clean ambient air will be suitable as zero air. If there is any doubt, use a commercial source of zero grade air and a second sampling bag. Span gas of the desired compound and concentration, required for calibration, may be obtained from a specialty gas supplier. See Appendix B.

Isobutylene at 100 ppm in air is recommended as span gas. To calibrate the instrument use the Calibration Kit (Photovac Part No. 390033) as follows:

1. Connect the supplied regulator to the span gas cylinder. Hand tighten the fittings. Observe proper handling techniques for all gases.

2. Open the valve on the gas bag by turning the valve stem fully counterclockwise.

3. Attach the nut to the regulator. Hand tighten the fittings.

4. Turn the regulator knob counterclockwise about half a turn to start the flow of gas.

5. Fill the gas bag about half full and then close the regulator fully clockwise to turn off the flow of gas.

6. Disconnect the bag from the adapter and empty it. Flush the bag a few times with the span gas and then fill it.

7. Close the gas bag by turning the valve clockwise.

```
Cal memory ? ↑↓
7
```

8. Press SETUP and select the desired Cal Memory with the arrow keys and press ENTER. Press EXIT to return to the normal display.

```
Response factor?
     1.00
```

9. Press CAL and enter the desired response factor. Use Table 2, on page 21, to find the response factor for the compound of interest. If the compound is not in Table 2 or you are not looking specifically for one compound then enter 1.00.

The concentration detected by MicroTIP will be multiplied by the response factor before it is displayed and logged.

```
Connect zero gas
then press ENTER
```

```
Calibrating now,
  please wait...
```

10. Expose MicroTIP to zero air. Press ENTER and MicroTIP sets its zero point.

```
Span conc ? ppm
     100.00
```

11. MicroTIP then asks for the span gas concentration. Enter the known span gas concentration and then connect the span gas bag adapter to the inlet.

```
Connect span gas
then press ENTER
```

12. Press ENTER and MicroTIP sets its sensitivity.

```
Calibrating now,
  please wait...
```

13. When MicroTIP's display reverts to normal, MicroTIP is calibrated and ready for use. Remove the span gas bag from the inlet.

MicroTIP has 10 Cal Memories and can be calibrated with 10 different span gases or response factors if desired. Only one Cal Memory can be used at a time. Each memory stores a different response factor, zero point and sensitivity. To program the Cal Memories:

1. Press SETUP and select the desired Cal Memory (1 to 10) with the arrow keys.

2. Exit from SETUP and press the CAL key.

3. Enter the desired response factor and press ENTER.

Note: It does not matter which Cal Memory is selected or which response factor is entered, MicroTIP's response is **not** specific to any one compound. The reading displayed represents the total concentration of all photoionizable compounds in the sample.

4. Follow the displayed calibration instructions. When the calibration is completed it is automatically stored in the selected Cal Memory. The span gas concentration entered here is specific to the selected Cal Memory.

Whenever the instrument is calibrated, MicroTIP updates the selected Cal Memory only. The instrument should be calibrated at least once a day.

5
PRINT

2.15 PRINT

MicroTIP was designed to be used with an Epson® FX-80 or 100% compatible printer with an RS232 serial interface. If an Epson compatible printer is to be used, the printer cable and suitable adapter (Photovac Part No. 395006) should work.

The printer **must** be set to 8 data bits and 1 stop bit to communicate with MicroTIP. MicroTIP's baud rate and parity must be set depending on the printer requirements. Refer to the printer user's manual for more information.

When MicroTIP is printing data it is also measuring and recording real time concentrations. To print data:

1. Use the printer cable and suitable adapter (Photovac Part No. 395006) to connect the MicroTIP I/O connector to the printer.

```
ENTER to Print
  * for Options
```

2. Press the PRINT key and then the SETUP (*) key to select the desired setup options.

```
Start at Event?
       1
```

```
Stop with Event?
       24
```

3. MicroTIP will ask for the number of the start and stop Events. Enter the desired values and press ENTER.

```
Output format? ↑↓
 Fit on one page
```

```
Output format? ↑↓
 Output all data
```

4. MicroTIP will then ask if the selected data are to be formatted to fit on one 8½" x 11" page or if all recorded data between the selected start and stop Events are to be printed. Use the arrow keys to select the desired option and press ENTER.

```
ID number?
25008
```

5. Enter an ID number if desired. Any number up to 16 digits long may be entered as an ID number. The ID number can be used to differentiate between users or instruments. If more than one user is using a single instrument, each user will use an individual number for printed reports. If more than one MicroTIP is being used, each instrument can have its own ID number on printed reports.

   If an ID number is not required, leave this option blank and move to the next option by pressing ENTER.

```
Baud rate ?   ↑↓
9600
```

```
Parity?       ↑↓
None
```

6. Enter the baud rate and parity. These values are specific to the printer being used and must be set correctly. Again, refer to the printer user's manual to determine the correct baud rate and parity.

7. When the setup is correct, ensure the printer is on line and press ENTER.

If "Fit on one page" is selected, MicroTIP will divide the selected data into 54 averaging intervals. One interval is printed on each line.

MicroTIP stores one set of readings (Min, Avg and Max) each 15 seconds. In each averaging interval the printed minimum is the minimum of all the stored readings in that

Photovac MicroTIP Statistical Monitoring Report

ID Number: Averaging Interval: 39 Readings 585 Seconds

Date	Time	Min	Avg	Max	Event	Status	Notes
Feb 03.92	12:10	0.0	0.0	[illegible]	[illegible]	Ready	
	12:19	0.0	0.7	[illegible]		Ready	
	12:29	2.0	[illegible]	3.9		Ready	
	12:39	4.0	4.4	[illegible]		Ready	
	12:49	4.2	4.8	[illegible]		Ready	
	12:58	[illegible]	4.4	[illegible]		Ready	
	13:08	4.9	5.4	[illegible]		Ready	
	13:18	5.3	5.7	[illegible]		Ready	
	13:28	4.5	5.2	[illegible]		Ready	
	13:37	4.3	4.7	[illegible]		Ready	
	13:47	[illegible]	4.3	[illegible]		Ready	
	13:57	4.5	[illegible]	[illegible]		Ready	
	14:07	5.7	[illegible]	[illegible]		Ready	
	14:16	6.9	8.3	[illegible]		Ready	
	14:26	8.5	[illegible]	[illegible]		Ready	
	14:36	[illegible]	[illegible]	[illegible]		Fault	
	14:46	[illegible]	[illegible]	[illegible]		Ready	
	14:55	[illegible]	[illegible]	[illegible]		Ready	
	15:05	[illegible]	[illegible]	[illegible]		Ready	
	15:15	[illegible]	[illegible]	[illegible]		Ready	
	15:25	[illegible]	[illegible]	[illegible]		Fault	
	15:34	[illegible]	[illegible]	[illegible]		Fault	
	15:44	[illegible]	[illegible]	[illegible]		Ready	
	15:54	[illegible]	[illegible]	[illegible]		Ready	
	16:04	[illegible]	10.0	[illegible]		Ready	
	16:13	[illegible]	10.1	[illegible]		Ready	
	16:23	[illegible]	10.4	[illegible]		Ready	
	16:33	[illegible]	[illegible]	[illegible]		Ready	
	17:40	0.0	[illegible]	[illegible]	037	Ready	
	17:49	[illegible]	[illegible]	[illegible]		Ready	
	17:59	[illegible]	[illegible]	[illegible]		Ready	
	18:09	0.0	[illegible]	[illegible]		Cal	
	18:19	3.5	4.0	[illegible]		Ready	
	18:28	2.7	[illegible]	[illegible]		Ready	
	18:38	2.4	3.9	[illegible]		Ready	
	18:48	[illegible]	7.9	[illegible]		Ready	
	18:58	3.2	8.6	[illegible]		Ready	
	19:07	4.1	4.8	[illegible]		Ready	
	19:17	2.8	7.7	[illegible]		Ready	
	19:27	[illegible]	[illegible]	[illegible]		Ready	
	19:37	2.7	3.3	[illegible]		Ready	
	19:46	[illegible]	3.8	[illegible]		Ready	
Feb 04.92	08:00	0.0	[illegible]	[illegible]	039	Cal	
	08:06	0.0	[illegible]	2500	040	Cal	
	11:34	0.0	170	2500	042	Cal	
	12:03	[illegible]	14.0	468		Alarm	
	12:13	[illegible]	-0.3	500		Alarm	
	12:23	[illegible]	[illegible]	493		Alarm	
	12:33	[illegible]	[illegible]	464		Alarm	
Feb 17.92	17:37	0.0	87.6	[illegible]	[illegible]	Fault	
	17:46	370	[illegible]	[illegible]		Fault	
Feb 24.92	12:42	0.0	321	2500	030	Fault	

Figure 3 Printed Output

interval. The printed Avg is the average of all the recorded average readings and the Max is the maximum of all the recorded maximum readings.

The following information is printed:

a. The number of readings in an interval and the length of the interval are printed at the top of the page. In Figure 3 there are 39 readings in an interval and the interval is 585 seconds.

b. The interval start time. The first interval in Figure 3 begins at 12:10.

c. The lowest Event number in the interval, only if the Event number has changed.

d. The highest priority instrument status of the interval. See page 38 for a description of instrument status.

e. Space for the user to add Notes to the report. Notes could include identification of particular samples or sampling location based on Event numbers.

If "Output all data" is selected the header, containing the title, averaging interval and column headings, will only be printed on the first page of the printed output. Each of the following pages will contain 66 lines of data.

```
Printing now
 please wait...
```

To print all 12 hours of recorded data will require about 45 pages and take about 10 minutes. The actual printing time will depend on the printer and print quality. While the information is being printed, the display shows that printing is in progress. The keypad will not accept commands until the present print job has been completed.

Pressing EXIT during printing stops the job and the display reverts to normal. The printer will continue to print until its buffer is empty.

6

GRAPH

2.16 GRAPH

Pressing the GRAPH key prints the recorded data in graphical format. When MicroTIP is graphing recorded data it is also measuring and recording real time concentrations. To graph logged data:

```
Range 0-? ppm ↑↓
200
```

1. Press SETUP to set the range for the graph. Use the arrow keys to select the 20, 200 or 2000 ppm range.

2. Use the printer cable and suitable adapter (Photovac Part No. 395006) to connect the MicroTIP I/O connector to the printer.

```
ENTER to Graph
  * for Options
```

3. Press the GRAPH key and then the SETUP (*) key to select the desired setup options.

```
Star. at Event?
      1
```

```
Stop with Event?
      24
```

4. MicroTIP will ask for the number of the start and stop Events. Enter the desired values and press ENTER.

5. Select which values are to be graphed. Use the arrow keys to select from:

```
Min Avg Max ? ↑↓
Avg
```

a.	Min	e.	Min & Max
b.	Avg	f.	Avg & Max
c.	Max	g.	Min & Avg & Max
d.	Min & Avg		

```
Output format? ↑↓
Fit on one page
```

```
Output format? ↑↓
Output all data
```

6. MicroTIP will then ask if the selected data are to be formatted to fit on a single 8½" x 11" page or if all recorded data between the selected start and stop Events are to be graphed. Use the arrow keys to select the desired option and press ENTER.

```
ID number?
25008
```

7. Enter an ID number if desired. Any number up to 16 digits long may be entered as an ID number. The ID number can be used to differentiate between users or instruments. If more than one user is using a single instrument, each user will use an individual number for printed reports. If more than one MicroTIP is being used, each instrument can have its own ID number on printed reports.

   If an ID number is not required, leave this option blank and move onto the next option by pressing ENTER.

```
Baud rate ?   ↑↓
9600
```

```
Parity?       ↑↓
None
```

8. Enter the baud rate and parity. These values are specific to the printer being used and must be set correctly. Again, refer to the printer user's manual to determine the correct baud rate and parity.

9. When the setup is correct, ensure the printer is on line and press ENTER.

If "Fit on one page" is selected, MicroTIP will divide the selected data into the appropriate averaging interval. Sixteen intervals are graphed on each line.

MicroTIP always stores one set of readings (Min, Avg and Max) each 15 seconds. In each averaging interval the

graphed mininum is the minimum of all the stored readings in that interval. The graphed Avg is the average of all the recorded average readings and the Max is the maximum of all the recorded maximum readings. The following information is printed:

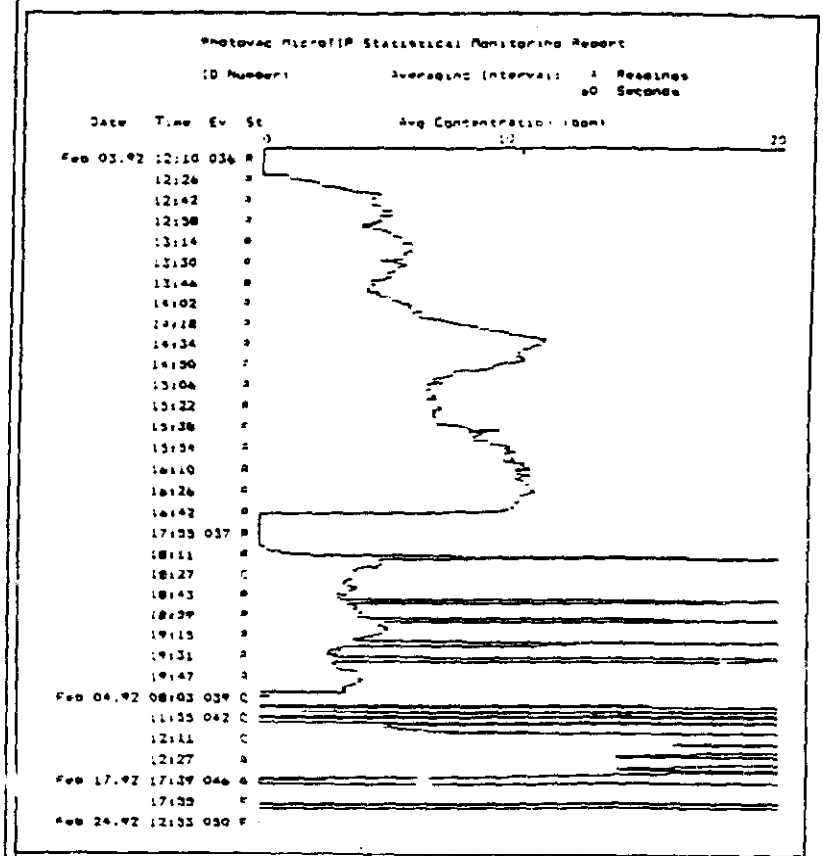


Figure 4 Graphed Output

a. The number of readings in an interval and the length of the interval are printed at the top of the page. In Figure 4 there are 4 readings in an interval and the interval is 60 seconds.

b. Time is printed once every 16 intervals or every 16 minutes for the example in Figure 4. This time will be the start time of the next 16 intervals.

E-24

c. The lowest Event number of the 16 intervals is printed, only if it has changed from the previous set of 16 intervals.

d. The highest priority instrument status code of the 16 intervals is printed. See page 38 for details of instrument status.

If "Output all data" is selected the header, containing the title, averaging interval and column headings, will only be printed on the first page of the graphed output.

To graph all 12 hours of recorded data will require about 4 pages and take about 10 minutes. The actual printing time will depend on the printer and print quality.

```
Printing now
 please wait...
```

While the information is being printed, the display shows that printing is in progress. The keypad will not accept commands until the present print job has been completed.

Pressing EXIT during printing stops the job and the display reverts to normal. The printer will continue to print until its buffer is empty.

2.17 HIGH SENSITIVITY OPERATION

MicroTIP can be used as a high sensitivity leak detector. In High Sensitivity operation, MicroTIP does not read directly in ppm but displays a reading proportional to the total concentration of photoionizable gases and vapors detected.

During calibration, no span gas is required. MicroTIP zeros its reading with zero air and then sets itself to the maximum sensitivity.

```
Range 0-? ppm ↑↓
200
```

```
Cal memory ?  ↑↓
High Sensitivity
```

```
HO ▬▬▬▬▬▬▬▬ 20
244 11:15 Feb 15
```

1. Press SETUP. Select the 0-20 ppm display range with the arrow keys and press ENTER.

2. Select High Sensitivity with the arrow keys and press ENTER.

3. Press EXIT. Select the bar graph with the DISPLAY key.

4. Press CAL and calibrate MicroTIP with zero air. High

Sensitivity operation does not require span gas.

```
Audio output? ↑↓
Continuous Audio
```

5. Press AUDIO and select "Continuous Audio" with the arrow keys.

As MicroTIP samples air closer to the leak, the length of the shaded area on the display increases. When used with the optional headset (Photovac Part No. 395030), the frequency of the tone increases as air closer to the leak is sampled, and the user need not watch the display.

MicroTIP's 3 second response time and detection limit of 0.1 ppm isobutylene permit fast detection of small leaks.

2.18 RESPONSE FACTORS FOR GASES AND VAPORS

In situations where only a single pure compound is present in air, MicroTIP should be calibrated with a standard of that specific compound as span gas. MicroTIP's 10 Cal Memories can be used to store calibration information for 10 different span gases.

MicroTIP's reading will always be influenced by any other photoionizable compounds present in the air sample. Even if MicroTIP has been calibrated with a specific compound, its response is not specific and the presence of another impurity may render the numerical result invalid.

It is often impractical to carry a range of different standards into the field. Approximate results can be obtained by calibrating MicroTIP with the recommended span gas and entering the appropriate response factor. The response factor is based on the ratio of the response of the specific compound to the response of the span gas. The response factor multiplies MicroTIP's reading then displays and records it (if the datalogger is on).

Table 2 gives response factors from which approximations can be made for guidance purposes. Data extrapolated from the use of response factors must be regarded as interim and approximate only. Table 2 should be used only for concentrations up to 100 ppm of the specific compound, because response factors change with concentration.

To use the data in Table 2:

1. Press the CAL key and enter the response factor for the specific compound.

2. Calibrate MicroTIP with zero air and 100 ppm span gas as described in Section 2.14.

3. Expose MicroTIP to the sample. The displayed reading is the approximate concentration of the specific compound.

Compound	Response Factor
Acetone	1.2
Benzene	0.6
Butyl Acetate	2.9
Cyclohexane	1.9
Cyclohexanone	0.9
Ethyl Acrylate	3.3
n-Heptane	3.7
Methyl Ethyl Ketone	0.9
Methyl Isobutyl Ketone	1.1
Methyl Methacrylate	1.5
n-Octane	2.6
Perchloroethylene	0.7
Styrene	0.5
Toluene	0.5
Trichloroethylene	0.6

Table 2 Response Factors

Note: These response factors serve as a guide to concentrations measured by MicroTIP with a 10.6 eV lamp. Results are expected to be accurate to within +/- 10ppm or +/-25% of result, whichever is greater. Accuracy of response factors to other gases and vapors may differ from that stated.

These responses are measured relative to isobutylene span gas.

Chapter 3 Accessories and Other Devices

3.1 PRINTER

MicroTIP is designed to interface with an Epson FX-80 or 100% compatible printer with an RS232 serial interface. The printer cable (Photovac Part No. 395006) has been configured for this application only. If the printer being used is compatible with the Epson FX-80, then the supplied cable should work. If it does not work then a different cable may be required or the existing one may require modification. If you are unsure of the compatibility of the printer refer to the printer user's manual or contact the printer manufacturer.

In order to modify the existing printer cable the pinout information for the printer connector is required. Refer to the printer user's manual for specific details. The pinouts for the MicroTIP printer cable are shown in Figure 5 and are listed in Table 3. If modifying the cable is not possible, take the electrical and pinout information to a computer store where a suitable adapter may be obtained.

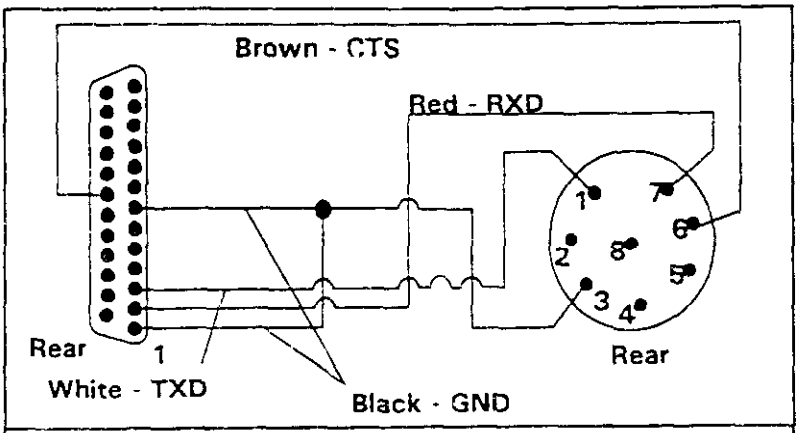


Figure 5 Printer Cable Electrical Connection

Once the proper cable has been obtained, ensure the printer is set to 8 data bits and 1 stop bit for communication and that the appropriate baud rate and parity have been selected. Use the PRINT and GRAPH keys as detailed in Sections 2-15 and 2-16.

MicroTIP I/O Conn. (8 pin) Pin #	MicroTIP Printer Cable (DB25) Pin #	Standard RS-232	Color
1	3	TXD	White
2	—	—	—
3	1 & 7	GND	Black
4	—	—	—
5	—	—	—
6	20	CTS	Brown
7	2	RXD	Red
8	—	—	—

Table 3 Printer Cable Pinouts

3.2 COMPUTER

MicroTIP will send information stored in its datalogger to a computer. The computer must be set up to emulate a terminal. The use of Crosstalk®, a communications software package, is recommended.

MicroTIP is set to 8 data bits and 1 stop bit for communication. The appropriate baud rate and parity must be selected. Use the PRINT key as detailed in Section 2-15.

Use the printer cable and a suitable adapter (Photovac Part No. 395006) to connect MicroTIP to the computer's serial port. The serial port may be labelled Com1 or Com2. Refer to the computer user's manual to determine the baud rate and parity as these values will depend on the computer.

The baud rate can be set from 300 to 19200. The parity can be set to Odd, Even or None and again will depend on the computer. The port number will be the computer port to which MicroTIP is connected. Stop Bits must be set to 1. Correspondingly, Data Bits must be set to 8.

The following instructions are for Crosstalk Mark IV Version 2.0. The commands may vary depending on which version of Crosstalk is being used.

To initiate communications between MicroTIP and the computer:

1. Start Crosstalk. From the dialing directory select DIRECT and press Alt-S. A dialing directory entry will be created for communications with MicroTIP.

2. *Enter the name of the dialing directory entry. Type in* MicroTIP as the name.

3. Leave the Description as Direct <hardwired> connection.

4. Leave the Number option blank as there is no telephone number to be dialed.

5. Press the spacebar to turn the Capture option on. This option will store the data received from MicroTIP to the specified download directory.

6. Type in the Download Directory. This is the disk drive and directory, on the computer, to which MicroTIP data will be stored.

7. The Device should be selected as Modem, even though a modem is not being used.

8. Enter the Computer Port to which MicroTIP has been connected. The port number will depend on how Crosstalk was configured when it was installed. Ensure MicroTIP has been connected to a serial port. See Section 5.3. MicroTIP will not work with a parallel port.

9. Set the desired speed. Ensure it matches the baud rate set on MicroTIP.

10. Set the WordFormat. Stop Bits must be set to 1, and Data Bits to 8. Parity may be set to odd, even or none depending on the computer.

11. Press Crtl-Enter to save the dialing directory entry. This entry may be saved with Crosstalk. Whenever Crosstalk is run, the MicroTIP entry will appear in the dialing directory. If nothing has changed this entry

may be used every time MicroTIP is connected to the computer. This will save the user having to set up Crosstalk each time.

12. Ensure the MicroTIP entry is selected in the dialing directory and press Enter.

13. Ensure the MicroTIP print setup matches that entered in the Crosstalk dialing directory and then press the MicroTIP PRINT key. The data will now appear in the Crosstalk communications window. It will be stored as it appears on the screen.

14. When all the data have been sent press Alt-C and turn the capture option off and then press Enter.

15. Press Alt-O to end the communications session.

The captured file will have an extension that corresponds to the current date. Convert this file to a text file using the MS-DOS® Copy Command:

16. At the DOS prompt type:
    Copy MICROTIP.814 MICROTIP.prn

17. The newly created file MICROTIP.prn can now be imported into Lotus 1-2-3® as a text file. See the Lotus 1-2-3 User's Manual for details.

3.3 CHART RECORDER

MicroTIP's output can be displayed as a 0 to 1 volt analog voltage on a chart recorder in real time. Set the chart recorder to 1V full scale and connect it to MicroTIP's I/O connector using the analog output cable (Photovac Part No. 395005). The concentration range of the analog output signal is selected with the SETUP key, and can be set to 20, 200, or 2000 ppm full scale.

3.4 SAMPLE BAG

MicroTIP is equipped with a sample outlet fitting (See Figure 1) from which samples may be collected for further analysis. Connect a sample bag to the fitting with a short length of 1/8" inside diameter flexible tubing.

Note: Readings may fluctuate due to changes in detector flow rate as the sample bag is filling.

The bag contents will not perfectly represent the sample. Ozone produced by MicroTIP's detector will be present, and sample composition may have been altered by passage through MicroTIP's sampling pump.

3.5 THREE METER (9.8') SAMPLE LINE

For remote sampling, connect the 3m (9.8') sample line (Photovac Part No. 390006) to MicroTIP's sample inlet in place of the 17cm (6.8") sample line supplied.

3.6 SHOULDER STRAP

Snap one end of the shoulder strap to the steel shoulder strap connector bail above MicroTIP's I/O connector. Snap the other end to one of the shoulder strap connectors beside the display. The connection point is selectable for right or left handed operation. Adjust the shoulder pad and strap length for comfort.

3.7 HEADSET

To connect the headset:

1. Remove the dust cap.
2. Connect the headset (Photovac Part No. 395030) to the I/O port and tighten the locking nut.
3. Use the knob on the headset to adjust the volume.

Operation of the headset is described in Sections 2.11-2.13.

3.8 REPLACEMENT DETECTOR LAMPS

MicroTIP is supplied with an ultraviolet (UV) lamp which produces an energy of 10.6 electron-volts (eV). With this

standard lamp installed, MicroTIP responds well to gases and vapors which ionize at 10.6 eV or less. Some of these are listed, with their response factors, in Table 2 on page 21. For special applications, MicroTIP's response can be changed by using other lamps.

With a 9.5 eV lamp, MicroTIP responds well only to gases and vapors which ionize at 9.5 eV or less. Some response factors with a 9.5 eV lamp are listed in Table 4 on page 28. To use the 9.5 eV lamp, MicroTIP must be calibrated with a compound which ionizes at below 9.5 eV. Toluene at 100 ppm in air is recommended.

Table 4 gives response factors from which approximations can be made for guidance purposes. Data extrapolated from the use of response factors must be regarded as interim and approximate only. Table 4 should be used only for concentrations up to 100 ppm of the specific compound, as response factors change with concentration. Data from Table 4 may be used in the same way as those from Table 2 if a single pure compound in air is being sampled.

1. Press the CAL key and enter the response factor for the specific compound.

2. Calibrate MicroTIP with zero air and 100 ppm toluene span gas as described in Section 2.14.

3. Expose MicroTIP to the sample. The displayed reading is the approximate concentration of the specific compound.

In a situation where a range of gases and vapors exists, MicroTIP displays a reading representing the total concentration of photoionizable compounds present. The change in the response factor given by the 9.5 eV lamp is especially useful when sampling mixtures, for example, a mixture of benzene and trichloroethylene. Both compounds are detected with MicroTIP, and benzene is the more toxic of the two. For health protection, a relatively higher response to benzene and a lower response to trichloroethylene are desired in order to detect lower concentrations of benzene in the presence of trichloroethylene.

COMPOUND	RESPONSE FACTOR
Acetone	20
Benzene	1.2
Methyl Isobutyl Ketone	2.5
Perchloroethylene	1.8
Toluene	1.0
Trichloroethylene	4.0

Table 4 Response Factors with 9.5 eV Lamp

Note: These response factors serve as a guide to concentrations measured by MicroTIP with a 9.5 eV lamp. Results for compounds with response factors of 3.3 and below are expected to be accurate to within +/- 10ppm or +/-25% of result, whichever is greater. Results for compounds with response factors above 3.3 are expected to be accurate to within +/- 40% of result. Accuracy of response factors for other gases and vapors may differ from that stated.

These responses are measured relative to toluene span gas.

The response factors in Tables 2 and 4 are inversely proportional to MicroTIP's response to a specific chemical, that is, a low response factor means that MicroTIP has a high response.

With a 10.6 eV lamp, the response factor of benzene is 0.6 and that of trichloroethylene is 0.6 (from Table 2).

10.6 eV response ratio (benzene/trichloroethylene) from Table 2:

$$= \frac{1/0.6}{1/0.6}$$

$$= 1$$

With 9.5 eV lamp, the response ratio (benzene/trichloroethylene) from Table 4:

$$= \frac{1/1.2}{1/4.0}$$
$$= 3.3$$

The response ratio for the 9.5 eV lamp is about three times that for the 10.6 eV lamp. Because of this difference in response, benzene vapor from the mixture contributes more to the MicroTIP reading with the 9.5 eV lamp than it does with the 10.6 eV lamp. MicroTIP with the 9.5 eV lamp is therefore better suited to detecting benzene in the presence of trichloroethylene.

The 9.5 eV lamp is installed, maintained, and removed according to the instructions in Section 4.2.

With an 11.7 eV lamp installed, MicroTIP functions as a leak detector responding to gases and vapors which ionize at 11.7 eV or less. This lamp is intended for High Sensitivity operation only. It is not suitable for direct-reading use, because of limitations of the lamp window material.

The 11.7 eV lamp window material is Lithium Fluoride (LiF). Unlike other lamp windows, LiF readily absorbs water from atmospheric humidity. When contaminated by moisture, the window loses its ability to transmit UV light.

Note: Never touch the window or let liquid water near it.

Furthermore, LiF is composed of two light elements which are easily disrupted within the crystal lattice by the same UV light generated by the lamp. Disruption of the lattice causes the crystal to turn a yellowish color, and again performance declines.

The 11.7 eV lamp is useful for detecting leaks of chemicals not ionized by the 10.6 eV lamp.

Carbon tetrachloride can be used as an example. Set MicroTIP to display a bar graph. The length of the shaded area on the display represents the concentration of carbon tetrachloride (and other ionizables) in the sample. As MicroTIP samples air closer to the carbon tetrachloride source, the display indicates higher concentrations.

Numerical values shown on MicroTIP's display should not

E-3C

be interpreted as parts per million concentrations. They indicate only the relative presence or absence of ionizable gases and vapors. MicroTIP's 3 second response time and detection limit of 10 ppm carbon tetrachloride permit fast determination of small leaks.

Because of the lamp window limitations, the lifetime of the 11.7 eV lamp is restricted and it must be used sparingly according to these instructions:

1. Remove the 11.7 eV lamp from the supplied dessicant bottle and install the lamp according to the instructions Section 4.2.

2. Switch on MicroTIP, wait for it to warm up, and press SETUP.

```
Range 0-? ppm ↑↓
200
```

3. Select the C-20 ppm range with the arrow keys and press ENTER.

```
Cal memory ?  ↑↓
High Sensitivity
```

4. Select High Sensitivity with the arrow keys and press ENTER.

```
H0 ██████     20
244 11:15 Feb 15
```

5. Press EXIT and then select the bar graph with the DISPLAY key.

```
Connect zero gas
then press ENTER
```

6. Press CAL and calibrate MicroTIP with zero air. High Sensitivity operation does not require a response factor or span gas.

7. Every 15 minutes of operation, recalibrate MicroTIP with zero air.

8. Every hour of operation, switch off MicroTIP and examine the lamp window for yellowing. If the window is yellow, then remove the lamp and regenerate the window according to the procedure below.

9. After use, remove the lamp from MicroTIP and store it in the supplied dessicant bottle.

To regenerate the 11.7 eV lamp window:

1. Clean the lamp window with dry aluminum oxide powder on a dry cotton swab. Do not use methanol

or water.

Aluminum oxide may be obtained from most chemical supply companies. When ordering specify 3.0 micron powder.

2. Heat the lamp in a 150°C (300°F) oven for 8 hours or more.

3. Allow the lamp to cool before use.

4. Alternatively, the lamp window can be regenerated without heat by storing the lamp in a dessicator for at least 5 days.

Chapter 4 Routine Maintenance

4.1 CHARGING THE BATTERY

LoBat	100 ppm
250 14:20	Feb 15

When the instrument status reads LoBat, the MicroTIP battery pack requires recharging. A fully charged battery powers MicroTIP for 7 hours. If the instrument is to be used for more than 7 hours, carry a spare battery pack. When the first one has been discharged, replace it with the spare. Upon return from field work, recharge both battery packs as outlined in Section 1.2. Two chargers are required to do this overnight. Use only the MP-1000 battery charger (Photovac Part No. 395016/17).

The charger automatically charges at a high charge rate until the battery is fully charged and then maintains the full charge with a low continuous charge rate indefinitely so there is no danger of over-charging.

Note: Leaving MicroTIP for more than three days without a charged battery pack will result in loss of recorded data and setup parameters. To avoid loss of data, charge the battery pack for at least 8 hours and put it back onto the instrument.

If MicroTIP is not used regularly, the battery should be charged at least once a month for between 8 and 72 hours.

4.2 CLEANING THE LAMP WINDOW

During the course of normal operation a film builds up on the window of the detector lamp. The rate at which the film develops depends on the type and concentration of the gases and vapors being sampled and results from the UV light interacting with them. As a guide, clean the window every 24 hours of operation.

1. Ensure the instrument is turned off.

2. Hold the black detector housing in one hand and unscrew it from the body of MicroTIP. Remove the housing, being careful not to lose the o-ring seal on top of the photoionization detector. The detector cell, lampholder, and high frequency (HF) driver

E-38

circuit board are now exposed.

3. Unplug the red and yellow wires from the HF driver circuit board.

4. Locate the black ground wire. Loosen the screw on the HF driver circuit board and disconnect the black wire.

5. Hold the lampholder in one hand so it will not rotate and carefully unscrew the detector cell with the red, yellow and black wires attached.

Note: **Do not** touch the fine wire mesh inside the detector cell. Any dust or dirt in the detector cell can be blown out with a gentle jet of compressed air.

6. Leaving the lamp spring in place, remove the lamp from the lampholder.

7. To remove the film, gently rub the window of the lamp with a lint free tissue moistened with methanol. Do not apply the methanol directly to the lamp.

   Use only HPLC grade or spectroscopic grade methanol to clean the lamp window.

8. Allow the window to dry and then, without touching the window, replace it in the lampholder.

9. Replace the detector cell squarely on the lampholder. Finger tighten only. Do not over-tighten.

10. Replace the black wire below the screw on the HF driver circuit board and tighten the screw down. Plug the yellow wire onto the gold pin and the red wire onto the silver pin on the HF driver circuit board.

11. Check the lampholder and ensure it is securely seated by hand. Check that the o-ring seal is in position

12. Replace the detector housing and tighten by hand.

Calibrate MicroTIP and then continue normal operation.

4.3 REPLACING THE DETECTOR UV LAMP

```
Fault      0.0 ppm
251 14:50  Feb 15
```

```
Detector light
 intensity is low
```

If the lamp has failed then it requires replacement:

1. Remove the lamp as outlined in Section 4.2.
2. Remove the lamp spring from the lampholder and replace it with the new lamp spring.
3. Without touching the window of the new lamp carefully place it in the lampholder.
4. Replace the detector cell and the detector housing as outlined in Section 4.2.

Once calibrated, MicroTIP is ready for operation.

4.4 REPLACING THE INLET FILTER

MicroTIP is equipped with a dust filter to reduce detector contamination. As the filter collects dust, MicroTIP's inlet flow rate and sensitivity decrease. Replace the filter every 240 hours of operation, or more frequently if MicroTIP is used in a dusty environment. Do not operate MicroTIP without an inlet filter.

1. Turn the instrument off. Hold the filter housing near the detector housing with a $^{9}/_{16}$" wrench.
2. Unscrew the top of the filter housing with another $^{9}/_{16}$" wrench. Be careful not to lose the metal sealing washer.
3. Remove the spring and filter and install the new filter, open end first. Press the filter into place so it will seal at the bottom of the filter housing. Replace the filter spring and the top of the filter housing. Tighten the top nut while holding the bottom one stationary with the wrench.
4. Once calibrated, MicroTIP is ready for operation.

4.5 REPLACING THE SAMPLE PUMP

Replace the sample pump after every 5000 hours of use.

1. Turn the instrument off and remove the battery pack.

2. Place MicroTIP upside down on a clean, flat surface and remove the 4 screws securing the handle to the control housing. Now remove the two screws in the handle flare.

3. Gently lift the handle away from the control housing. The two parts remain connected by the wires from the I/O connector.

4. Locate the sample pump mounted on the bottom of the power supply printed circuit board. See Figure 6.

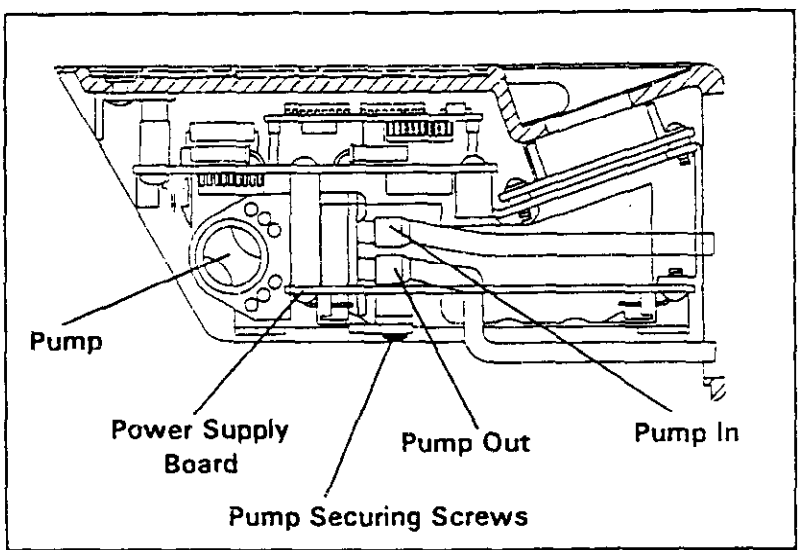


Figure 6 Pump Placement

5. Disconnect the 2-pin pump connector from the power supply board and then remove the 2 screws securing the pump to the power supply board.

6. Gently pull the pump out about 1/2" and remove the 2 pieces of clear tubing from the pump.

7. Connect the clear tubing to the new pump and slide the new pump into position.

8. Secure the new pump with the 2 screws.

9. Connect the 2-pin connector from the new pump to the socket on the power supply board.

10. Carefully position the handle over the control housing and replace the 6 screws in the bottom of the handle and in the handle flare. Ensure that no wire is pinched between the handle flare and the control housing.

11. Snap the battery pack into place. Turn the MicroTIP on and calibrate it.

Chapter 5 Troubleshooting

5.1 IF MICROTIP DRAWS IN LIQUID

MicroTIP accepts only gas and vapor samples. Aspirating a liquid may result in damage to the lamp and the pump. If water is drawn in, the affected parts of the instrument may be cleaned and dried. Contact Photovac Service if another liquid is aspirated.

1. Before taking the instrument apart allow MicroTIP to run until no more liquid comes out of the sample outlet fitting below the detector housing. This will clean out the pump.

2. Turn the instrument off. Remove the detector cell and lamp as outlined in Section 4.2.

3. Dry the lamp with a clean lint free tissue and clean the window. See Section 4.2.

4. Clean the detector cell in distilled water, preferably in an ultrasonic cleaner for no more than 5 minutes.

Note: **Do not** touch the fine wire mesh in the detector cell. **Do not** use solvents as they will degrade the detector cell.

5. Dry the detector cell overnight at 50°C (125°F).

6. Dry the inside of the lamp holder.

7. Remove the filter cartridge as in Section 4.4 and dry the inside of the filter housing.

8. Install a new filter (Photovac Part No. 395000) and re-assemble the filter housing.

Once calibrated, MicroTIP is ready for operation.

5.2 INSTRUMENT STATUS AND FAULT DISPLAYS

The instrument status appears at the left of the upper line of the display and on the Print and Graph outputs. Each status has a priority assigned to it. If more than one status

is in effect, then the status with the highest priority is displayed until the condition is corrected or until the option is turned off.

Status	Code	Priority	Description
Fault	F	1	One of 3 faults is occurring. Press TUTOR for details.
Cal	C	2	Will never be observed on the display during normal operation as various calibration prompt messages are displayed while MicroTIP is calibrating. If the instrument is turned off when it is calibrating with span gas, Cal will appear on the display when MicroTIP is turned on again indicating the last calibration was incomplete. Cal status is also shown on printed and graphed output.
Over	O	3	Detected concentration exceeds 2500 on the display.
Alarm	A	4	Detected concentration exceeds the set alarm level.
LoBat	L	5	Battery pack power is low. Recharge or replace pack.
Play	P	6	The instrument is playing back previously recorded data.
HiSens	H	7	High Sensitivity operation.
Ready	R	8	Normal operation.

Table 5 Instrument Status

When the bar graph display or the Graph output is selected, the instrument status is reduced to a one-letter code. Table 5 summarizes the instrument status and priority.

When the Fault status is displayed, MicroTIP's operation is compromised. Press the TUTOR key for a two-line description of the fault.

Detector light intensity is low

Fault: Detector light intensity is low

Cause: Defective UV lamp.
Action: Replace the UV lamp. See Section 4.3.

Cause: Poor connection between lamp holder and HF driver circuit board.
Action: Check the green wire joining lamp holder to HF driver circuit board. Ensure it is securely connected to both the lampholder and to the circuit board.

Signal from zero gas is too high

Fault: Signal from zero gas is too high

Cause: Contamination of sample line or fittings before the detector.
Action: Clean or replace the sample line or the inlet filter. See Section 4.4.

Cause: Span and Zero gases mixed up.
Action: Ensure clean gas is used to zero MicroTIP and that the span gas is of a reliable concentration.

Cause: Contamination in the detector.
Action: Clean the detector. See Section 5.1.

Cause: Short circuit in the detector.
Action: Ensure resistance between red wire and yellow wire exceeds 10 megohms while the detector is in place.

Detector field voltage is low

Fault: Detector field voltage is low

Cause: Internal fault in the electronics.
Action: Contact Photovac Service at (516)254-4283.

5.3 ESTABLISHING COMPUTER COMMUNICATIONS

If, after having followed the procedure in Section 3.2, communications cannot be established with a computer, the problem may lie with the hardware connections or the printer cable configuration.

1. Ensure MicroTIP is connected to the serial port of the computer.

   The serial port will usually be a male connector, typically 9 pins on an IBM-AT® and compatibles and 25 pins on an IBM-XT® and compatibles. The 25 (or more) pin female connector is usually a parallel port. MicroTIP **cannot** be connected to a parallel port.

   An exception to this rule is Tandy® Computers, which use a female 25 pin connector for the serial port.

2. Ensure the cable being used is compatible with the device.

   An IBM-AT and compatibles with a 9 pin serial connector will first require a null modem to switch pins 2 and 3. Next a gender changer that converts the male DB25 connector on the printer cable from male 25 pins to female 9 pins is required. These two adapters have been combined into one, which is supplied with the MicroTIP printer cable.

   An IBM-XT and compatibles should not require a null modem but will require a gender changer. A gender changer will convert the male DB25 connector on the printer cable to a female connector. A gender changer is supplied with the MicroTIP printer cable.

   The pin definitions of interest are listed in Table 6. Only pins 2 and 3 are shown since these are the problem pins:

Pin #	MicroTIP	IBM-AT	IBM-XT
2	Rxd	Rxd	Txd
3	Txd	Txd	Rxd

Table 6 Pin Definition

Pins 2 and 3 should be **mismatched** between MicroTIP and the computer. Ensure this is the case. It is also possible that the cable being used may switch pins 2 and 3 even if it is not necessary. i.e. the cable is a null modem. IBM-XT cables are usually null modems, since a null modem is required for connection to a printer.

If you are using an IBM-AT and find that a null modem is not required it is possible that an IBM-XT serial port has been added to an expansion slot and thus does not require a null modem. The opposite may be the case if an IBM-AT serial port was added to an IBM-XT expansion slot, in which case the null modem is required.

3. Ensure all hardware is working properly.

Use a printer to test both MicroTIP and the computer. Connect MicroTIP to the printer and ensure that this arrangement produces the desired results. If correct printout is obtained then the MicroTIP and the printer cable are okay.

Now connect the computer to the printer and ensure this works. If the desired printout is obtained this ensures the computer is sending data correctly.

Chapter 6 Technical Description

6.1 OVERVIEW

MicroTIP is a microprocessor controlled instrument for measuring the presence of photoionizable chemicals in air at parts per million levels. The block diagram in Figure 7 shows the main components of MicroTIP. The microprocessor controls the components of the instrument and interprets and records the signal generated by the photoionization detector (PID). Recorded data and setup information entered into the microprocessor's memory are retained when MicroTIP is turned off.

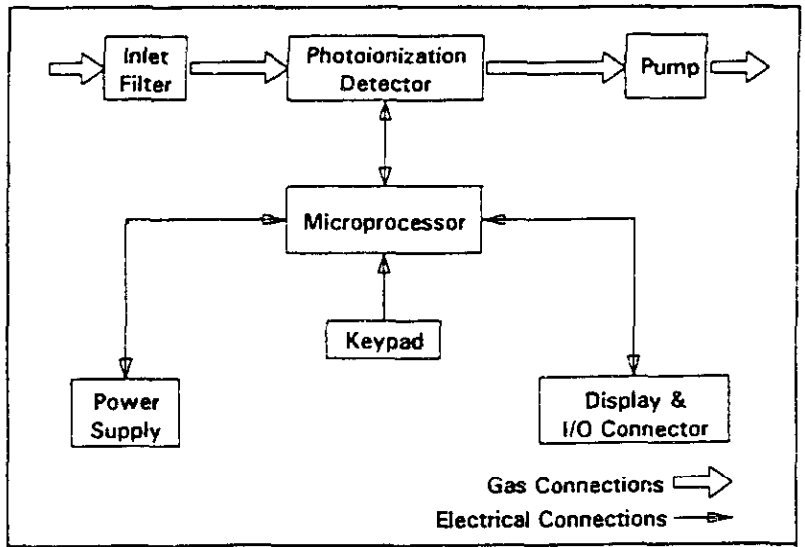


Figure 7 Block Diagram

A pump continuously pulls the air under test through MicroTIP's PID. The PID converts the concentration of ionizable chemicals in a sample into an electrical signal. The microprocessor subtracts any background from the signal and divides this signal by a sensitivity previously obtained by calibrating with a standard gas of known concentration. This concentration appears on MicroTIP's display and, depending on the values entered through MicroTIP's keypad, an alarm message may be displayed or an audio signal may be heard.

MicroTIP can detect thousands of different types of airborne

gases and vapors and its response depends on the type as well as the concentration. MicroTIP does not distinguish one type of chemical from another, but displays a number indicating the total concentration of all photoionizable compounds in the sample.

A standard of isobutylene at a known concentration may be used for setting the sensitivity. If MicroTIP is calibrated with isobutylene, it displays concentrations in units equivalent to ppm of isobutylene. If isobutylene were the only photoionizable chemical in the sample, then MicroTIP would display its concentration directly.

MicroTIP responds more or less readily to other chemicals than it does to isobutylene. Because it has a medium sensitivity to isobutylene, this gas has been chosen as a reliable means of reporting an average concentration of total photoionizables present.

For special applications, gases other than isobutylene can be used to calibrate MicroTIP.

6.2 PHOTOIONIZATION DETECTOR

MicroTIP's PID is shown in Figure 8. The PID measures the concentration of ionizable chemicals in the gas stream from the sample inlet and produces an electrical signal for the microprocessor.

A UV lamp generates photons which ionize specific molecules in the gas stream. The permanent air gases (argon, carbon dioxide, nitrogen, oxygen, water vapor etc.) require a relatively high energy for ionization, and are not ionized by the UV photons. Many of the chemicals considered pollutants, including most hydrocarbons, are ionized.

The gas stream is directed into the PID through a small port at the center of the lamp window and through a series of larger ports around the perimeter of the lamp window. This bypass arrangement permits a high sample flowrate and short response time, while minimizing contamination of the lamp window

The ionized molecules in the detector cell are subjected to a continuous electric field between the repeller electrode and

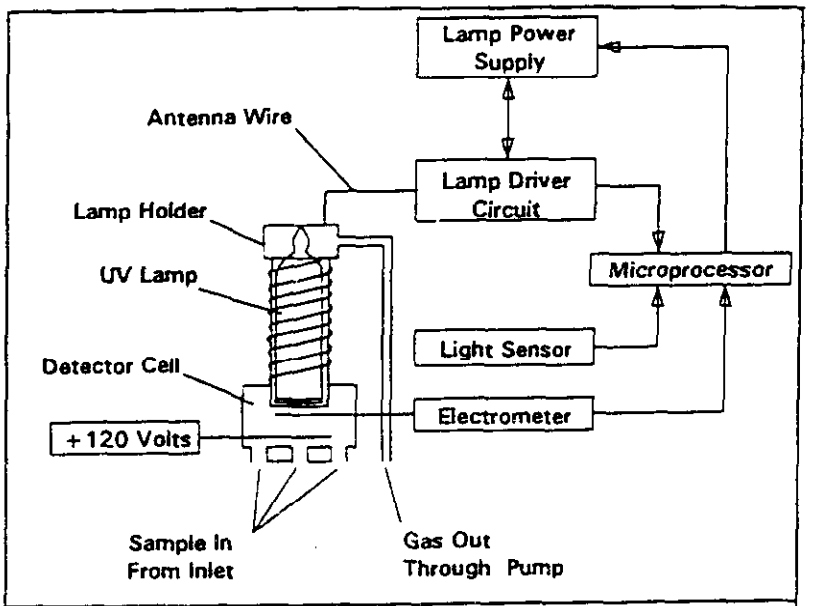


Figure 8 Photoionization Detector

the collector electrode. The ions move in the electric field, generating a current which is proportional to the concentration of the ionized molecules in the detector cell. An electrometer circuit converts the current to a voltage which is then fed to the microprocessor.

The detector lamp is operated by an HF lamp driver circuit which delivers high frequency energy to the lamp through an antenna wrapped around the lamp holder. The lamp driver power supply is controlled by the microprocessor based on a feedback signal from a light sensor on the HF driver circuit board.

6.3 CALIBRATION AND RECORDING

Periodic calibration is required to compensate for PID output changes due to inlet filter restriction, lamp window cleanliness, sample pump wear and other factors.

During calibration, MicroTIP's PID is first exposed to zero air. A small signal is generated. This zero signal is stored by the microprocessor.

In High Sensitivity operation, the microprocessor subtracts

the zero signal from the PID signal, and multiplies the difference by 1000. This number is then displayed.

When one of the 10 Cal Memories is selected, MicroTIP's PID is next exposed to span gas. This span signal is stored. The microprocessor subtracts the zero signal from the span signal and divides the difference by the user-entered span gas concentration. The resulting sensitivity is stored in the selected Cal Memory with the zero signal. In operation, the microprocessor first subtracts the zero signal from the PID signal, then divides the difference by the sensitivity. This number is then multiplied by the response factor and displayed.

The microprocessor accumulates all readings over a 15 second interval and determines the minimum, average and maximum readings. It stores these numbers along with the highest priority instrument status and the most recent time, date and Event number which occurred during the 15 second interval. MicroTIP automatically records these results for 12 hours of operation when the datalogger is turned on.

These recorded data can now be played back on MicroTIP's display. The display is identical to the numeric or bar graph display, but the instrument status is "Play" indicating that recorded data not real-time data, are being displayed. During playback MicroTIP continues to analyze and record new data.

Recorded data can also be printed as either a table or a graph. Data may be automatically averaged to fit on one 8½" x 11" page, or all recorded data may be printed or graphed. The averaging interval and number of readings averaged are shown at the top of the page.

For each averaging interval, MicroTIP prints the minimum of all the minima, the average of all the averages and the maximum of all the maxima.

Chapter 7 Specifications

Size:	43 cm (16.9") long, 9.5 cm (3.75") wide, 14.6 cm (5.75") high
Weight:	2.5 kg (5.5 lbs)
Detector:	Photoionization, bypass-type, with standard 10.6 eV HF-excited electrodeless discharge tube
Keypad:	16-key silicone with tactile feedback
Display:	2-line, 16-character dot-matrix, liquid crystal with adjustable backlighting, for alphanumeric and bar graph readouts
Datalogging memory:	25k
Chart recorder output:	0 to 1 volt full scale
Serial output:	RS-232, 300-19200 baud with odd, even or no parity, for tabular and graphic printouts
Audio output:	Continuous concentration-modulated tone or tone on alarm only
Inlet connection:	1/8" (3.2 mm) stainless steel compression fitting
Outlet connection:	1/8" (3.2 mm) stainless steel barb fitting
Battery type:	Sealed lead-acid, field-replaceable pack
Charge/discharge time:	8 hr/7 hr
Battery charger:	Automatically charges and maintains full charge in battery pack
Materials in sample stream:	Stainless steel, Teflon®, Viton®
Inlet filter:	Replaceable stainless steel, 2 um
Inlet flowrate:	Exceeds 500 mL/min

Operating temperature range:	0 to 40°C (32 to 105 °F)
Operating humidity range:	0 to 100% Relative Humidity (non-condensing)
Operating concentration range:	0.1 to 2000 ppm isobutylene equivalent
Accuracy:	Isobutylene: (after calibration with zero air and 100 ppm isobutylene span gas): within +/-2 ppm or +/-10% for 0 to 100 ppm; within +/-10% for 100 to 1000 ppm; within +/-15% for 1000 to 2000 ppm Acetone: (after calibration with zero air and 100 ppm acetone span gas): within +/-4 ppm or +/-20% for 0 to 100 ppm; within +/-30% for 100 to 1000 ppm; within +/-30% for 1000 to 2000 ppm Benzene: (after calibration with zero air and 100 ppm benzene span gas): within +/-4 ppm or +/-10% for 0 to 100 ppm; within +/-20% for 100 to 1000 ppm; within +/-25% for 1000 to 2000 ppm Toluene: (after calibration with zero air and 100 ppm toluene span gas): within +/-2 ppm or +/-20% for 0 to 100 ppm; within +/-30% for 100 to 1000 ppm; within +/-30% for 1000 to 2000 ppm Trichloroethylene: (after calibration with zero air and 100 ppm trichloroethylene span gas): within +/-5 ppm or +/-10% for 0 to 100 ppm; within +/-25% for 100 to 1000 ppm; within +/-45% for 1000 to 2000 ppm
Precision:	+/-1% (100 ppm isobutylene)
Response time:	Less than 3 seconds
Detection limit:	0.1 ppm isobutylene

Chapter 8 Warranty

MicroTIP is warranted for one year against defects in materials and workmanship.

Photovac Incorporated warrants that its manufactured products (except Detector UV Lamps which carry specific warranties) will be free from defects in materials and workmanship for a period of one (1) year from the date of receipt by the Customer. This Warranty applies to proper use of the equipment by the customer and may be voided if, in the opinion of Photovac Incorporated, the product has been abused or treated in a negligent manner so as to cause damage or failure. Negligent use includes, but is not limited to, exposure of the internal parts of the equipment to water. Damage caused thereby is expressly excluded from this Warranty.

When Photovac is made aware of a problem in MicroTIP which would be eligible for remedy under Warranty, it will issue a Return Authorization Number to the Customer. No return will be accepted unless such authorization has been obtained.

If upon receipt of the equipment Photovac determines that repairs should be done und .r Warranty, Photovac's sole liability shall be for labor and materials necessary to put the equipment into proper order and return this to the Customer as promptly as possible. Photovac is in no way responsible for any inconvenience or loss, consequential or incidental, caused to the Customer as a result of the equipment being out of commission.

The Customer is responsible for shipping and insurance to the designated Photovac Service/Repair facility.

In USA:	In Canada:
Photovac International Incorporated	Photovac Incorporated
25-B Jefryn Boulevard West	105 Doncaster Avenue
Deer Park, New York	Thornhill, Ontario
11729	L3T 1L6
(516)254-4283	(416)881-8225

Outside USA and Canada: Contact the Photovac representative in your area.

Appendices

APPENDIX A - INSTALLING AC PLUG

The MicroTIP battery charger is supplied with a grounded North American plug for 100-130 VAC operation. For use in other countries it must be replaced with a suitable plug.

1. Cut the power cord near North American style plug.

2. Separate and strip the two wires.

3. Connect one wire to the neutral terminal of the new plug. Connect the other wire to the live terminal of the new plug and assemble the new plug.

4. Set the voltage selection switch, on the bottom of the battery charger, to the correct voltage.

Warning: Incorrect AC connection can be lethal. Photovac cannot be responsible for errors in connection.

APPENDIX B - SPAN GAS SUPPLIER

The MicroTIP Calibration Kit includes the following items:

1. Gas pressure regulator with contents gauge, to fit a 6D size cylinder of span gas

2. Gas sampling bag

3. Adapter tubing with fittings for regulator and MicroTIP inlet (attached to gas bag).

The kit does not include a tank of span gas. The recommended span gas, isobutylene at 100ppm +/-5% in air, may be obtained from Alphagaz:

Alphagaz Specialty Gases Division,
Liquid Air Corporation,
P. O. Box 149, Woods Road
Cambridge, MD 21613, USA

Telephone: (301)228-6400

Outside the US contact the Alphagaz office above for the nearest location.

Index

Symbols

9.5 eV UV Lamp 27
10.6 eV UV Lamp 27
11.7 eV UV Lamp 29
  Operation 30
  Regenerate 30

A

AC Plug
  Replacing 49
Accessories 22
  Standard 1
Accuracy
  10.6 eV Response 21
  9.5 eV UV Lamp 28
  Specifications 47
Acetone
  10.6 eV Response 21
  9.5 eV Response 28
  Accuracy 47
Alarm Status 10, 38
ALARM Key 10
  Tutorial 4
Alarm Level 10
Aluminum Oxide
  Specifications 30
Analog Output 25
  Set Range 9
Arrow Keys 9
AUDIO Key 9, 10
  Tutorial 4
Audio Options
  Playback 10
Audio Output 46
  Set Range 9
Averaging Interval 14

B

Bar Graph Display 5
  Set Range 9
BATT Key 6
  Tutorial 4
Battery
  Charging 1, 32
  Pack 2
  Type 46
Battery Charger
  MP-1000 1, 32
  Replacing AC plug 49
  Specifications 46
  Voltage Selection
    Switch 49
Battery Pack
  Replacing 1
Baud Rate
  Setting 13, 23
Benzene
  10.6 eV Response 21
  9.5 eV Response 28
  Accuracy 47
Butyl Acetate
  10.6 eV Response 21

C

Cal
  Status 38
CAL Key 11, 27
  Tutorial 5
Cal Memory
  Programming 13
Calendar 4
Calibration 11
  High Sensitivity 19
  Technical Description 44
Calibration Kit 49
  Operation 11
Carbon Tetrachloride
  11.7 eV Response 29
Charge/Discharge Time 46
Charging the Battery 32
Chart Recorder
  Operation 25
Chart Recorder Output 46
CLEAR Key 7

F-56

Tutorial 5
Clock 4
Communications
Computer 24
Computer
Communications 24, 40
Operation 23
Serial Port 23
Concentration Range 47
Contamination
Lamp Window 29
Control Housing 35
Crosstalk 23
Operation With
MicroTIP 24
Set Up 24
Cyclohexane
10.6 eV Response 21
Cyclohexanone
10.6 eV Response 21

D

Data Bits 13, 23
Datalogger
Deleting 7
On/Off 7
Printing 13
Searching 11
Datalogger Memory 3, 46
Date 4
Setting 8
DB25 Connector 40
Detected Concentration 3
Detection Limit 20, 47
Detector
Contamination 39
Fault 39
Short Circuit 39
Specifications 46
Detector Cell
Removal 33
Detector Field Voltage
Fault 39
Detector Housing 2
Display 2
Options 5
Playback 10
Specifications 46
DISPLAY Key 5
Tutorial 4
Display Update 3
Displayed Reading 3
DOS Copy Command 25
Dust Cap 2
Dust Filter 37

E

Electronics Fault 39
ENTER Key
Tutorial 5
Epson FX-80
Printer 13, 22
Establishing Computer Communications 40
Ethyl Acrylate
10.6 eV Response 21
Event Counter
Resetting 7
EVENT Key 7
Tutorial 4
Event Number 3
Event Options 7
Events 7
Deleting 7
EXIT Key 4, 8
Tutorial 5

F

Fault Displays 37
Fault Status 38
Filter 37
Housing 2
Spring 34
Fit on One Page

Option 14, 17
Fitting
Sample Outlet 25

G

Gas Bag 12
Graph
Options 17
Range 16
GRAPH Key 16
Tutorial 5
Graph Output 18
Setting 9
Graphing
Duration 19
Stopping 19

H

Handle Flare 2
Headset
Connecting 9
Connection 26
Operation 9, 20
Volume 9
Volume Adjustment 26
HF Lamp Driver Circuit 44
High Sensitivity
Calibration 19
Selecting 9
Operation 19, 29, 30
Technical Description 44
HiSens Status 38
Humidity Range 47

I

I/O Connector 2, 14
IBM-AT 40
IBM-XT 40
ID Number
Graph 17
Print 14
Infrequent Operation 32
Inlet Connection 46
Inlet Filter
Replacement 34
Replacing 34
Specifications 46
Inlet Flowrate 46
Installation
AC Plug 49
Instrument Status 37
Code 19
Internal Fault 39
Isobutylene
Accuracy 47
Span Gas 11, 43

K

Key
ALARM 10
AUDIO 9
BATT 6
CAL 11
CLEAR 7
DISPLAY 5
EVENT 7
EXIT 8
GRAPH 16
LIGHT 5
MAX 6
PLAY 10
PRINT 13
SETUP 8
TUTOR 4
Key Functions 5
Key Options 5
Keypad 2, 4, 16, 19
Specifications 46

L

Lamp Driver Circuit 44
Lamp Holder 33
Lamp Spring 33
Replacing 34
Lamp Window
Cleaning 32
Contamination 29
Leak Detector 29
LIGHT Key 5
Tutorial 4

Liquid
If MicroTIP Draws In 37
LoBat
Status 32, 38
Lotus 1-2-3
Operation with
MicroTIP 25

M

Materials in Sample
Stream 46
MAX Key 6
Tutorial 4
Max Register 6
Memory, Datalogger 3
Methyl Ethyl Ketone
10.6 eV Response 21
Methyl Isobutyl Ketone
10.6 eV Response 21
9.5 eV Response 28
Methyl Methacrylate
10.6 eV Response 21
MicroTIP
Specifications 46
MicroTIP Calibration Kit 49
MicroTIP I/O Connector 14
Pinouts 23
MP-1000 Battery
Charger 1, 32

N

n-Heptane
10.6 eV Response 21
n-Octane
10.6 eV Response 21
Null Modem 40
Numerical
Display 5

O

Operating Concentration
Range 47
Operating Humidity
Range 47
Operating Temperature
Range 47
Operation
11.7 eV UV Lamp 30
Calibration Kit 11
Computer 23
Crosstalk 24
Headset 9, 20
High Sensitivity 19, 29, 30
Infrequent 32
Lotus 1-2-3 25
Overview 3
Printer 22
Options
EVENT Key 7
GRAPH Key 17
PLAY Key 10
PRINT Key 14
SETUP Key 8
Outlet Connection 46
Output all Data
Option 16, 19
Over Status 38

P

Parity, Setting 13, 23
Perchloroethylene
10.6 eV Response 21
9.5 eV Response 28
Photoionization Detector 42, 43
Photovac Service 39, 48
PID 42
PLAY Key 10
Options 10
Tutorial 5
Play Status 11, 38
Playback
Audio Options 10
Display 10
Power Supply Board 35
Power Switch 2
Precision 47
PRINT Key 13, 23, 25
Tutorial 4

PRINT Options 14
Printed Output 15
Printer
  Operation 22
Printer Cable 22
  Compatibility 40
  Modification 22
Printer Compatiblity 22
Printing
  Duration 16
  Stopping 16
Pump Connector 35

R

Ready
  Status 38
Recorded Data
  Printing 13
Recording
  Technical Description 44
Regenerate
  11.7 eV Lamp 30
Regulator
  Span Gas 49
Release Button 2
Repair Facility 48
Replacement UV Lamps 26
Replacing
  AC Plug 49
  Battery Pack 1
  Inlet Filter 34
Response
  Changing 27
  Non-specific 9
Response Factor 12, 20, 27
  9.5 eV Lamp 28
Response Time 20, 47
Return Authorization Number 48
Routine Maintenance 32
RS232 Serial Interface 13, 22

S

Sample Bag
  Operation With 25
Sample Inlet 2
Sample Line 2
  3 Meter 26
  Contamination 39
Sample Outlet 2
Sample Outlet Fitting 25
Sample Pump
  Replacing 35
Sensitivity
  Setting 11, 43
Serial Output 46
Serial Port
  Computer 23, 40
Service/Repair Facility 48
Setting Sensitivity 43
SETUP Key 8, 25
  Tutorial 4
SETUP Options 8
Shoulder Strap 26
  Connectors 2
Signal
  Fault 39
Size 46
Span Gas
  Cylinder Specifications 49
  Regulator 11, 49
  Supplier 49
Special Applications 27, 43
Specifications 46
  Aluminum Oxide 30
Speed Search 11
Standard Accessories 1
Standard Lamp 27
Stop Bit 13, 23
Styrene
  10.6 eV Response 21

T

Technical Description 42
Temperature Range 47

Time
  Setting 8
Toluene
  10.6 eV Response 21
  9.5 eV Response 28
  Accuracy 47
Trichloroethylene
  10.6 eV Response 21
  9.5 eV Response 28
  Accuracy 47
Troubleshooting 37
TUTOR Key 4, 39
Tutorial 4

U

UV Lamp
  10.6 eV 27
  11.7 eV 29
  9.5 eV 27
  Replacing 34
  Technical Description 43
UV Lamp Window
  Cleaning 32

V

Voltage Selection Switch
  Battery Charger 49

W

Warranty 48
Weight 46
Wire Mesh 33, 37

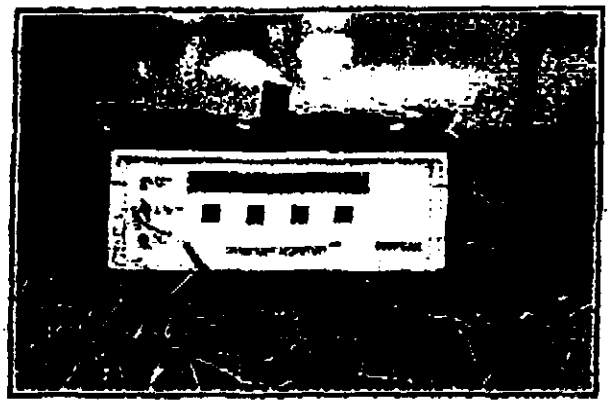
Z

Zero Air
  Source 11

REOTEMP INSTRUMENT CORPORATION

PLUS

REOTEMP COMPOST MONITOR QUICK REFERENCE OPERATION



INTRODUCTION

The Compost Monitor, and it's associated software, have been designed to make operation as easy as possible.

Before the Compost Monitor can be used in the field, it needs to be given information about the windrows that you are going to measure. This information (name, height, length, width, number of samples to be taken and depth of samples) is 'uploaded' to the unit from the "Windrows for Windows" software.

To begin, start the "Windrows for Windows" software by clicking on it's icon .

CREATING NEW WINDROW PROTOCOLS

From the top menu bar, click on [Windrow] and then on [New]. The software will now guide you step by step through creating a windrow. You will first be asked to give the windrow a name (this can be an alpha/numeric up to 16 characters). Fill in the data for length, width and height and also the number of depths at which you want to sample. Click on [Next].

Enter the number of places (segments) along the windrow that you wish to take readings. Click on [Next]. At this point you can either [Finish] or click on [Next] to move on and enter information about the make-up of the windrow (i.e. it's ingredients). The ingredient information is for reporting only.

UPLOADING WINDROW PROTOCOLS

Once a windrow protocol has been created on the computer, it has to be 'uploaded' to the Compost Monitor ready to make measurements.

Click on the [Compost Monitor] menu bar and then on [Upload Protocol]. Each of the current active protocols are listed in the left hand window. Simply move the protocols over to the right hand window by either clicking on the windrow name, dragging and dropping it into the right hand space, or clicking on the name and then clicking the [>] button.

The windrow names should be placed in the order in which the measurements will be taken in the field. The order can be sorted if desired.

Click on [Next]. You will now be instructed to connect the serial cable to the Compost Monitor. Press the far right hand button on the Compost Monitor until you reach ■ PC LINK, then press ■ RUN.

The windrow protocols will be automatically loaded into the Compost Monitor. Once completed, the Monitor is ready to use.

CALIBRATING THE COMPOST MONITOR

If the Monitor has not been in use for 15 minutes, it will automatically shut down. To turn on the Monitor, press any ■ key on the front panel. Adjust the display contrast if necessary.

Press the far right hand key until you reach ■ AUTOCAL. Now press ■ RUN. The instrument will sample room air and set a reading of 20.9% O2 automatically. Be careful not to breathe near the *Sample Inlet* during this procedure.

CONNECT THE SAMPLE PROBE

Make sure the gas sampling holes in the probe are free of any blockages.

Connect the sample tube to the *Sample Inlet* on the Monitor front panel, and insert the phone jack into the *Temperature Sensor* socket.

TAKING MEASUREMENTS

Press the far right-hand ■ key until you reach ■ DATALOGGING.

The display on the Monitor will show the following:

Wind:Example		Seg:01	Depth: 3'
SAMPLE 000	LAST	NEXT	DATALOGGING

■ ■ ■ ■

The windrow name, should be the first location requested during 'upload', and the Sample # should be 000. (Note: the first sample is always 000). The Segment will be 01 and the Depth either 1', 2' or 3'.

Simply insert the probe into the windrow at the appropriate depth (note the 1' incremental markings on the probe shaft) then press ■ SAMPLE.

The pump will turn on and a countdown of seconds will displayed together with Temperature and Oxygen. Once completed, the data is stored and the unit will automatically flush with air.

The display now moves on to the next data location as called for in the protocol. Simply move to the next location or depth and press ■ SAMPLE.

If ever you take a bad reading, simply press the ■ LAST key to return to the previous data. An * will be displayed next to the Sample # to indicate that data has been stored in this location. Pressing ■ SAMPLE will allow you to overwrite the data.

The Compost Monitor was designed to guide you step by step through the day's measurements. Simply take the readings and move to the next location. However, if you need to repeat a measurement, or skip over measurements, the ■ LAST and ■ NEXT keys give you ability to do so. This may be necessary if you are delayed in making a measurement and the Monitor has turned itself off. Simply turn on the Monitor, go to DATALOGGING and advance the ■ NEXT key until you reach the empty data location (i.e. no *).

DOWNLOADING STORED DATA

Click on the [Compost Monitor] menu bar and then on [Download Protocol]. You will now be instructed to connect the serial cable to the Compost Monitor. Click on [Next]. Press the far right hand button on the Compost Monitor until you reach ■ PC LINK, then press ■ RUN.

The data will be automatically downloaded into the Computer then stored in the appropriate windrow files.

VIEWING STORED WINDROW DATA

To review data on the computer, click on the desired windrow name listed on the left of the screen.

Once the data has been loaded, the four 'Tabs' (Overview, Graphs, Measurements & Composition) can be used to quickly look at a summary, or overview, of the data to date, together with graphs, numerical listing of the measurements, and a pie chart of the windrow composition.

To change the graph, simply click on the [Graphs] option on the upper menu bar. The pull-down list of graph options is self explanatory.

PRINTING REPORTS

To print a report, highlight the desired windrow name listed on the left of the screen. Click on the [Windrow] menu bar and then on [Report].

A dialogue box will appear that allows you to configure the report, design the graph configuration and change the printer set-up. Once you click [OK] the report will print.

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ISO 9002

11568 Sorrento Valley Rd. • Suite 10 • San Diego, CA 92121 U.S.A. • Telephone 619/481-7737 • FAX 619/481-7150
http://www.reotemp.com/reotemp • Email: reotemp@reotemp.com

Compost Monitor Price List 1996

Cat No.	Instrument Description	Price $
CM200	Compost Monitor - *Plus* Portable digital instrument for the measurement of compost Temperature and Oxygen complete with: padded carrying bag, 5 foot stainless steel probe with depth indication markings, multi-function liquid crystal display, non-volatile memory for storing up to 256 data sets, internal sampling pump, automatic operation for continuous or pulse pump sampling, battery charger, RS-232 output, Windows 95/NT ® operating software for data down-loading and analysis and instruction manual. *Note: No calibration gas required.*	2,600.00
CM300	Compost Monitor - *Pro* Portable digital instrument for the measurement of compost Temperature, Oxygen and Carbon Dioxide complete with: padded carrying bag, 5 foot stainless steel probe with depth indication markings, multi-function liquid crystal display, non-volatile memory for storing up to 256 data sets, internal sampling pump, automatic operation for continuous or pulse pump sampling, battery charger, RS-232 output, Windows 95/NT ® operating software for data down-loading and analysis and instruction manual. *Note: Calibration gas (25% CO2 in N2) required.*	3,950.00

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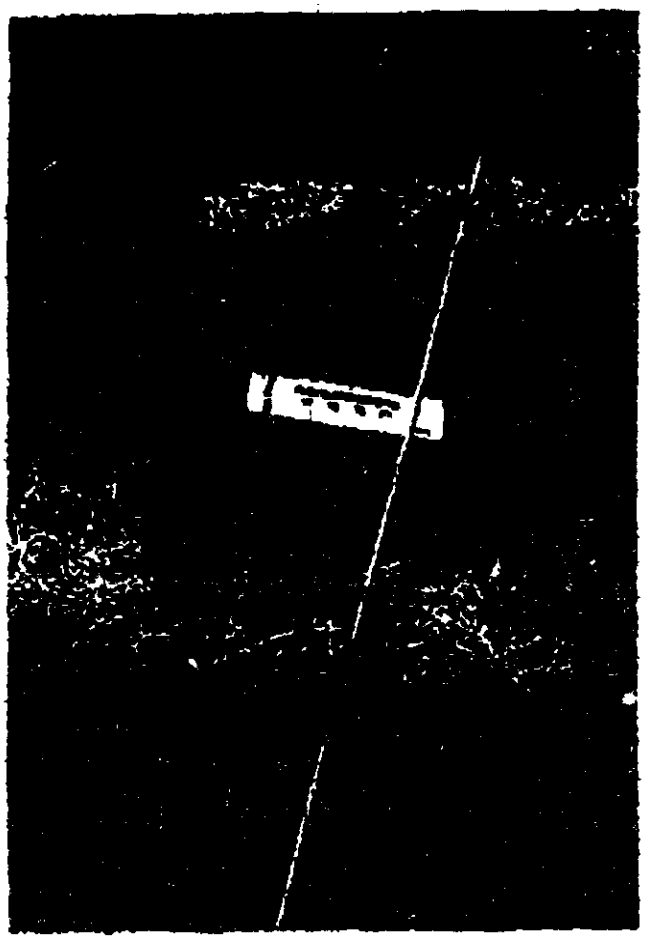
COMPOST MONITORS



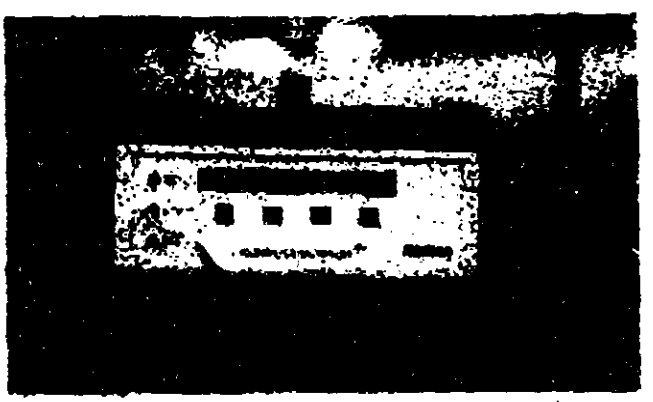
SPECIFICATIONS:

Plus Model:

Size	inches	11.5 W x 4.5 H x 7.5 D
	cms	29.2 W x 11.4 H x 19.1 D
Weight		4 lbs (8.8 kgs)
Front panel		Wipe-clean overlay
Batteries		2 x 1.2V 'D' size rechargeable
Battery Life		6 hours
Charger Unit		6 volts
Data Storage		256 sets of measurements
Data Back-up		2nd Battery for data security
Clock		Date and time recorded
Temperature Sensor		Thermistor
	Range	0 - 250°F (121°C)
Oxygen Analyzer		Galvanic cell
	Range	0 - 21% O_2
Pump		Flow 0 - 3 Liters/Minute
Measurement Time		45 seconds for each reading
Outputs		Analog 0-2.5 volts & RS232
Carrying Bag		Canvas padded and lined
Sample Probe		
	Length	4' 8" marked in 1' increments
	Weight	1.5 lbs (3.3kgs)
	Cleaning	Shaft disassembles

Pro Model:
Same as above with the following additions:

Weight		4.5 lbs (9.9 kgs)
CO_2 Analyzer		Single beam infra red
	Range	0 - 25% CO_2
	Response	6 seconds full
	Warm up	10 minutes



Minimum computer specifications needed to operate the "Windrows for Windows™" software:

PC compatible with 12MB RAM running Windows 95/NT™
Free space required - 10MB

Precision and Quality for Half a Century

pH METER INFORMATION TO BE PROVIDED

APPENDIX F

LABORATORY STANDARD OPERATING PROCEDURES (SOPs FOR SAMPLE PREPARATION AND ANALYSIS)

APPENDIX F
LABORATORY STANDARD OPERATING PROCEDURES

TABLE OF CONTENTS

SECTION		PAGE
1	LABORATORY EQUIPMENT CALIBRATION	F-1
2	STANDARD OPERATING PROCEDURES FOR ANALYSIS	F-30
	MS300 (VOLATILE ORGANICS BY METHOD 8240)	F-30
	(PAGES 50-62 DELETED)	
	GC200 (EXPLOSIVES BY METHOD 8330)	F-63
	MT913B (METALS BY METHOD 6010)	F-86
	MT600B (SW846 DIGESTION OF AQUEOUS AND SOIL MATRICES FOR ICP ANALYSIS) (ATTACHMENT TO MT913B)	
	MT310B (MERCURY BY METHOD 7471/7470)	F-99
	HW700/HW710 (TCLP NON-VOLATILE EXTRACTION BY METHOD 1311)	F-108
	IN045S, IN045W (TOTAL ORGANIC CARBON BY METHOD 9060 (soil) AND METHOD 415.1 (water))	F-114
	IN170 (TOTAL KJELDAHL NITROGEN BY METHOD 351.3)	F-129
	(PAGES 135-228 DELETED)	
	IN275 (TOTAL SUSPENDED SOLIDS BY METHOD 160.2)	F-229
	IN090D, IN090C (CHEMICAL OXYGEN DEMAND BY METHOD 410.2)	F-234
	IN025 (BIOCHEMICAL OXYGEN DEMAND BY METHOD 405.1)	F-244
	IN200/IN205 (OIL AND GREASE BY METHOD 413.1)	F-248

TABLE OF CONTENTS

SECTION		PAGE
	IN030 IN055, IN135, IN185, IN195, IN255, IN295 (NITRATE BY METHOD 300.0)	F-257
	IN020 (AMMONIA-N BY METHOD 350.2)	F-265
3	PREVENTIVE MAINTENANCE	F-270

RED STAMP INDICATES ORIGINAL

AMERICAN ANALYTICAL & TECHNICAL SERVICE, INC. / SOUTHWEST LABORATORY OF OKLAHOMA, INC.

STANDARD OPERATING PROCEDURE FOR THE ANALYSIS OF VOLATILE ORGANICS

SW846/8240

MS300

REV 5.4— 11/26/96

Document Control #AATSWL-MS300- 031

CONTROLLED DOCUMENT: DO *NOT* COPY!

APPROVALS BY:

Name: Gina Jackson _____ *Title: Volatile Section Supervisor* _____

Signature: Gina B Jackson

Name: Chuck Hoover _____ *Title: QA/QC Officer* _____

Signature: Chuck Hoover

Contents

GC/MS ANALYSIS OF PURGEABLE ORGANICS 1
I. Scope/Summary of Method 1
II. Sample Preservation/Holding Times 1
III. Apparatus and Materials 1
IV. GC/MS System 2
V. Reagents and Standards 2
VI. GC/MS Operating Conditions 3
VII. Calibration 3
VIII. QC Samples 5
IX. Sample Analysis 6
X. Interferences 8
XI. Quality Assurance/Quality Control (QA/QC) 9
XII. Analysis/Report Criteria/Validation 9
XIII. Health & Safety 10
XIV. Data Archival 10
XV. References 10
TABLE 1. 11
TABLE 2. 11
TABLE 3. 12
TABLE 4. 12
TABLE 5. 12
TABLE 6. 13
TABLE 7. 14
TABLE 8. 14
TABLE 9. 14
TABLE 10. 15
TABLE 11. 16
TABLE 12 17
TABLE 13. 18

GC/MS ANALYSIS OF PURGEABLE ORGANICS

I. SCOPE/SUMMARY OF METHOD

A. An inert gas, helium, is bubbled through a 5 mL sample contained in a specifically designed purging chamber at ambient temperature. The purgeables are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column where the purgeables are trapped. After purging is completed, the sorbent column is heated and backflushed with the helium to desorb the purgeables onto a gas chromatographic column. The gas chromatograph is temperature programmed to separate the purgeables which are then detected with a mass spectrometer.

B. An aliquot of the sample is diluted with reagent water when dilution is necessary. A 5 mL aliquot of the dilution is taken for purging.

C. If the amount of TCL compound detected is outside the linear range of the instrument, then a smaller sample size is analyzed.

II. SAMPLE PRESERVATION/HOLDING TIMES

A. Water and soil samples are analyzed within 14 days of sampling.

B. Waters should be preserved with HCL (pH <2) and received and stored cool (4°C) until analysis. Sodium Thiosulfate (4 drops of 10%) is also used when residual chlorine is present in the water.

C. Soils should be received and stored cool (4°C) until analysis.

III. APPARATUS AND MATERIALS

A. *Micro syringes* — 10 µL and larger, 0.006 inch ID needle.

B. *Syringe valve* — two-way, with Luer ends (three each), if applicable to the purging device.

C. *Syringe* — 5 mL, gas tight.

D. *Balance, Analytical* — capable of accurately weighing 0.0001 g. and a top-loading balance capable of weighing 0.1 g.

E. *Glassware*

1. Bottle — 40 mL, screw cap, with Teflon cap liner.

F. *Purge and trap device* — The purge and trap device consists of three separate pieces of equipment; the sample purger, trap and the desorber. (Tekmar 2000 with 2016 and 2032 autosamplers). A heating blanket is added to the Tekmar autosamplers when low level soils are analyzed.

IV. GC/MS SYSTEM

A. *Gas chromatograph* — An analytical system complete with a temperature programmable gas chromatograph suitable for on-column injection and all required accessories including syringes, analytical column, and gases.

B. *Column* — DB624, 75m, 0.53mm ID, 3.0 um (J&W Scientific Cat. No. 125-1334) or equivalent.

C. *Jet Separator* — Glass (SIS Cat. No. 113505).

D. *Mass spectrometer* — Capable of scanning from 35 to 300 amu every three seconds, utilizing seventy volts (nominal) electron energy in the electron impact ionization mode and producing a mass spectrum which meets all the criteria when 50 ng of 4-Bromofluorobenzene (BFB) is injected through the gas chromatograph inlet (see Table 4).

E. Calibration requirements can be found in Tables 8 and 9.

V. REAGENTS AND STANDARDS

A. *Reagent water* — Produced by filtering tap water through an organic filtration system containing activated charcoal prior to use.

1. *Methanol* — Pesticide quality or equivalent.

2. *Stock standard solutions* — Stock standard solutions are purchased and are traceable EPA supplied standards. These solutions are stored in Teflon sealed screwcap bottles at 10°C to 20°C.

B. *Secondary dilution standards* — Using stock standard solutions, prepare secondary dilution standards in methanol that contain the compounds of interest, either singly or mixed together. (See GC/MS Calibration in Exhibit E). Secondary dilution standards should be stored with minimal headspace and should be checked frequently for signs of degradation or evaporation especially just prior to preparing calibration standards from them.

C. *Surrogate Standard Spiking Solution.*

1. Prepare stock standard solutions for toluene-d_8, p-bromofluorobenzene, and 1,2-Dichloroethane-d_4 in methanol. Prepare a surrogate standard spiking solution from these stock standards at a concentration of 25 µg/mL in methanol.

D. *Purgeable Organic Matrix Standard Spiking Solution.*

1. Prepare a spiking solution in methanol that contains the following compounds at a concentration of 25 µg/mL: 1,1-Dichloroethene, trichloroethene, chlorobenzene, toluene, benzene.

2. Matrix spikes also serve as duplicates; therefore, add an aliquot of this solution to each of two portions from one sample chosen for spiking.

E. *Internal Standard Solution*

1. The three internal standards are Bromochloromethane, 1,4-Difluorobenzene and chlorobenzene-d_5. Prepare a spiking solution containing each of the internal standards. It is recommended that the secondary dilution standard be prepared at concentration of 25 µg/mL of each internal standard compound. The addition of 10 µL of this standard to 5.0 mL of sample or calibration standard would be equivalent of 50 µg/L.

VI. GC/MS OPERATING CONDITIONS

A. These performance test require the following instrumental parameters:

1. Electron Energy: 70 Volts (nominal)
2. Mass Range: 35-300
3. Scan Time: to give at least 5 scans per peak, not to exceed one second per scan

B. All samples and standard solutions must be allowed to warm to ambient temperature before analysis.

C. Recommended operating conditions for the gas chromatograph - Column Conditions: DB-624, 75m, 0.53mm ID, 3 um film thickness or equivalent at a flow rate of 30 mL/min. Column temperature is isothermal at 45°C for five minutes, then programmed at 5°C per minute to 105°C. further programmed at 10°C/min. to 180°C.

D. Adjust the purge gas (helium) flow rate to 35 ± 3 mL/min. Variations from this flow rate may be necessary to achieve better purging and collection efficiencies for some compounds, particularly chloromethane and bromoform.

VII. CALIBRATION

A. *Tune*

1. Before analysis of samples can begin, the instrument must meet all calibration criteria. The first step in the calibration of Volatiles GC/MS system is to demonstrate the ionization and fragmentation of standard mass spectral tuning compounds. This is accomplished, as well as sensitivity check, with the use of 4-Bromofluorobenzene. This standard is run daily to validate the GC/MS system and to begin the 12-hour clock. BFB is analyzed by direct injection of a 50 ng aliquot into the instrument.

2. When the analysis is complete, the tune is checked against standard criteria (See Table 3 for Tune Criteria). SW846 Method 8240 tune criteria for BFB is used for all volatile test codes, and before any other calibration standards or sample analysis is performed.

3. If the tune fails to meet the criteria, rerun BFB. If it fails again, the instrument must be retuned through "Auto Tune" or "Manual Tune". BFB should be run again. If it meets criteria, continue with the calibration sequence. If it fails again, contact your supervisor. Instrument maintenance such as source cleaning may be required.

B. *Initial Calibration*

1. Once BFB has met its tune criteria, calibration may begin. Initial calibration of the GC/M established and validated by the analysis of EPA traceable standards at five concentration levels which are 5, 20, 50, 100, and 200 ppb. These standards are made up in 5 mL of reagent water and purged on the Tekmar before analysis by GC/MS. The internal standard calibration procedure is used. In addition to surrogate recovery compounds, standards and sample extracts are spiked with internal standards that span the retention time range of the analytes of interest.

2. The Response Factors (RF) for the analytes in the standard are calculated and stored in a Target Method File. The concentration of the analytes during sample analysis is calculated using the RF of each analyte determined in each day's calibration. These equations are shown in Table 13.

3. The calibration standards are prepared by filling a syringe with 5 mL of reagent water and spiking with the standards as shown below.

Internal Std. (µL)	Surrogate (µL)	25 ppm Stock Std. (µL)	Int. Std. Final conc. (ng/mL)	Stock Std. Final conc. (ng/mL)
10	1	1	50	5
10	4	4	50	20
10	10	10	50	50
10	20	20	50	100
10	40	40	50	200

4. Attach the syringe to the syringe valve assembly on the Tekmar. Open the syringe valve and the sample into the purging chamber. Close the purging device valve. Low level soil calibra must be heated to 40°C before purging. Water, medium level soils, and high level soils pur ambient temperature. Purge the sample for 8.0 min. At the conclusion of the purge time, introduce the trapped materials to the GC column by the Desorb Mode where the trap is rapidly heated to 180°C for four minutes.

5. While the trap is being desorbed into the GC, you may empty the purging chamber. After desorbing the sample for four minutes, bake the trap for four minutes at 225°C. After bake, allow the trap to cool to less than 30°C making it ready to purge the next standard or sample.

6. The standard will automatically be quantitated into a report from which concentrations, areas, response factors, standard deviations, and percent differences are calculated.

7. SW846 Method 8240 criteria for Volatiles Calibration Check Compounds (CCC) is employed:

CCC
1,1-Dichloroethene
Chloroform
1,2-Dichloropropane
Toluene
Ethylbenzene
Vinyl Chloride

8. The %RSD is calculated automatically for all compounds. This %RSD must be within 30% for all CCC Compounds.

9. SW846 Method 8240 criteria for System Performance Check Compounds (SPCC) is em

for the compounds below.

SPCC
Chloromethane
1,1-Dichloroethane
Chlorobenzene
Bromoform
1,1,2,2-Tetrachloroethane

10. The average RF is calculated for all compounds and the RF for SPCC compounds must be at least 0.300, except for Bromoform, which has a minimum RF of 0.100. If the five-point does not meet criteria, check to see if a standard needs to be rerun. The standards could have been added at the wrong amount or the purge may have been bad. If reanalyzing a standard will not solve the problem, contact your supervisor. It could be related to purge chambers, multiplier, flows, etc.

11. If the five-point does meet criteria, and there is time remaining in the 12-hour time period, samples may be run. The 50 ppb Standard's response factors are used to calculate the concentrations in samples of TCL (Target Compound List) compounds.

C. *Continuing Calibration*

1. A continuing calibration of the GC/MS uses the same EPA traceable standards, but at only one level. This level is 50 ppb The analysis is performed in the same manner as stated for the initial calibration. The continuing calibration checks the same CCC and SPCC compounds as the initial calibration. The CCC compounds must have a %Difference (%D) of less than 20% and the SPCC must have an RF from that particular day's calibration of greater than 0.300, except for Bromoform, which must be greater than 0.100. The continuing calibration is performed after the BFB and must meet all criteria before any other analysis continues.

2. If the criteria is not met, the standard must be rerun. It is possible it was not prepared correctly, or that there was a bad purge. However, if the second standard still doesn't meet criteria, an initial calibration must be run or corrective action performed.

VIII. QC SAMPLES

A. *Method Blank*

1. The method blank is analyzed once the calibration has been met, whether it be an initial or continuing calibration. The method blank, called the "VBLK", consists of loading a syringe with 10 mL of reagent water spiked with 10 µL of Internal Standard and 10 µL of Surrogate which yields a concentration for both of 50 ppb. It is used to establish the analytical curve by taking into account any background responses during the calibration process. It is also used to check for carryover contamination after a standard run or after a contaminated sample run.

2. An acceptable blank must meet the internal standard criteria stated in the "Sample Analysis" section and the surrogate recoveries in Table 4. It must also contain less than or equal to five times the Practical Quantitation Limits (PQL) of Methylene Chloride, Acetone, Toluene, and 2-Butanone. For all other compounds, the blank must contain less than or equal to the PQL of any single analyte.

3. If a blank exceeds this criteria, corrective measures must be taken immediately. This action first consists of immediately reanalyzing the blank. If it still exceeds criteria, consult your supervisor immediately.

B. *Laboratory Control Spike (LCS and LCSD)*

1. The Laboratory Control Spikes (LCS and LCSD) are the last analysis of the calibration sequence. They are used as internal QA/QC checks. The LCS and LCSD are analyzed daily following the method blank to check the proficiency of the analysis in terms of working standards preparation, to monitor standard degradation, and to check traceability of the prepared standards from commercial reference materials to EPA reference materials. These results are also used to create our laboratory control charts.

2. The sample is prepared by loading a syringe with 5 mL of reagent water spiked with 10 µL of Internal Standard, 10 µL of Surrogate, and 10 µL of LCS Mix. This yields a final concentration for all three standards of 50 ppb.

3. The LCS and LCSD contain most of the compounds found in the daily calibration standards. Their limits may be found in Table 6. The LCS and LCSD must meet these limits, as well as the surrogate and internal standard criteria. If these criteria are not met, corrective action must be taken immediately. This includes rerunning the LCS and/or LCSD. If the compounds are still out of limits, a new standard should be prepared. LCS limits are advisory like Matrix Spike Compounds, but since they are internal QC limits set forth by our laboratory, every effort should be made to meet these limits.

IX. SAMPLE ANALYSIS

A. *Water Samples*

1. Note any headspace in the bottle before opening. Open the sample bottle which has been allowed to come to ambient temperature, and carefully fill the sample into the syringe barrel to just short of overflowing, and adjust the sample volume to 5.0 mL. This process of taking an aliquot destroys the validity of the sample for future analysis. Care must also be taken to prevent air from leaking into the syringe.

2. Add 10 µL of the Surrogates spiking solution and 10 µL of the internal standard spiking solution through the valve bore of the syringe. This yields a concentration of 50 µg/L of each standard.

3. Water samples are loaded in the following manner to ensure the proper sample is placed into its designated purge chamber on the Tekmar Autosampler:

   a. A test tube rack is filled with samples' 40 mL VOA vials. The syringes to be used are placed in front of the samples.

   b. All sample purge tubes are labeled with the SWLO ID number and autosampler position. Each analyst verifies that the syringe matches the ID on the purge tube as he/she loads each sample into the purge chambers.

   c. Follow the same purging method as stated during calibration.

4. For water samples requiring matrix spike and matrix spike duplicates, add 10 µL of the matrix spike solution to the 5 mL of the sample to be purged. Disregarding any dilutions, this is equivalent to a concentration of 50 µg/L of each matrix spike standard.

B. *Soil Sample Analysis — Low Level*

1. The low level method is based on purging a heated sediment/soil mixed with reagent containing the surrogate and internal standards.

2. The sample (for volatile organics) consists of the entire contents of the sample container.

Do not discard any supernatant liquids. Mix the contents of the sample with a narrow spatula. Weigh the sample into a disposable test tube. Use 5 grams of sample, unless a dilution is required in which 1 - 4 grams of sample is used. Use a top loading balance. Note and record the actual weight to the nearest 0.1 g.

3. Immediately after weighing the sample into the labeled test tube, load it onto the Tekmar ALS. Remove the plunger from a 5 mL Luerlock syringe and fill to overflowing with reagent water. Replace the plunger and compress the water to vent trapped air. Adjust the volume to 5.0 mL. Add 10 µL each of the surrogate spiking solution and the internal standard solution to the syringe. This addition to the soil is equivalent to 50 µg/Kg of each standard. Add this spiked reagent water to the test tube. Heat the sample to 40°C and begin purging. The samples follow the same purge method as stated in the calibration.

4. For soil samples requiring matrix spike and matrix spike duplicates, add 10 µL of the matrix spike solution to the 5 mL of the reagent water being added to the soil. Disregarding any dilutions, this is equivalent to 50 µg/Kg of each matrix spike standard.

C. *Soil Sample Analysis — Medium Level*

1. Medium level extraction/analysis is required if the low level of analysis at 1 g level indicates peak saturation or compounds are out of linear range. The medium level method is based on extracting the sediment/soil sample with methanol. An aliquot of the methanol extract is added to reagent water containing the internal standards. The surrogate is spiked directly into the extract, thus making the addition directly into the reagent water unnecessary. The extract is purged at ambient temperature.

2. The medium level extract is prepared by weighing 4 g of the sample into the scintillation vial. Use a top loading balance. Note and record the actual weight to the nearest 0.1 g. Add 9900 µL of methanol and 100 µL of the medium concentration surrogate standard. Cap and shake by hand for 2 minutes. The extract should be made up quickly in order to avoid loss of volatile organics.

3. The sample is loaded in the following manner. Fill a syringe just short of overflowing, and adjust the sample volume to 4.9 mL. Adjust the plunger to 5 mL to allow for the addition of the extract and standards. Add 10 µL of internal standard solution. Also add the volume of the methanol extract and the volume of methanol, if needed, to total 100 µL.

4. Load the sample into the purging device (Tekmar) and follow the same purging method as stated during the calibration.

5. For samples requiring matrix spike and matrix spike duplicates, prepare an extract in the same manner as stated above, but add only 9800 µL of methanol, 100 µL of medium concentration surrogate, and 100 µL of medium concentration matrix spike solution. Load and analyze them in the same manner as the samples.

6. The method blank must be analyzed in the same manner as with low level soils and waters, but it is prepared differently The method blank consists of a 4 g purified solid matrix spike with the surrogates made up to a total volume of 10 mL An acceptable blank must contain less than or equal to five times the PQL of Methylene Chloride, Acetone, and 2-Butanone, and less than or equal to the PQL of any other volatile target compound. It must meet criteria or corrective action must be taken immediately.

7. With all matrices, all dilutions should keep the response of the major constituents (previously saturated peaks) in the upper half of the linear range of the curve.

8. An estimated concentration for Non-target components tentatively identified shall be quantif by the internal standard method. For quantification, the nearest internal standard free o interferences shall be used.

9. The formula for calculating concentrations is found in Table 13. Total area counts from the total ion chromatograms are to be used for both the compound to be measured and the internal standard. A response factor (RF) of one (1) is to be assumed. The value from this quantitation shall be qualified as estimated. This estimated concentration should be calculated for all tentatively identified compounds as well as those identified as unknowns.

10. Xylenes (o,m, & p - isomers) are to be reported as total Xylenes. The concentration of all Xylene isomers must be added together to give the total.

11. All soil samples must have their percent moisture determined. This is performed by the Extractions Department and submitted to the Volatiles Data Clerk for reporting. The percent moistures may also be obtained from the LIMS.

X. INTERFERENCES

A. Interferences purged or coextracted from the samples will vary considerably from source to source, depending upon the particular sample or extract being tested. The analytical system, however, should be checked to ensure freedom from interferences, under the analysis conditions, by analyzing method blanks.

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

C. Cross contamination can occur whenever high-concentration and low-concentration samples are analyzed sequentially. Whenever an unusually concentrated sample is analyzed, it should be followed by the analysis of organic-free reagent water to check for cross contamination. The purge-and-trap system may require extensive bake-out and cleaning after a high-concentration sample.

D. The laboratory where volatile analysis is performed should be completely free of solvents.

E. Impurities in the purge and gas and from organic compounds outgassing 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 reagent blanks. The use of non-TFE plastic coating, non-TFE thread sealants, or flow controllers with rubber components in the purging device should be avoided.

XI. QUALITY ASSURANCE/QUALITY CONTROL (QA/QC)

A. The GC/MS system should be tuned to meet the BFB specifications in Section VI.A. and Table 3.

B. There should be an initial calibration of the GC/MS system as specified in Section VI.B. or a contin

calibration (Section VI.C.)

C. Before processing any samples, the analyst should demonstrate, through the analysis of a method blank, that interferences from the analytical system, glassware, and reagents are under control. Each time a set of samples is extracted, or there is a change in reagents, a method blank should be processed as a safeguard against chronic laboratory contamination. The blank samples should be carried through all stages of sample preparation and measurement.

D. A Laboratory Control Spike (LCS) and a Laboratory Control Spike Duplicate (LCSD) are analyzed for each batch of 20 samples. The percent recoveries of the LCS/LCSD are compared to historically generated control limits to determine whether the analytical system is in control (see table 6.)

E. A Matrix Spike (MS) and a Matrix Spike Duplicate (MSD) are analyzed as per client request to determine whether there are matrix effects on the recoveries of particular compounds. Percent Recoveries are compared to Method Limits and outliers are flagged to alert the data user of matrix effects (see table 5.)

F. Surrogate Recoveries from every sample are compared to method control limits (see table 4.) If any surrogates are out of control, the sample is reanalyzed. The reanalysis is then reported if surrogates are within limits or the reanalysis is used to establish a matrix effect on the surrogate recoveries.

XII. ANALYSIS/REPORT CRITERIA/VALIDATION

A. A sample is acceptable when it meets the following criteria:

1. The internal standards' EICP areas are within a factor of 2(-50%/+100%) of the calibration's internal standards.
2. The internal standards' retention times are within ±30 seconds of the calibration's internal standards.
3. Surrogate recoveries are within the limits stated in Table 4.
4. Any compounds found in the sample are within the linearity range, with any dilutions showing compounds in an acceptable range of linearity. If a sample analysis contains any target compounds above the linear range, all subsequent samples will be reviewed for those target compounds. The target compounds must be below PQL to be acceptable; otherwise they must be reanalyzed.
5. Chromatogram shows no signs of carryover or instrument deficiencies.

B. If sample does not meet internal standards' or surrogates' criteria, it must be reanalyzed. If the reanalysis meets all criteria, it is submitted. If the reanalysis duplicates the results from the first analysis, both analyses are submitted.

C. A sample report is complete when it contains all of the following in order:

1. Chromatogram
2. Quant Report
3. Manual Integrations
4. Spectra of any compounds, excluding internal standards and surrogates.
5. Tentatively Identified Compounds (TICs)
6. Internal Standards/Surrogates

XIII. HEALTH & SAFETY

A. Care must be used when handling standards and samples. All samples should be treated as if they were toxic. Consult the Material Safety Data Sheets for information on the hazards involved with each compound and/or standard mixes. Any spills that may occur are cleaned up immediately.

XIV. DATA ARCHIVAL

A. Instrument data is stored in two separate locations before being archived to ensure data does not become erased or corrupt. Data is stored on both the instrument's Chemstation and the Unix-based processing system. Data is archived from the Unix-based system using the "TAR" format onto 60 m DAT tapes. No more than two months worth of data is archived onto the same tape. Once a tape contains two months of data, a tape directory is printed and bound for permanent storage. Each tape is labeled with the instrument ID and the date range of data contained on the tape.

XV. REFERENCES

A. EPA's "Test Methods for Evaluating Waste" (SW846, 3rd Edition, Rev. 1, July, 1992.)

B. EPA's "Test Methods for Evaluating Waste" (SW846, 3rd Edition, Rev. 2, September, 1994.)

TABLE 1.

Characteristic Ions for Surrogates and Internal Standards for Volatile Organic Compounds

Compound	Primary Ion	Secondary Ion(s)
SURROGATES		
4-Bromofluorobenzene	95	174, 176
1,2-Dichloroethane d-4	65	102
Toluene d-8	98	70, 100
INTERNAL STANDARDS		
Bromochloromethane	128	49, 130, 51
1,4-Difluorobenzene	114	63, 88
Chlorobenzene d-5	117	82, 119

TABLE 2.

Characteristic Ions for Volatile TCL Compounds

Parameter	Primary Ion	Secondary Ion(s)
Chloromethane	50	52
Bromomethane	94	96
Vinyl chloride	62	64
Chloroethane	64	66
Methylene chloride	84	49, 51, 86
Acetone	43	58
Carbon disulfide	76	78
1,1-Dichloroethene	96	61, 98
1,1-Dichloroethane	63	65, 83, 85, 98, 100
1,2-Dichloroethene (total)	96	61, 98
Chloroform	83	85
1,2-Dichloroethane	62	64, 100, 98
2-Butanone	43	57,72
1,1,1-Trichloroethane	97	99,117,119
Carbon tetrachloride	117	119, 121
Bromodichloromethane	83	85, 129
1,1,2,2-Tetrachloroethane	83	85, 131, 133, 166
1,2-Dichloropropane	63	65, 114
trans-1,3-Dichloropropene	75	77
Trichloroethene	130	95, 97, 132
Dibromochloromethane	129	208, 206
1,1,2-Trichloroethane	97	83, 85, 99, 132, 134
Benzene	78	
cis-1,3-Dichloropropene	75	77
Bromoform	173	171, 175, 250, 252, 254, 256
2-Hexanone	43	58, 57, 100
4-Methyl-2-pentanone	43	58, 100
Tetrachloroethene	164	129, 131, 166
Toluene	92	91
Chlorobenzene	112	114
Ethyl Benzene	106	91
Styrene	104	78, 103
Xylenes (total)	106	91
Vinyl Acetate	43	86
2-Chloroethyl Vinyl Ether	63	106, 43

TABLE 3.

BFB Key Ions and Abundance Criteria

Mass	Ion Abundance Criteria
50	15.0 - 40.0 percent of mass 95
75	30.0 - 60.0 percent of mass 95
95	base peak, 100 percent relative abundance
96	5.0 - 9.0 percent of mass 95
173	less than 2.00 percent of mass 174
174	greater than 50.0 percent of mass 95
175	5.0 - 9.0 percent of mass 174
176	greater than 95.0 percent but less than 101.0 percent of mass 174
177	5.0 - 9.0 percent of mass 176

TABLE 4.

Surrogate Recovery Limits

Fraction	Surrogate	Water	Soil
Volatile	Toluene-d_8	88-110	81-117
Volatile	4-Bromoflourobenzene	86-115	74-121
Volatile	1,2-Dichloroethane-d_4	76-114	70-121

TABLE 5.

Matrix Spike Recovery Limits

Fraction	Matrix Spike Compound	Water	Soil
Volatile	1,1-Dichloroethene	61-145	59-172
Volatile	Trichloroethene	71-120	62-137
Volatile	Chlorobenzene	75-130	60-133
Volatile	Toluene	76-125	59-139
Volatile	Benzene	76-127	66-142

TABLE 6.

Laboratory Control Spike Recovery Limits

Fraction	Laboratory Control Spike Compound	Water	Soil
Volatile	Acetone	53-146	30-160
Volatile	Bromodichloromethane	48-164	71-124
Volatile	Benzene	70-134	79-128
Volatile	Bromoform	34-151	58-119
Volatile	Bromomethane	50-191	46-161
Volatile	Chlorobenzene	72-135	80-125
Volatile	Carbon Disulfide	50-162	52-129
Volatile	cis-1,2-Dichloroethene	54-153	80-128
Volatile	cis-1,3-Dichloropropene	34-199	63-123
Volatile	Chloroethane	32-212	44-154
Volatile	Chloroform	57-152	79-129
Volatile	Chloromethane	32-161	21-170
Volatile	Carbon Tetrachloride	37-182	65-140
Volatile	Dibromochloromethane	43-157	68-120
Volatile	1,1-Dichloroethene	54-161	60-141
Volatile	1,2-Dichloropropane	40-182	81-122
Volatile	Ethylbenzene	55-147	82-123
Volatile	2-Hexanone	27-176	20-181
Volatile	Methylene Chloride	52-168	77-141
Volatile	4-Methyl-2-Pentanone	23-208	33-163
Volatile	o-Xylene	68-144	72-126
Volatile	Styrene	64-147	71-129
Volatile	Trichloroethene	67-149	71-134
Volatile	trans-1,2-Dichloroethene	74-162	72-133
Volatile	trans-1,3-Dichloropropene	10-164	48-141
Volatile	Toluene	75-140	80-122
Volatile	Tetrachloroethene	61-141	80-125
Volatile	Vinyl Acetate	22-122	17-153
Volatile	Vinyl Chloride	65-167	31-168
Volatile	1,1-Dichloroethane	55-159	75-129
Volatile	1,1,1-Trichloroethane	38-179	66-140
Volatile	1,1,2,2-Tetrachloroethane	14-157	49-127
Volatile	2-Butanone	42-173	16-130
Volatile	1,2-Dichloroethane	59-151	81-132
Volatile	1,1,2-Trichloroethane	40-179	68-120
Volatile	m,p-Xylene	55-151	81-125
Volatile	2-Chloroethyl Vinyl Ether	73-156	D-146

TABLE 7.

Volatile Internal Standards with Corresponding Analytes Assigned for Quantitation

Bromochloromethane	1,4-Difluorobenzene	Chlorobenzene-d_5
Chloromethane	1,1,1-Trichloroethane	2-Hexanone
Bromomethane	Carbon Tetrachloride	4-Methyl-2-Pentanone
Vinyl Chloride	Bromodichloromethane	Tetrachloroethene
Chloroethane	1,2-dichloropropane	1,1,2,2-Tetrachloroethane
Methylene Chloride	trans-1,3-Dichloropropene	Toluene
Acetone	Trichloroethene	Chlorobenzene
Carbon Disulfide	Dibromochloromethane	Ethylbenzene
1,1-Dichloroethene	1,1,2-Trichloroethane	Styrene
1,1-Dichloroethane	Benzene	Xylene (total)
1,2-Dichloroethene (total)	cis-1,3-Dichloropropene	Bromofluorobenzene (surrogate)
Chloroform	Bromoform	Toluene-d_8 (surrogate)
1,2,-Dichloroethane	Vinyl Acetate	
2-Butanone	2-Chloroethyl Vinyl Ether	
1,2-Dichloroethane-d_4,(surrogate)		

TABLE 8.

Volatile Calibration Check Compounds (CCC)

1,1-Dichloroethene Chloroform 1,2-Dichloropropane Toluene Ethylbenzene Vinyl Chloride	Initial Calibration 5,20,50,100,200ppb Max %RSD: 30% Continuing Calibration 50 ppb Max %D: 20%

TABLE 9.

Volatile System Performance Check Compounds (SPCC)

Chloromethane 1,1-Dichloroethane Bromoform 1,1,2,2-Tetrachloroethane Chlorobenzene	Minimum Relative Response factor (RRF): 0.300 (0.100 for Bromoform)

F-45

TABLE 10.

Compound List and
Practical Quantitation Limits (PQL)

Volatiles	CAS Number	Quantitation Limits Water Low µg/L	Quantitation Limits Soil/Sediment µg/Kg
1. Chloromethane	74-87-3	10	10
2. Bromomethane	74-83-9	10	10
3. Vinyl Chloride	75-01-4	10	10
4. Chloroethane	75-00-3	10	10
5. Methylene Chloride	75-09-2	5	5
6. Acetone	67-64-1	10	10
7. Carbon Disulfide	75-15-0	5	5
8. 1,1-Dichloroethene	75-35-4	5	5
9. 1,1-Dichloroethane	75-34-3	5	5
10. 1,2-Dichloroethene (total)	540-59-0	5	5
11. Chloroform	67-66-3	5	5
12. 1,2-Dichloroethane	107-06-2	5	5
13. 2-Butanone	78-93-3	10	10
14. 1,1,1-Trichloroethane	71-55-6	5	5
15. Carbon Tetrachloride	56-23-5	5	5
16. Vinyl Acetate	108-05-4	10	10
17. Bromodichloromethane	7527-4	5	5
17. 1,2-Dichloropropane	78-87-5	5	5
19. cis-1,3-Dichloropropene	10061-01-5	5	5
20. Trichloroethene	79-01-6	5	5
21. Dibromochloromethane	124-48-1	5	5
22. 1,1,2-Trichloroethane	79-00-5	5	5
23. Benzene	71-43-2	5	5
24. trans-1,3,-Dichloropropene	10061-02-6	5	5
25. Bromoform	75-25-2	5	5
26. 4-Methyl-2-Pentanone	108-10-1	10	10
27. 2-Hexanone	591-78-6	10	10
28. Tetrachloroethene	127-18-4	5	5
29. 1,1,2,2-Tetrachloroethane	79-34-5	5	5
30. Chlorobenzene	108-90-7	5	5
31. Ethyl Benzene	100-41-4	5	5
32. Styrene	100-42-5	5	5
33. Xylenes (Total)	1330-20-7	5	5
34. Toluene	108-88-3	5	5
35. 2-Chloroethyl Vinyl Ether	110-75-8	10	10

*Medium Soil/Sediment Practical Quantitation Limits (PQL) for Volatile compounds are 125 times the individual Low Soil/Sediment PQL and reported to two significant figures.

TABLE 11.

Standards Preparation

Compound or Standard Mix	Begin Conc. µg/mL	Aliquot µL	Total Vol.	Final Conc. µg/mL	Volume to be used
Restek Internal Standard					
VOA Internal Std. Mix	2,500	200	20 mL	25	10µL/5mL
Restek Surrogate					
VOA Surrogate Spike Mix	2,500	200	20 mL	25	10µL/5mL
Restek Calibration Mix					
VOA Calibration Mix 1	5,000	100		25	
VOA Calibration Mix 2	2,000	250		25	
VOA Calibration Mix 3	2,000	250		25	
VOA Calibration Mix 4	2,000	250		25	
VOA Calibration Mix 5	2,000	250	20 mL	25	10µL/5mL
*Trichlorofluoromethane	5,000	100		25	
*1,3-Dichlorobenzene	5,000	100		25	
*1,4-Dichlorobenzene	5,000	100		25	
*1,2-Dichlorobenzene	5,000	100		25	
2-Chloroethyl Vinyl ether	10,000	5	2 mL	25	10µL/5mL
Restek VOA Matrix Spike Mix	2,500	40	4 mL	25	10µL/5mL
MEDIUM LEVEL SOIL SPIKES					
Restek VOA Surrogate Spike Mix	2,500	500	5 mL	250	100µL/10 mL
Restek Matrix Spike Mix	2,500	500	5 mL	250	100µL/10mL

*Compound contained in calibration standard, but not required by SW846/8240, PQL Table II

TABLE 12
Instrument Paramenters

PARAMETERS	INSTRUMENT						
	C	I	K	L	N	R	U
Tune File	CTUNE	KTUNE	ITUNE	LANTUNE	NTUNE	RTUNE	UTUNE
Method Files	BFB VOA	BFB VOA	IBFB IVOA	BFB VOA	BFBN VOAN	BFBR VOAR	BFBU VOAU
Software	Target 3.0	Target 3.0	Target 3.0	Target 3.0	Target 3.0	Target 3.0	Target 3.0
Data System	HP 710	HP 715	HP HP710	HP HP710	HP715715	HP HP715	HP715
Tekmar LCS 2000 Method	1	1	1	1	1	1	1
Standby	30°C	30°C	30°C	30°C	30°C	30°C	30°C
Purge	8 min.	8 min.	8 min.	8 min.	8 min.	8 min.	8 min.
Preheat	—	—	—	—	—	—	—
Sample	—	—	—	—	—	—	—
Desorb Preheat	175°C	175°C	175°C	175°C	175°C	175°C	175°C
Desorb	4 min/180°C	4 min/180°C	4 min/180°C	4 min/180°C	4 min/180°C	4 min/180°C	4 min/180°C
Bake	4 min/225°C	4 min/225°C	4 min/225°C	4 min/225°C	4 min/225°C	4 min/225°C	4 min/225°C
Auto Drain	On	Off	Off	On	Off	Off	Off
Bake Gas Bypass	Off	On	On	Off	On	On	On
BGB Delay	—	—	—	—	—	—	—
Valve	185°C	150°C	150°C	100°C	150°C	150°C	150°C
Line	200°C	200°C	150°C	150°C	200°C	200°C	200°C
Mount	45°C	40°C	40°C	40°C	40°C	40°C	40°C
Aux. Heaters	—	—	—	—	—	—	—
2016 Valve	165°C	150°C	150°C	150°C	150°C	150°C	150°C
Line	150°C	175°C	150°C	150°C	175°C	175°C	175°C
2032 Valve	150°C	150°C	150°C	150°C	150°C	150°C	150°C
Line	150°C	175°C	150°C	150°C	175°C	175°C	175°C
Purge Pressure (PSI)	20	20	20	20	20	20	20

TABLE 13.

Response Factor and Concentration Calculation

$$RF = \frac{(A_x)(C_{is})}{(A_{is})(C_x)} \quad \text{and} \quad conc_x = \frac{(A_x)(C_{is})}{(A_{is})(RF_x)}$$

Where:

A_x = Area of characteristic ion for the measured compound.
A_{is} = Area of the characteristic ion for the specified internal standard
C_{is} = Concentration of the internal standard (ng/µL)
C_x = Concentration of the measured compound (ng/µL)
RF_x = Response Factor of the measured compound.

Surrogate Recovery:

$$\% \text{ Surrogate Recovery} = \frac{Qd}{Qa} \times 100\%$$

Where:

Qd = Quantity determined by analysis
Qa = Quantity added to sample

Matrix Spike Recovery and RPD:

$$\text{Matrix Spike Recovery} = \frac{SSR - SR}{SA} \times 100$$

Where:

SSR = Spiked Sample Result
SR = Sample Result
SA = Spike Added

$$\text{Matrix Spike RPD} = \frac{|MSR - MSDR|}{(½)(MSR + MSDR)} \times 100$$

Laboratory Control Spike (LCS) Recovery:

$$\text{LCS Spike Recovery} = \frac{SSR - SR}{SA} \times 100$$

Where:

SSR = Spiked Sample Result
SR = Sample Result
SA = Spike Added

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SOUTHWEST LABORATORY OF OKLAHOMA, INC.

STANDARD OPERATING PROCEDURE 3010A/3050A FOR THE SW846 DIGESTION OF AQUEOUS AND SOIL MATRICES FOR ICP ANALYSIS

MT600B

REV 1.0 — 2/14/96

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Table of Contents

I. PURPOSE 1
II. REAGENTS AND STANDARDS 1
III. APPARATUS AND MATERIALS 2
V. PROCEDURE OVERVIEW 2
V. QA/QC REQUIREMENTS 2
VI. DISSOLVED METALS 4
VII. SW846 DIGESTION METHODS 5
Table 1. Standards 7
Table 2. Aqueous Matrices 8
Table 3. Soil Matrices 9

I. PURPOSE

A. This SOP will give the digestion procedure for soil matrices for the SW846 Method 3050A. These digestion procedures will be used for the preparation of sediments, sludges, and solid waste samples for analysis by ICP spectroscopy.

B. This SOP will give the digestion procedure for aqueous samples for the SW846 Method 3010A. These digestion procedures will be used for the preparation of aqueous samples, drinking water, surface water, domestic and industrial wastewater's, and wastes that contain suspended solids for analysis by ICP spectroscopy.

C. The digestion supervisor will designate the proper method to be used per batch of samples. If the method is not clearly identified on the internal custody forms, check with the Project Officer responsible for the samples before digestion is started.

II. REAGENTS AND STANDARDS

A. Acids used in the preparation of standards and for sample processing must conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society (ACS) and be of the Mallinckrodt ArSelect grade or the equivalent.

1. Nitric Acid ($HN0_3$), concentrated

2. Hydrochloric Acid (HCl), concentrated

3. Hydrochloric Acid (1+1) - Add 500 ml of hydrochloric acid to 400 ml of ASTM type II water and dilute to 1L.

4. Hydrogen Peroxide (30%), H_2O_2

B. Water - For all sample preparations and dilutions, ASTM Type II water will be used. This water is prepared by passing distilled water through a mixed bead of anion and cation exchange resins.

C. Standards - All mixed and individual elemental standards must be NIST traceable or equivalent.

1. SS1 - A stock standard used to make a working spike for the matrix spikes, LCS, & LCSD. See Table 1 for elements and concentration requirements.

2. SS2 - A stock standard used to make a working spike for the matrix spikes, LCS, & LCSD. See Table 1 for elements and concentration requirements.

3. SS3 - A stock standard used to make a working spike for the matrix spikes, LCS, & LCSD. See Table 1 for elements and concentration requirements.

4. SS4 - A stock standard used to make a working spike for the matrix spikes, LCS, & LCSD. See

Table 1 for elements and concentration requirements.

5. SS5 - A stock standard used to make a working spike for the matrix spikes, LCS, & LCSD. See Table 1 for elements and concentration requirements.

III. APPARATUS AND MATERIALS

A. Corning fleakers - 150 ml, or equivalent.

B. Whattman grade 4.0 filter paper or equivalent.

C. Analytical Balance - Capable of accurately weighing to the nearest 0.01g.

D. Disposable plastic funnels.

E. Electric hotplate or equivalent - adjustable and capable of maintaining a temperature of 90-95°C.

F. Disposable Fisher Polypropylene 100 ml Vials or equivalent.

G. Dual Range pH paper.

H. 1 mL Eppendorf Pipette or equivalent

I. Ribbed watch glass or equivalent cover.

J. Class A 100 and 200 ml volumetric flask.

K. Teflon or glass beads.

V. PROCEDURE OVERVIEW

A. Each morning the digestion supervisor will designate the day's work by sample batch and method to be completed. This will be done by giving the Metals Digestion Technician a written list of batches to be done that day.

B. The technician will then locate samples in the walk-in refrigerator, Log all samples out, and bring them back to the Metal Digestion Area.

C. The technician will then make the computer generated digestion log according to the Digestion/ Distillation Log SOP.

D. Follow the appropriate Digestion Method (for aqueous or soil matrices).

V. QA/QC REQUIREMENTS

A. The following QA/QC samples will be designated as XXXYYMMDDIZ. XXX designates the type of sample (PBW or PBS, LCW or LCS, LDW or LDS). YY is the last two digits in the current

MMDD is the month and day, I designates a ICP digestion. Z is the batch letter done that day (if located in batch YYMMDDI1 - Z = "A"; if located in batch YYMMDDI4 - Z = "D").

B. Aqueous QA/QC

1. PBW - Is the Method Blank. Bring ASTM Type II Water up to the 100 ml mark on the fleaker.

2. LCW *- Is the Laboratory Control Sample and is made from the following:

   Add 1 ml of SS1, SS2, & SS3. If any sample in batch requires As, Pb, Se, or Tl by Trace; substitute 1 ml of SS5 for 1 ml of SS2. Then dilute to the 100 ml mark on the fleaker with ASTM Type II water.

3. LDW* - Is the Laboratory Control Sample Duplicate and is made from the following:

   Add 1 ml of SS1, SS2, & SS3. If any sample in batch requires As, Pb, Se, or Tl by Trace; substitute 1 ml of SS5for1 ml of SS2. Then dilute to the 100 ml mark on the fleaker with ASTM Type II water.

4. Matrix Spike & Matrix Spike Duplicates* - This sample is made from digesting spiked sample with an known amount and then comparing it with the original sample. This is designed to provide information about the effect of the sample matrix on the digestion and/or measurement methodology.

   Spike the sample with 1 ml of SS1, SS2, & SS3. If any sample in the batch requires As, Pb, Se, or Tl by Trace ICP, spike the sample with 1 ml SS1, SS2, and SS4.

5. Matrix Duplicate - This sample is made by digesting a second aliquot of the same sample to determine the precision of the method.

C. Soil QA/QC

1. PBS - Is the Method Blank. Add 1.0 gram of glass beads (use teflon beads if silicon is required) into the fleaker.

2. LCS *- Is the Laboratory Control Sample and is made from the following:

   Add 1.0 gram of glass beads (use teflon beads if silicon is required) and 1 ml of SS1, SS2, & SS3. If any sample in batch requires As, Pb, Se, or Tl by Trace ICP; substitute 1 ml of SS5 for 2 ml of SS2.

3. LDS* - Is the Laboratory Control Sample Duplicate and is made from the following:

   Add 1.0 gram of glass beads (use teflon beads if silicon is required) and 1 ml of SS1, SS2, & SS3. If any sample in batch requires As, Pb, Se, or Tl by Trace ICP; substitute 1 ml of SS5 for 2 ml of SS2.

4. Matrix Spike & Matrix Spike Duplicates* - This sample is made from digesting spiked sample with an known amount and then comparing it with the original sample. This is designed to provide

information about the effect of the sample matrix on the digestion and/or measurement methodology.

Spike the sample with 1 ml of SS1, SS2, & SS3. If any sample requires As, Pb, Se, or Tl by Trace ICP, spike the sample with 1 ml SS1, SS2, and SS4.

5. Matrix Duplicate - This sample is made by digesting a second aliquot of the same sample to determine the precision of the method.

D. Frequency Requirements:

1. A PBW, LCW, & LDW (or PBS, LCS, LDS) are required for every batch of samples (maximum number of samples in a batch is 20. The matrix duplicate, spike, and spike duplicate will be done on request per client basis.

E. Calculations: The below calculation is used to calculate the final Concentration of a in-house stock standard. All units must be the same for C_I & C_F, also for V_I & V_F for the formula to be valid.

$$C_F = C_I \times V_I / V_F$$

where: C_F = Final Concentration of standard

C_I = Initial Concentration of standard

V_I = Final Volume of standard

V_F = Initial Volume of standard

Units normally used for concentration are the following:

ug/L = PPB = 1/1000 mg/L

mg/L = PPM = ug/mL = 1000 ug/L

**NOTE - The final concentrations of the LCS, LDS & Matrix Spike ARE noted in TABLE 2 and 3. Before use, the in-house made stock standards must be screened on the ICP and have a 90 - 110 % Recovery of the true value.*

VI. DISSOLVED METALS

A. All designated dissolved metal analysis will be taken through the digestion procedure, unless directed otherwise. If the dissolved metals have not been field filtered, the technician will filter the sample through Whatman Grade 4.0 filter paper (or equivalent) before any digestion steps are taken.

VII. SW846 DIGESTION METHODS

VII. SW846 DIGESTION METHODS

A. Aqueous Matrices (Method 3010A)

1. Take the pH of all samples with a dual range pH paper. Record as <2 or >2 on your digestion log in the pH column. If any sample is >2 pH the digestion supervisor and the Project Officer must be immediately notified for a decision on how to proceed with the sample

2. Shake the sample thoroughly to achieve homogeneity.

3. Transfer 100 ± 0.5 grams of sample to a 250 ml digestion fleaker.

4. Make the appropriate QA/QC samples as described in Section V.

5. Add 3 ml of concentrated $HN0_3$ and 10 ml of (1+1) HCl to the sample.

6. Cover with a watch glass and heat for 2 hours at 92-95°C or until the sample volume is reduced to between 25 and 50 ml. Make certain the sample does not boil. All samples in a batch should be located on the same hot plate.

7. Rinse watch glass into the fleaker with ASTM Type II water. Cool the sample and filter to remove insoluble material that could clog the nebulizer. Rinse fleaker into the filter three times with ASTM Type II water. NOTE: Filtering of the sample is not required for samples that do not contain insoluble material.

8. After the filtering is complete adjust the volume to 100 ml with ASTM Type II water. This will be done by measuring reagent water into a 100 ml Class A volumetric flask and then transferring the water into a Disposable Fisher Polypropylene 100 ml Vials or equivalent. This vial will then be set side by side with the sample to be brought up to volume and be used as a measuring device.

9. Place all samples and QA/QC samples in a container with a copy of the digestion log and give to the ICP group for analysis.

A. Soil Matrices (Method 3050A)

1. Mix the sample thoroughly to achieve homogeneity.

2. Weigh 1.0 ± 0.05 gram portion of sample and transfer to a fleaker.

3. Make the appropriate QA/QC samples as described in Section V.

4. Add 10 ml of (1+1) HNO_3, mix and cover with a watch glass.

5. Heat the sample to 95° C and reflux for 10 minutes <u>without</u> boiling. All samples in a batch should be located on the same hotplate.

6. Allow the sample to cool, then add 5 ml of concentrated HNO_3. Replace the watch glass and reflux for 30 minutes.

   Note: Do <u>not</u> allow the volume to be reduced to less than 5 ml.

7. Allow the sample to cool. then add 2 ml ASTM Type II water and 3 ml of 30% hydrogen peroxi (H_2O_2).

8. Return the beaker for heating to start the peroxide reaction. Heat until effervescence subsides.

9. Continue to add 30 % H_2O_2 in 1.0 ml aliquots with warming until the effer escence is minimal.

   Note: Do not add more than a total of 10 ml 30% H_2O_2.

10. Allow the sample to cool. Add 5.0 ml of concentrated HCl and 10 ml of ASTM Type II water and heat for an additional 15 minutes.

11. Rinse watch glass into the fleaker with ASTM Type II water Cool the sample and filter to remove insoluble material that could clog the nebulizer. Rinse fleaker into the filter three times with ASTM Type II water. NOTE: Filtering of the sample is not required for samples that do not contain insoluble material.

12. After the filtering is complete adjust the volume to 100 ml with ASTM Type II water. This will be done by measuring reagent water into a 100 ml Class A volumetric flask and then transferring the water into a Disposable Fisher Polypropylene 100 ml Vials or equivalent. This vial will then be set side by side with the sample to be brought up to volume for use as a measuring devise.

13. Place all samples and QA/QC samples in a container with a copy of the digestion log and give to the ICP group for analysis.

TABLE 1. STANDARDS

ELEMENT	SS1 (mg/L)	SS2 (mg/L)	SS3 (mg/L)	SS4 (mg/L)	SS5 (mg/L)
Aluminum - Al	200				
Antimony - Sb	50				
Arsenic - As		200		4	4
Barium - Ba	200				
Beryllium - Be	5				
Boron - B			100		
Cadmium - Cd	5				
Calcium - Ca			2000		
Chromium - Cr	20				
Cobalt - Co	50				
Copper - Cu	25				
Iron - Fe	100				
Lead - Pb		50		2	4
Magnesium - Mg			2000		
Manganese - Mn	50				
Molybdenum - Mo			100		
Nickel - Ni	50				
Potassium - K			2000		
Scandium - Sc			50		
Selenium - Se		200		1	4
Silicon - Si			100		
Silver - Ag	5				
Sodium - Na			2000		
Strontium - Sr			100		
Thallium - Tl		200		5	4
Titanium - Ti			100		
Tin - Sn			100		
Vanadium - V	50				
Zinc - Zn	50				

NOTE: All above standards will have maximum a shelf life of 2 months.

TABLE 2. AQUEOUS MATRICES

ELEMENT	ICP* MS/MSD LCS/LCSD VALUES (ug/L)	TRACE* MATRIX SPIKE VALUES (ug/L)	TRACE* LCS/ LCSD VALUES (ug/L)
Aluminum - Al	2000	2000	2000
Antimony - Sb	500	500	500
Arsenic - As	2000	40	40
Barium - Ba	2000	2000	2000
Beryllium - Be	50	50	50
Boron - B	1000	1000	1000
Cadmium - Cd	50	50	50
Calcium - Ca	20000	20000	20000
Chromium - Cr	200	200	200
Cobalt - Co	500	500	500
Copper - Cu	250	250	250
Iron - Fe	1000	1000	1000
Lead - Pb	500	20	40
Magnesium - Mg	20000	20000	20000
Manganese - Mn	500	500	500
Molybdemum - Mo	1000	1000	1000
Nickel - Ni	500	500	500
Potassium - K	20000	20000	20000
Scandium - Sc	500	500	500
Selenium - Se	2000	10	40
Silicon - Si	1000	1000	1000
Silver - Ag	50	50	50
Sodium - Na	20000	20000	20000
Strontium - Sr	1000	1000	1000
Thallium - Tl	2000	50	40
Titanium - Ti	1000	1000	1000
Tin - Sn	1000	1000	1000
Vanadium - V	500	500	500
Zinc - Zn	500	500	500

** Note: The above spikes are made by the following:*

ICP MS/MSD/LCS/LCSD - 1 ml of SS1, SS2, and SS3 spiked into 100 ml of sample or ASTM, Type II water

Trace Matrix Spike - 1 ml of SS1, SS3, and SS4 spiked into 100 ml of sample or ASTM, Type II water

Trace LCS/LCSD - 1 ml of SS1, SS3, and SS5 spiked into 100 ml of sample or ASTM, Type 2 water

TABLE 3. SOIL MATRICES

ELEMENT	ICP* MS/MSD LCS/LCSD VALUES (mg/kg)	TRACE* MATRIX SPIKE VALUES (mg/kg)	TRACE* LCS/ LCSD VALUES (mg/kg)
Aluminum - Al	400	400	400
Antimony - Sb	100	100	100
Arsenic - As	400	8	8
Barium - Ba	400	400	400
Beryllium - Be	10	10	10
Boron - B	200	200	200
Cadmium - Cd	10	10	10
Calcium - Ca	4000	4000	4000
Chromium - Cr	40	40	40
Cobalt - Co	100	100	100
Copper - Cu	50	50	50
Iron - Fe	100	200	200
Lead - Pb	100	4	8
Magnesium - Mg	4000	4000	4000
Manganese - Mn	100	100	100
Molybdemum - Mo	200	200	200
Nickel - Ni	100	100	100
Potassium - K	4000	4000	4000
Scandium - Sc	100	100	100
Selenium - Se	400	2	8
Silicon - Si	200	200	200
Silver - Ag	10	10	10
Sodium - Na	4000	4000	4000
Strontium - Sr	200	200	200
Thallium - Tl	400	10	8
Titanium - Ti	200	200	200
Tin - Sn	200	200	200
Vanadium - V	100	100	100
Zinc - Zn	100	100	100

**Note*: *The above spikes are made by the following:*

ICP MS/MSD/LCS/LCSD - 2 ml of SS1, SS2, and SS3 spiked into 1.0g of sample or beads

Trace Matrix Spike - 2 ml of SS1, SS3, and SS4 spiked into 1.0g of sample

Trace LCS/LCSD - 2 ml of SS1, SS3, and SS5 spiked into 1.0g of beads

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STANDARD OPERATING PROCEDURE FOR THE ANALYSIS OF NITROAROMATICS & NITRAMINES BY HPLC

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Contents

I. SCOPE AND APPLICATION 1

II. SUMMARY OF METHOD 1

III. INTERFERENCES 2

IV. APPARATUS AND MATERIALS 2

V. REAGENTS 3
TABLE 1. Intermediate Standard Stock Solution A 4
TABLE 2. Intermediate Standard Stock Solution B 4
TABLE 3. Intermediate Stock Solution for Spike A 6
TABLE 4. Intermediate Stock Solution for Spike B 6

VI. SAMPLE COLLECTION, PRESERVATION, AND HANDLING 6

VII. PROCEDURE 7

VIII. QUALITY CONTROL 9
TABLE 5. Laboratory Control Spike Recovery QC Limits 10
TABLE 6. Matrix Spike/Matrix Spike Duplicate Recovery QC Limits 10

IX. METHOD PERFORMANCE 11

X. Data Verification 11

XI. REFERENCES 11

XII. SAFETY 11

TABLE 7. PRACTICAL QUANTITATION LIMITS 12
TABLE 8. RETENTION TIMES FOR ANALYTES ON ODS-II AND CN COLUMNS 12
TABLE 9. MULTILABORATORY PRECISION OF METHOD FOR SOIL SAMPLES 13
TABLE 10. MULTILABORATORY ERROR OF METHOD FOR SOIL SAMPLES 14
TABLE 11. INTERLABORATORY ERROR OF METHOD FOR WATER SAMPLESa 14
Figure 1 METHOD 8330 NITROAROMATICS AND NITRAMINES BY HIGH PERFORMANCE CHROMATOGRAPHY (HPLC) 15

APPENDIX A ..... SALTING-OUT EXTRACTION METHOD

APPENDIX B ..... Chromatogram Examples

I. SCOPE AND APPLICATION

A. Method SWL 846-8330, Rev 0, September, 1994 is intended for the analysis of explosives residues. This method is limited to use by analysts experienced in handling and analyzing explosive materials. This method is used to determine the concentration of the following compounds in a water, soil or sediment matrix:

Compound	*Abbrev*	*CAS No*[a]
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine	HMX	2691-41-0
Hexahydro-1,3,5-trinitro-1,3,5-triazine	RDX	121-82-4
1,3,5-Trinitrobenzene	TNB	99-35-4
1,3-Dinitrobenzene	DNB	99-65-0
Methyl-2,4,6-trinitrophenylnitramine	*Tetryl*	479-45-8
Nitrobenzene	NB	98-95-3
2,4,6-Trinitrotoluene	TNT	118-96-7
2,4-Dinitrotoluene	24DNT	121-14-2
2,6-Dinitrotoluene	26DNT	606-20-2
o-Nitrotoluene	2NT	88-72-2
m-Nitrotoluene	3NT	99-08-1
p-Nitrotoluene	4NT	99-99-0
The two additional compounds are analyzed upon the clients request		
2-amino-4,6,dinitrotoluene	2ADNT	355-72-78-2
4-amino-2,6-dinitrotoluene	4ADNT	1946-51-0

[a] Chemical Abstracts Service Registry number

B. All of these compounds are either used in the manufacture of explosives or are the degradation products of compounds used for that purpose. When making stock solutions for calibration, treat each compound as if it were extremely explosive.

C. The practical quantitation limits (PQLs) of target analytes are presented in Table 7.

D. 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.

II. SUMMARY OF METHOD

A. Aqueous samples are diluted 2X (v/v) with methanol, filtered, separated on a Spherisorb ODS-II reverse phase column (or equivalent), determined at 254 nm, and confirmed on an LC-CN column.

B. Aqueous Samples:

1. Direct Injection Method

a. The samples are diluted 2x (v/u) with filtered Methanol, separated on a spherisorb ODS-II reverse phase column (or equivalent), determined at 254nm, and confirmed on a CN reverse phase column.

2. Salting out:

See Appendix A

C. Soil and sediment samples are extracted using acetonitrile in an ultrasonic bath, filtered, and chromatographed as in Section 2.1.

III. INTERFERENCES

A. 2,4,6-TNT, Nitrobenzene, 2-amino-4,6-dinitrotoluene and 4-amino-2,6-dinitrotoluene elute as unresolved peaks on the primary quantitation column used in this procedure. The compounds must be confirmed on the LC-CN column if any are detected above the PQL on the primary ODS column.

B. Tetryl decomposes rapidly in methanol/water solutions, as well as with heat and light. All aqueous samples expected to contain tetryl should be diluted with acetonitrile prior to filtration. All samples expected to contain tetryl should not be exposed to temperatures above room temperature.

C. Degradation products of tetryl appear as a shoulder to the TNT peak.

IV. APPARATUS AND MATERIALS

A. Primary HPLC System

1. *Pump* — HP Model 1050 quaternary system (or equivalent.)
2. *Column* — Spherisorb ODS-II, 5 μm, 250 mm x 4.6 mm ID (or equivalent).
3. *Detector* — HP Model 1050 Variable UV (or equivalent.)
4. *Autosampler* — HP Model 1050 autosampler (or equivalent.)
5. *Data System* — VG Minichrom data system or equivalent.

B. Confirmation HPLC System

1. *Pump* — Waters model 510 high pressure pump capable of 6000 psi at 10 ml/min. or equivalent.
2. *Column* — Supelco LC-CN, 5 μm, 25cm x 4.6mm ID (or equivalent).
3. *Detector* — Waters model 486 variable wavelength Detector set at 254 nm or equivalent
4. *Autosampler* — Waters model 715 WISP or equivalent.
5. *Data System* — VG Minichrom or equivalent.

C. Other Equipment

1. 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.
2. BLUE M Convection Heating Oven with the temperature range of 0-300°C or equivalent.
3. Balance ± 0.0001 g.

D. Materials

1. *Filters* — Gelman Accuspec CR 0.45 μm filters for filtering extracts, or equivalent
2. *Filters* — Supelco Nylon 66 0.2 μm x 47 mm membranes for filtering chromatographic solvents.
3. *Pipettes* — volumetric, Class A, glass, 50 mL, 10 mL, 5 mL, 4 mL, 2 mL, 1 mL
4. *Vials* — 20 mL, glass.
5. *Vials* — 15 mL, glass, Teflon lined screw cap or crimp top.
6. *Vials* — 40 mL VOA vials with Teflon lined screw caps.
7. *Syringes* — 3 mL and 10 mL
8. *Volumetric flasks* — Class A - 10 mL, 20 mL, 50 mL, 100 mL, 200 mL, 250 mL.
9. *Mortar and pestle.*
10. *SMI glass pipettor* — 50-250 μL and 10-60 μL.

V. REAGENTS

A. 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.

1. Acetonitrile, CH_3CN — HPLC grade.
2. Methanol, CH3OH — HPLC grade.
3. Water, H_2O — HPLC grade.

B. Stock Standard Solutions. Analyte Standards obtained from AccuStandard at 1000 μg/mL.

1. HMX — Standard Analytical Reference Material.
2. RDX — Standard Analytical Reference Material.
3. DNB — Standard Analytical Reference Material.
4. Tetryl — Standard Analytical Reference Material.
5. TNT — Standard Analytical Reference Material.
6. 2,4-DNT — Standard Analytical Reference Material.
7. 2,6-DNT — Standard Analytical Reference Material.

8. 1,3,5-TNB — Standard Analytical Reference Material.
9. NB — Standard Analytical Reference Material.
10. 2-NT — Standard Analytical Reference Material.
11. 3-NT — Standard Analytical Reference Material.
12. 4-NT — Standard Analytical Reference Material.
13. 2ADNT — Standard Analytical Reference Material.
14. 4ADNT — Standard Analytical Reference Material.

C. Intermediate Stock Standards Solutions

1. 2ADNT and 4ADNT are incompletely resolved from TNT and NB, therefore prepare two separate intermediate stock standard solutions in methanol according to the examples in Tables 1 and 2.

TABLE 1.
Intermediate Standard Stock Solution A

Compound	AccuStandard #	Orig. Conc. (µg/mL)	Aliquot (mL)	Final Conc. (µg/mL)
HMX	M-8330-04	1000	0.600	60.00
RDX	M-8330-05	1000	0.250	25.00
TETRYL	M-8330-10	1000	0.220	2[illegible]
TNT	M-8330-11	1000	0.160	16[illegible]
2,4-DNT	M-8330-02	1000	0.140	14.00
2,6-DNT	M-8330-03	1000	0.230	23.00
1,3-DNB	M-8330-01	1000	0.96	9.60
1,3,5-TNB	M-8330-12	1000	0.179	17.90
NB	M-8330-06	1000	0.195	19.50
2NT	M-8330-07	1000	0.200	20.00
3NT	M-8330-08	1000	0.190	19.00
4NT	M-8330-09	1000	0.200	20.00
Total Volume is 10.0 mL				

TABLE 2.
Intermediate Standard Stock Solution B

Compound	AccuStandard #	Orig. Conc. (µg/mL)	Aliquot (mL)	Final Conc. (µg/mL)
2ADNT	M-8330-13	1000	0.500	50.00
4ADNT	M8330-14	1000	0.460	46.00
Total Volume is 10.0 mL				

These immediate stock standard solutions are kept at 4°C in the dark for up to 30 days (1 m

2. The two concentrated intermediate stock solutions are diluted with methanol, refrigerated at 4°C and kept in the dark upon preparation.

E. Working Standards

1. Prepare working standards daily by diluting intermediate stock standards by 50:50 v/v with a 5 g/L (0.5%) Calcium Chloride solution.

2. Following SW-846 method 8330, Sep. 1994, five-level intermediate standards have been prepared in the manner such that the low level standard is at or near the MDL, and the high level standard is at detector linear range. Each level is injected one time.

F. 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 (see examples in Tables 3 and 4.) LCS contains the analytes of interest at midpoint level or higher.

G. MS (Matrix Spike) and MSD (Matrix Spike Duplicate)

MS and MSD are prepared as V.F. Spike the sample at midpoint or higher calibration range. (See examples in Tables 3 and 4.)

H. Surrogate

Since method doesn't specify any particular surrogate, SWLO, is in the process of finding a non-target surrogate which doesn't co-elute with any target analytes. Preliminary indicators are to use a target ISOMER 3,4 DNT. This will be tested as a surrogate and if successful will be given temporary QC limits of (60-140) percent recovery to generate control limits..

I. Eluent

1. To prepare 1 liter 50% Methanol/H_2O for mobile phase, add 500 ml of methanol to 500 mL of HPLC water.

2. To prepare 1 liter of 50% Acetonitrile/H_2O, add 500 ml of Acetonitrile to 500 ml of HPLC water.

NOTE: Both eluents can be used to analyze high level water. However, if HMX is an important target, 50% methanol/H_2O is preferred.

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

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

F-69

TABLE 3.
Intermediate Stock Solution for Spike A

Compound	AccuStandard #	Orig. Conc. (µg/mL)	Aliquot (µL)	Final Conc. (µg/mL)
HMX	M-8330-04	1000	1920	64.0
RDX	M-8330-05	1000	750	25.0
TETRYL	M-8330-10	1000	600	20.0
TNT	M-8330-11	1000	465	15.5
2,4-DNT	M-8330-02	1000	420	14.0
2,6-DNT	M-8330-03	1000	690	23.0
1,3-DNB	M-8330-01	1000	288	9.6
1,3,5-TNB	M-8330-12	1000	459	15.3
NB	M-8330-06	1000	585	19.5
2NT	M-8330-07	1000	870	29.0
3NT	M-8330-08	1000	570	19.0
4NT	M-8330-09	1000	600	20.0
Total Volume is 30.0 µL				

TABLE 4.
Intermediate Stock Solution for Spike B

Compound	AccuStandard #	Orig. Conc. (µg/mL)	Aliquot (µL)	Final Conc. (µg/mL)
2ADNT	M-8330-13	1000	0.688	68.8
4ADNT	M8330-14	1000	0.752	75.2
Total Volume is 10.0 µL				

VI. SAMPLE COLLECTION, PRESERVATION, AND HANDLING

A. Grab samples must be collected and stored in glass containers. Follow conventional sampling procedures.

B. Samples must be kept below 4°C from the time of collection prior to analysis.

C. 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 subsample on a wet sample. If wet soil samples are to be analyzed, a moisture determination must be made on a separate subsample.

F-70

VII. PROCEDURE

A. Sample Preparation

1. Aqueous Samples

Sample Filtration. Place a 5 mL portion of each water sample in a scintillation vial, add 5 mL of methanol, shake thoroughly, and filter through a 0.5 um filter. Discard the first 3 mL of filtrate, and retain the remainder for analysis.

2. Soil and Sediment Samples

a. Sample homogenization. Dry soil samples in air at room temperature or colder, being careful not to expose the samples to direct sunlight. Grind sample thoroughly in an acetonitrile rinsed mortar.

b. Sample extraction. Place a 2.0 g subsample of each soil sample in a 40 mL glass voa. Add 10.0 mL of acetonitrile, cap with teflon lined cap, vortex swirl for one minute, and place in ultrasonic bath for 18 hours. Keep ultrasonic bath at room temperature or below.

c. After sonication, allow sample to settle for 30 minutes. Place supernatant in syringe and filter through a Gelman Accuspec CR 0.45 μm. Discard first 1 mL and retain remainder in 15 mL glass with teflon-lined screwcap. Keep the extracts in 4°C and in a dark place until analysis.

d. Prior to analysis, dilute each extract 2X with 0.5% calcium chloride solution.

B. Chromatographic Conditions

Mobile Phase: 50/50 (v/v) methanol/HPLC water

Flow rate: 1.0 mL/min

Injection volume: 200 ml

UV Detector: 254 nm

NOTE: Number of standards are analyzed prior to the actual initial calibration standard in order to make certin that the HPLC is within operating conditions and the system has established the retention time windows.

C. Calibration of HPLC

1. Following SW-846 method 8330, September 1994, the initial calibration standard at the minimal 5 concentration levels is injected only one time. The %RSD of the calibration factors should be $\leq$ 20%. The curve should be linear and intercept zero. Use the mean calibration factors for all calculations as long as continuing calibrations described below meet specifications.

2. Calibration Factor (CF): 5-Level

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

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

F-71

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

Where:

$\overline{CF}$ = Mean of the calibration factor
n = (5) Total number of values
STD = Standard deviation of the calibration factors
CF_1 = Calibration factors

3. Continuing Calibration using the mid point standards should be injected at the beginning of an analysis day and about every ten injections and at the end of the run. These standards should agree within a percent deviation of no more than ± 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.
4. Percent Deviation (%D)

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

Where:

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

5. LC-CN column or equivalent is used to confirm compounds. Nitrotoluenes are prepared separately due to coelution. 2ADNT and 4ADNT are prepared separately from the remainder of the compounds. Only the compounds detected above the PQL are confirmed.
6. Retention windows should be established for each new calibration. Average the retention time for each injection of each component for all initial calibration runs and continuing calibration runs. Determine the standard deviation. The window for any compound is ± 3 times the standard deviation. If chromatographic retention drift should occur, make sure 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.

D. Sample Analysis

1. Analyze the samples using the chromatographic conditions given in VII.B. Confirm each measurement by injecting onto the CN column.
2. Table 8 presents the retention times for the analytes on both the ODS-II and CN col Appendix C presents typical chromatograms.

F-72

3. 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	=	Calibration factor (ng)
VO	=	Volume of water (g) or amount of soil extracted (mL)
VI	=	Volume of extract injected (μL)
VT	=	Volume of the concentrated extracted (μL)
DF	=	Dilution factor

VIII. QUALITY CONTROL

A. 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.

B. Method Blanks

1. Method blanks for the analysis of aqueous samples should be organic-free reagent water carried through all sample storage and handling procedures.
2. Method blanks for the analysis of soil samples should be uncontaminated soil carried through all sample storage, extraction, and handling procedures.

C. Laboratory Control Spikes

(See Table 5 for limits)-LCS are done with each extraction batch at a rate of one LCS per 20 samples or less.

D. Matrix Spikes

(See table 6 for limits)-Matrix spike/ Matrix spike duplicates are analyzed at a rate of 1 set per batch of 20 samples or fewer.

E. Surrogate

Information to be determine upon, sucessfully determination of surrogate compound.

F-73

TABLE 5.
Laboratory Control Spike Recovery QC Limits

Compound	Low Level Water % Recovery QC Limits	High Level Water %Recovery QC Limits	Soil %Recovery QC Limits
HMX	28-117	46-151	57-137
RDX	25-114	72-129	77-113
TNB	24-129	74-118	64-125
TETRYL	25-119	58-120	23-124
DNB	22-117	79-132	82-118
TNT	45-118	61-145	75-118
NB	18-119	68-135	69-143
26DNT	34-126	77-125	78-117
24DNT	35-119	70-134	74-126
2NT	27-119	70-131	78-123
4NT	28-118	73-116	79-125
3NT	27-115	71-127	78-121
2ADNT	ND	ND	ND
4ADNT	ND	ND	ND

TABLE 6.
Matrix Spike/Matrix Spike Duplicate Recovery QC Limits

Compound	Water %Recovery QC Limits	Water%RPD QC Limits	Soil %Recovery QC Limits	Soil %RPD QC Limits
HMX	44-145	30	54-128	30
RDX	66-139	30	66-109	30
TNB	56-129	30	57-130	30
TETRYL	76-120	45	32-119	45
DNB	76-122	30	74-122	30
TNT	76-137	35	44-124	35
NB	82-111	30	70-135	30
26DNT	80-118	35	58-125	35
24DNT	72-123	35	59-120	35
2NT	74-130	35	79-124	35
4NT	78-116	35	74-128	35
3NT	72-126	35	79-121	35
2ADNT	ND	ND	ND	ND
4ADNT	ND	ND	ND	ND

ND = not determined

F-74

IX. METHOD PERFORMANCE

A. Method 8330 was tested by six laboratories. The results of this testing indicate that the results presented in Tables 9 through 11 are to be expected.

B. See attached chromatograms of standards obtained with this procedure (Appendix B).

X. DATA VERIFICATION

A. If the compound is found above the PQL 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 all the compounds in the QA/QC samples exceeded the established laboratory QC limits, the batch of samples will be reextracted and reanalyzed. If some of the compounds in the QA/QC samples are out of the established limits, the compounds will be flagged and the data will be reported as is.

XI. REFERENCES

A. 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.

B. 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.

C. 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.

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

XII. SAFETY

Standard precautionary measures used for handling other organic compounds should be sufficient for safe handling of the analytes targeted by Method 8330.

F-75

TABLE 7.
PRACTICAL QUANTITATION LIMITS

Compound	Low Level Water (µg/L)	High Level Water (µg/L)	Soil (µg/kg)
HMX	1.0	13.0	2200
RDX	0.84	14.0	1000
TNB	0.26	7.3	250
DNB	0.11	4.0	250
Tetryl	0.25	10.0	650
NB	0.30	7.0	260
TNT	0.12	6.4	250
24DNT	0.08	5.7	250
26DNT	0.31	9.4	260
2NT	0.78	12.0	250
3NT	0.65	7.9	250
4NT	0.65	8.0	250
2ADNT	0.03	ND	250
4ADNT	0.06	ND	250

ND = not determ

TABLE 8.
RETENTION TIMES FOR ANALYTES ON ODS-II AND CN COLUMNS

ODS-II		*CN*	
Analyte	Retention Time (min)	Analyte	Retention Time (min)
HMX	3.2	NB	5.5
RDX	4.3	2NT	6.1
TNB	5.4	3NT	6.1
Tetryl	6.0	4NT	6.1
DNB	6.8	DNB	6.2
TNT	7.4	TNB	6.3
NB	7.7	26TNT	6.8
26DNT	8.8	24DNT	7.2
24DNT	9.6	TNT	7.5
2NT	11.0	RDX	10.2
4NT	11.8	Tetryl	12.3
3NT	12.5	HMX	16.3
2A DNT	7.8	2ADNT	8.7
4A DNT	7.3	4ADNT	7

F - 76

TABLE 9.
MULTILABORATORY PRECISION OF METHOD FOR SOIL SAMPLES

Spiked soils	*Field-contaminated soils*					
	Mean Concentration (mg/g)	**SD**	**%RSD**	**Mean Concentration (mg/g)**	**SD**	**%RSD**
HMX	46.0	1.7	3.7	14.0 153.0	1.8 21.6	12.8 14.1
RDX	60.0	1.4	2.3	104.0 877.0	12.0 29.6	11.5 3.4
TNB	8.6 46.0	0.4 1.9	4.6 4.1	2.8 72.0	0.2 6.0	7.1 8.3
DNB	3.5	0.14	4.0	1.1	0.11	9.8
tetryl	17.0	3.1	17.9	2.3	0.41	18.0
TNT	40.0	1.4	3.5	7.0 669.0	0.61 55.0	9.0 8.2
24DNT	5.0	0.17	3.4	1.0	0.44	42.3

TABLE 10.
MULTILABORATORY ERROR OF METHOD FOR SOIL SAMPLES

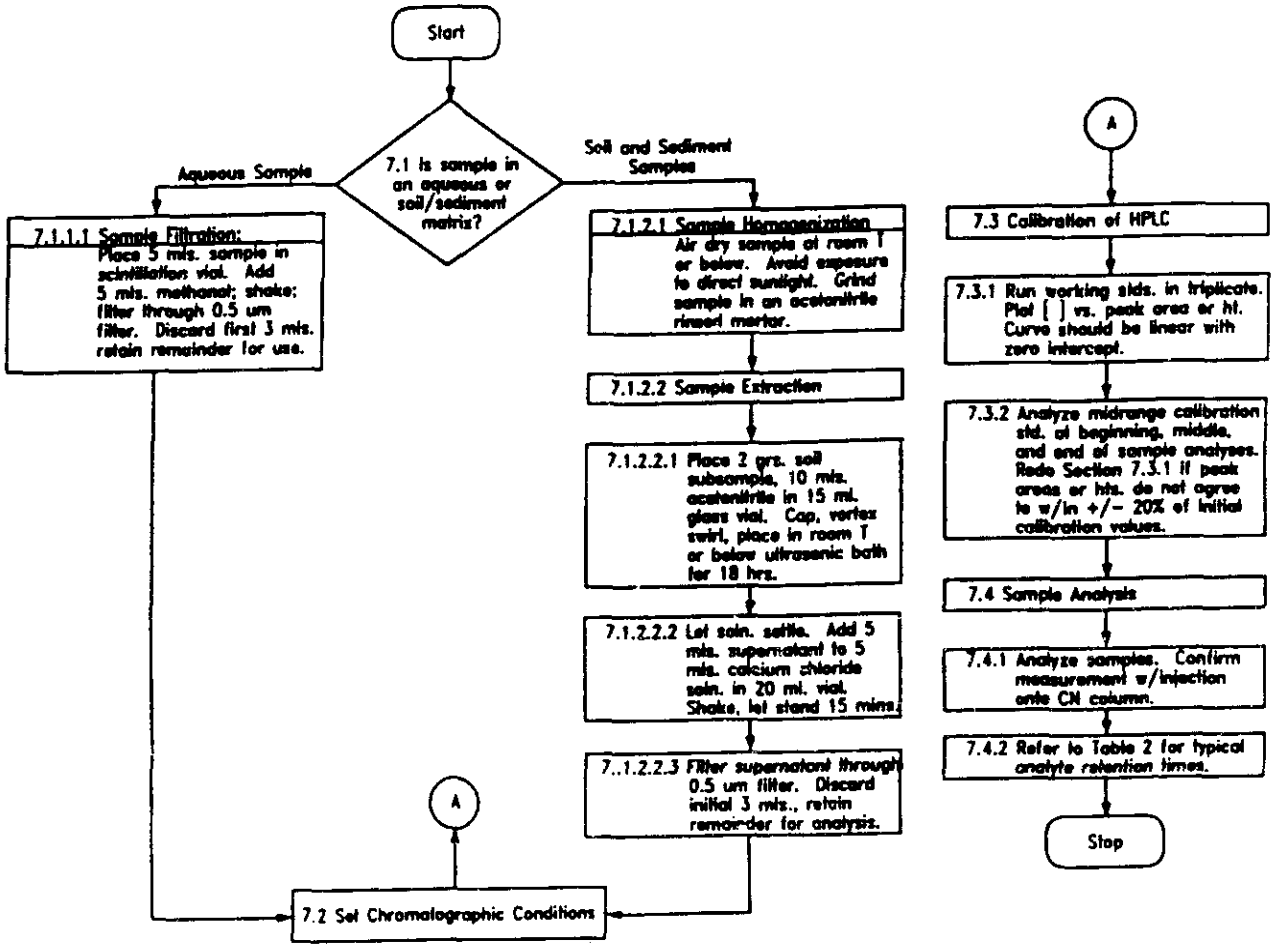
	Spiked soils			*Field contaminated soils*		
	Mean Concentration (mg/g)	**SD**	**%RSD**	**Mean Concentration (mg/g)**	**SD**	**%RSD**
HMX	46.0	2.6	5.7	14 153	3.7 37.3	26.0 24.0
RDX	60.0	2.6	4.4	104 877	17.4 67.3	17.0 7.7
TNB	8.6 460	0.61 2.97	7.1 6.5	2.8 72.0	0.23 8.8	8.2 12.2
DNB	3.5	0.24	6.9	1.1	0.16	14.5
tetryl	17.0	5.22	30.7	2.3	0.49	21.3
TNT	40.0	1.88	4.7	7.0 669.0	1.27 63.4	18.0 9.5
24DNT	5.0	0.22	4.4	1.0	0.74	74.0

TABLE 11.
INTERLABORATORY ERROR OF METHOD FOR WATER SAMPLES[a]

	Sample 1		**Sample 2**	
	Mean conc. (mg/L)	**%RSD**	**Mean conc. (mg/L)**	**%RSD**
HMX	nd	-	184.0[b]	8.4
RDX	431.0	22.9	2117.0	29.5
TNB	74.3	3.2	27.6[c]	4.2
TNT	10635.0	59.4	1746.0	26.8

a 10 replicate determinations, except where noted
b 6 replicate determinations
c 7 replicate determinations

Figure 1
METHOD 8330
NITROAROMATICS AND NITRAMINES BY
HIGH PERFORMANCE CHROMATOGRAPHY (HPLC)



F-79

APPENDIX A

SALTING-OUT EXTRACTION METHOD FOR LOW-LEVEL DETERMINATION OF NITROAROMATICS AND NITRAMINES IN GROUNDWATER BY SW 846 METHOD 8330, SEPTEMBER, 1994.

METHOD

A. Low Level Method (Salting-out Extraction)

1. Add 251.3 grams of sodium chloride to a 1 liter volumetric flask (round.) Measure out 770 mL of a water sample (using a 1 liter graduated cylinder) and transfer it to the volumetric flask containing the salt. Add a stir bar and mix the contents at maximum speed on a magnetic stirrer until the salt is completely dissolved.

2. Add 164 mL of acetonitrile (measured with a 250 mL graduated cylinder) while the solution is being stirred and stir for an additional 15 minutes. Turn off the stirrer and allow the phases to separate for ten minutes.

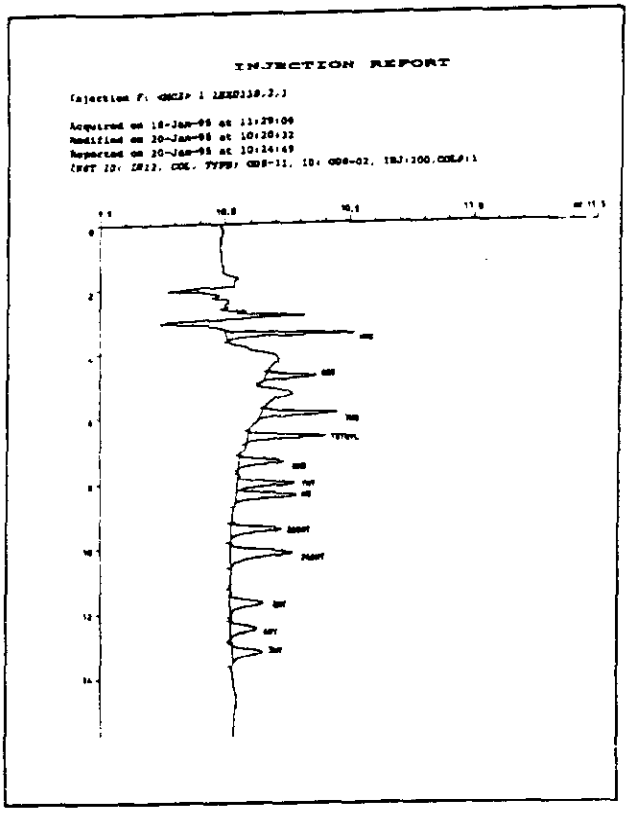
3. Remove the acetonitrile (upper) layer (about 8 mL) with a Pasteur pipet and transfer it to a 100 mL volumetric flask (round.) Add 10 mL of fresh acetonitrile to the water sample in the 1 liter flask. Again stir the contents of the flask for 15 minutes follo by 10 minutes for phase separation. Combine the second acetonitrile portion with initial extract. The inclusion of a few drops of salt water at this point is unimport

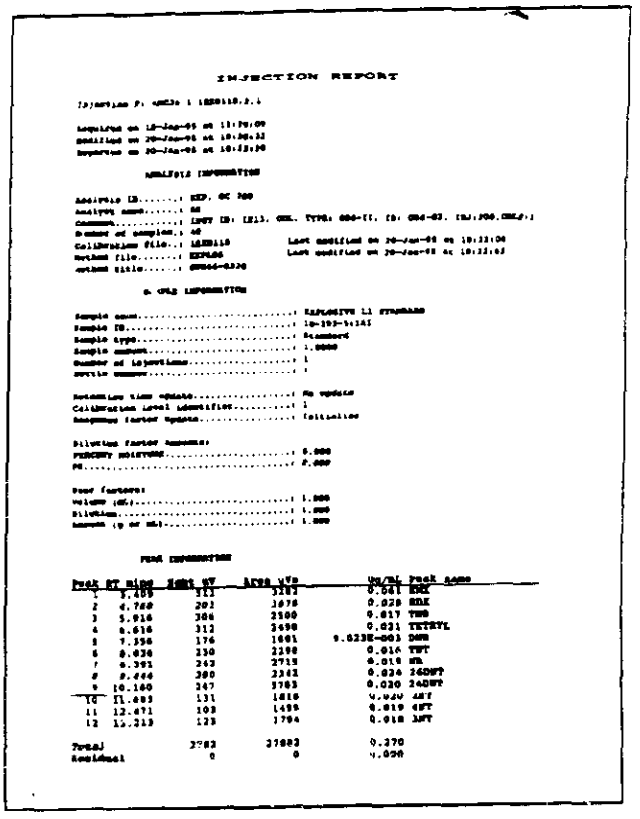
4. Add 84 mL of salt water (325 grams NaCl) per 1000 mL of reagent water) to the acetonitrile extract in the 100 mL volumetric flask. Add a stir bar and stir the contents on a magnetic stirrer for 15 minutes followed by 10 minutes for phase separation. Carefully transfer the acetonitrile phase to a 10 mL graduated cylinder using a Pasteur pipet. At this stage the amount of water transferred with the acetonitrile must be minimized. The water contains a high concentration of NaCl that produces a large peak at the beginning of the chromatogram where it could interfere with the HMX determination.

5. Add an additional 1.0 mL of acetonitrile to the 100 mL volumetric flask. Again stir the contents of the flask for 15 minutes followed by 10 minutes for phase separation. Combine the second acetonitrile portion with the initial extract in the 10 mL graduated cylinder (transfer to a 25 mL graduated cylinder if the volume exceeds 5 mL.) Record the total volume of acetonitrile extract [V(t)] in the calculation of concentration after converting to μL.) The resulting extract is 5 mL final volume which is then diluted 1:1 with reagent water prior to analysis.

6. If the diluted extract is turbid, filter it through a 0.45 μ teflon filter using a disposable syringe. Discard the first 0.5 mL of filtrate, and retain the remainder in a Teflon-capped vial for RP-HPLC analysis.

APPENDIX B


INJECTION REPORT
TETRYL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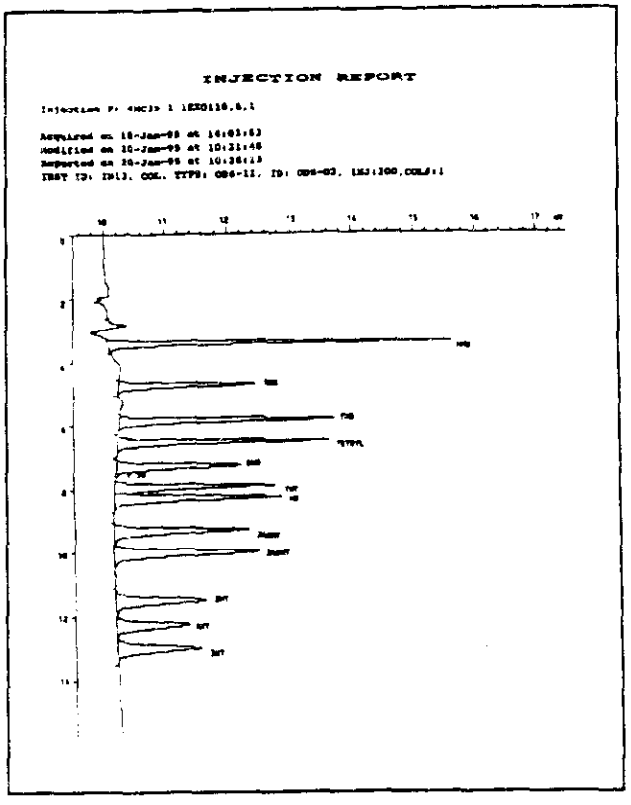
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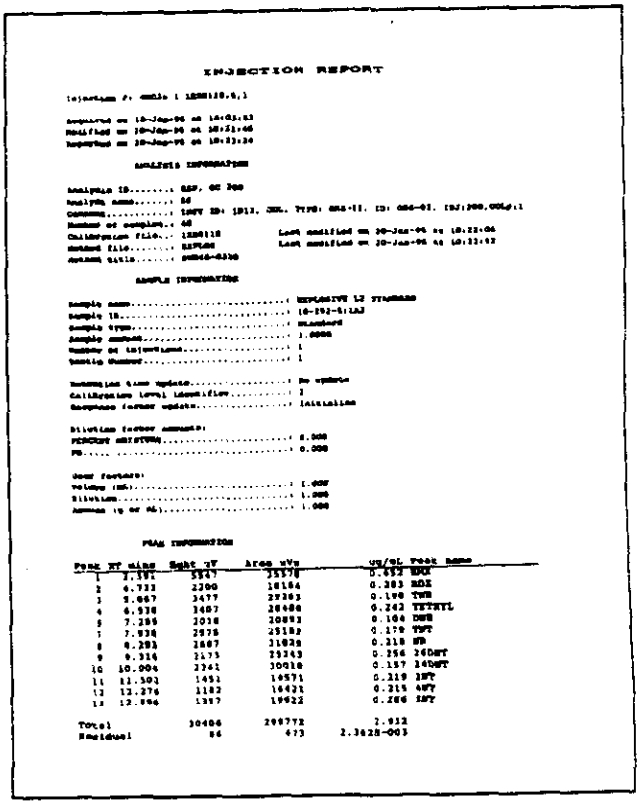
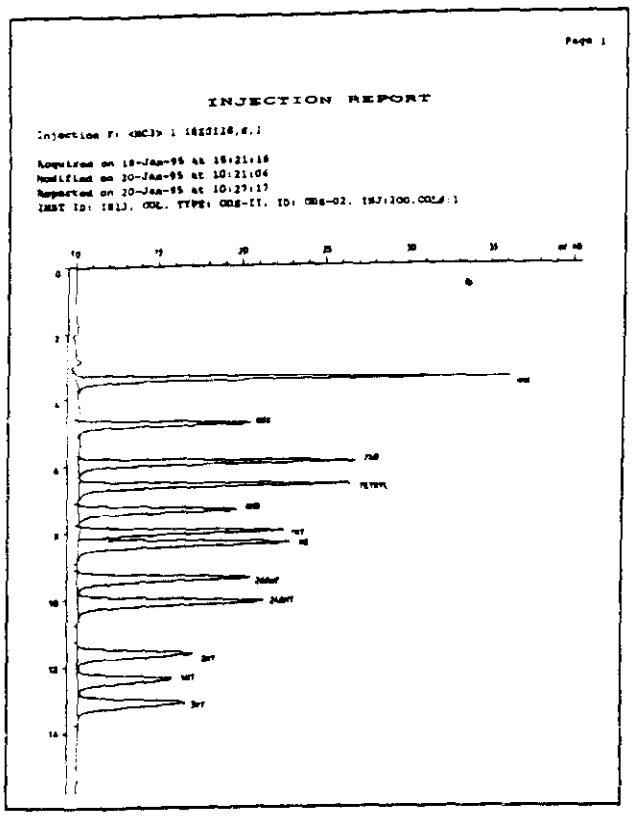
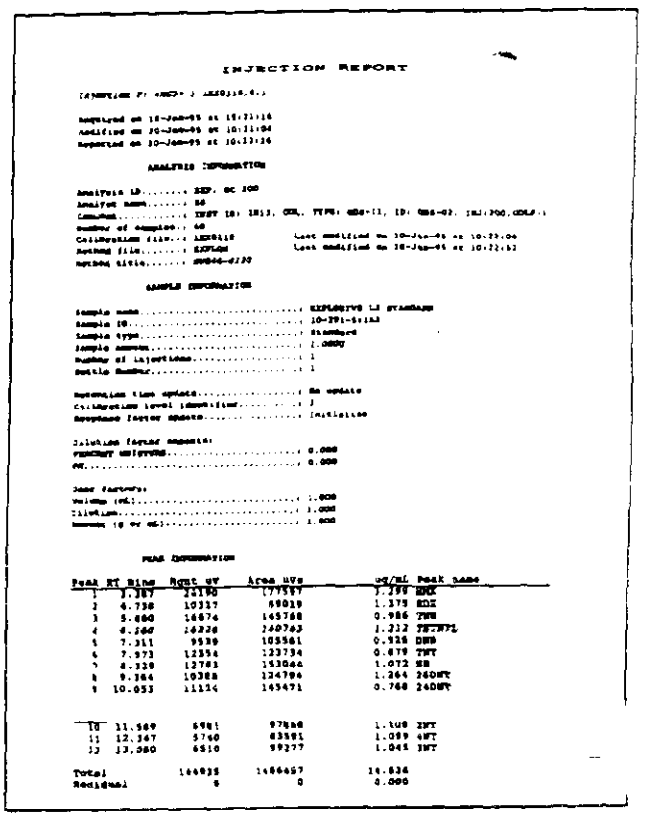
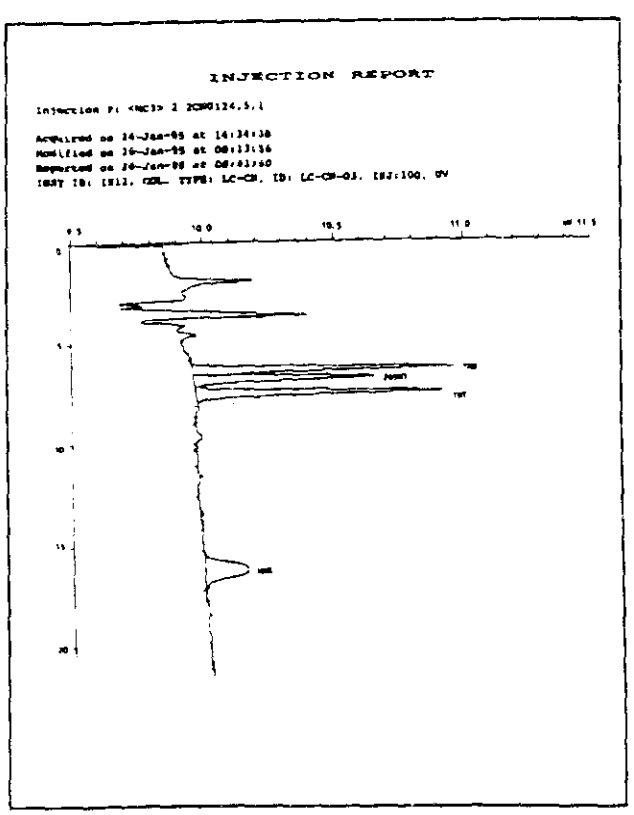

INJECTION REPORT

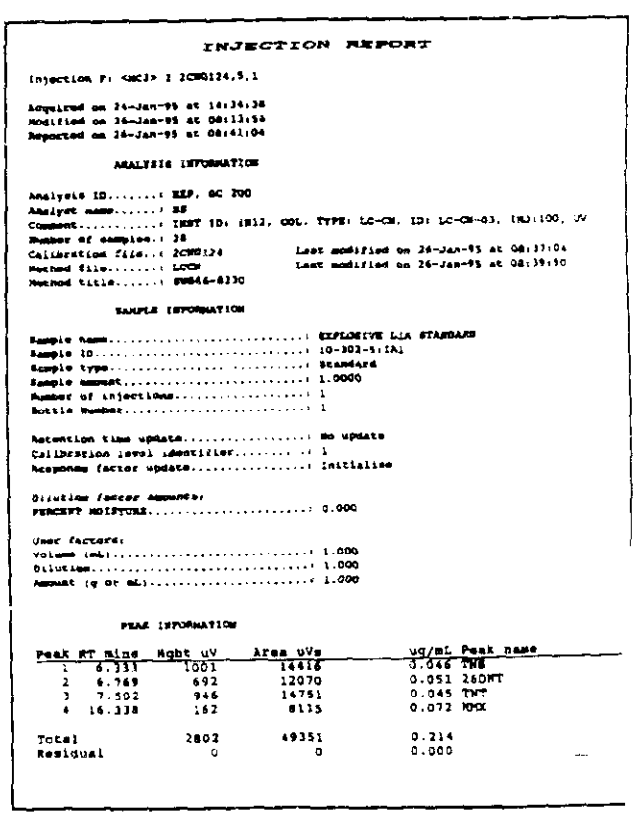
APPENDIX B

(continued)









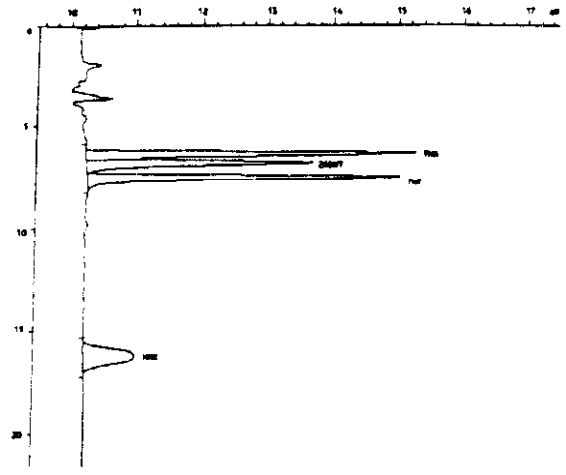
APPENDIX B

(continued)

INJECTION REPORT

Injection F: <NCJ> 2 2CH0124.6.1

Acquired on 24-Jan-95 at 15:11:55
Modified on 26-Jan-95 at 08:14:06
Reported on 26-Jan-95 at 08:41:58
INST ID: IN12, COL. TYPE: LC-CN, ID: LC-CN-03, INJ:100, UV



INJECTION REPORT

Injection F: <NCJ> 2 2CH0124.6.1

Acquired on 24-Jan-95 at 15:11:55
Modified on 26-Jan-95 at 08:14:06
Reported on 26-Jan-95 at 08:41:05

ANALYSIS INFORMATION

Analysis ID.....: EXP. GC 200
Analyst name.....: SS
Comment.....: INST ID: IN12, COL. TYPE: LC-CN, ID: LC-CN-03, INJ:100, UV
Number of samples.: 28
Calibration file..: 2CH0124 Last modified on 26-Jan-95 at 08:37:04
Method file.....: LCCN Last modified on 26-Jan-95 at 08:39:50
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: EXPLOSIVE LJA STANDARD
Sample ID.....: 10-301-5:IAJ
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.....: Initialize

Dilution factor amounts:
PERCENT MOISTURE.....: 0.000

User factors:
Volume (mL).....: 1.000
Dilution.....: 1.000
Amount (g or mL).....: 1.000

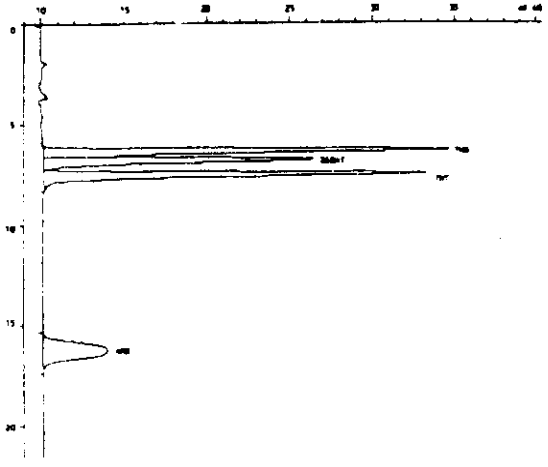
PEAK INFORMATION

Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
1	6.342	5031	71975	0.229	TNB
2	6.791	3436	59350	0.251	26DNT
3	7.516	4770	75032	0.229	TNT
4	16.209	788	38992	0.347	HMX
Total		14025	245350	1.057	
Residual		0	0	0.000	

INJECTION REPORT

Injection F: <NCJ> 2 2CH0124.7.1

Acquired on 24-Jan-95 at 15:49:11
Modified on 26-Jan-95 at 08:16:14
Reported on 26-Jan-95 at 08:42:34
INST ID: IN12, COL. TYPE: LC-CN, ID: LC-CN-03, INJ:100, UV



INJECTION REPORT

Injection F: <NCJ> 2 2CH0124.7.1

Acquired on 24-Jan-95 at 15:49:11
Modified on 26-Jan-95 at 08:16:14
Reported on 26-Jan-95 at 08:41:07

ANALYSIS INFORMATION

Analysis ID.....: EXP. GC 200
Analyst name.....: SS
Comment.....: INST ID: IN12, COL. TYPE: LC-CN, ID: LC-CN-03, INJ:100, UV
Number of samples.: 28
Calibration file..: 2CH0124 Last modified on 26-Jan-95 at 08:37:04
Method file.....: LCCN Last modified on 26-Jan-95 at 08:39:50
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: EXPLOSIVE LJA STANDARD
Sample ID.....: 10-300-5:IAJ
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.....: Initialize

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	6.342	24454	360994	1.150	TNB
2	6.800	16293	295223	1.250	26DNT
3	7.520	23052	377196	1.150	TNT
4	16.289	3898	196445	1.750	HMX
Total		67696	1229858	5.301	
Residual		0	0	0.000	

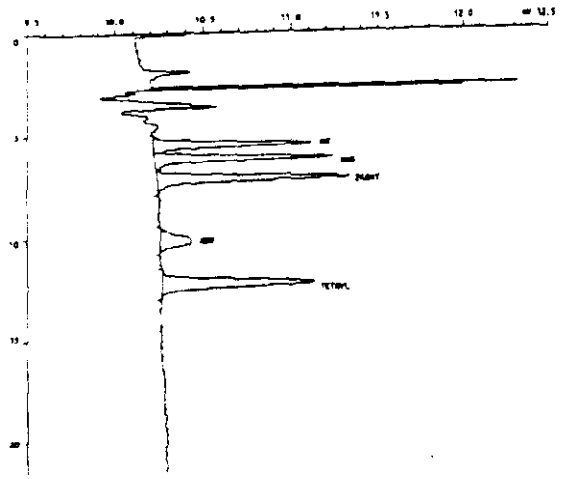
APPENDIX B

(continued)

INJECTION REPORT

Injection F: <MC3> 2 2CH0124.8.1

Acquired on 24-Jan-95 at 16:26:18
Modified on 26-Jan-95 at 08:14:24
Reported on 26-Jan-95 at 08:43:06
INST ID: IH12, COL. TYPE: LC-CN, ID: LC-CN-03, INJ:100, UV



INJECTION REPORT

Injection F: <MC3> 2 2CH0124.8.1

Acquired on 24-Jan-95 at 16:26:18
Modified on 26-Jan-95 at 08:14:24
Reported on 26-Jan-95 at 08:41:08

ANALYSIS INFORMATION

Analysis ID..... EXP, GC 200
Analyst name..... SS
Comment..... INST ID: IH12, COL. TYPE: LC-CN, ID: LC-CN-03, INJ:100, UV
Number of samples.: 28
Calibration file..: 2CH0124 — Last modified on 26-Jan-95 at 08:37:04
Method file..... LOCK — Last modified on 26-Jan-95 at 08:39:50
Method title..... SW846-8330

SAMPLE INFORMATION

Sample name..... EXPLOSIVE L18 STANDARD
Sample ID..... 10-306-9: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..... Initialize

Dilution factor summary:
PERCENT MOISTURE..... 0.000

User factors:
Volume (mL)..... 1.000
Dilution..... 1.000
Amount (g or mL)..... 1.000

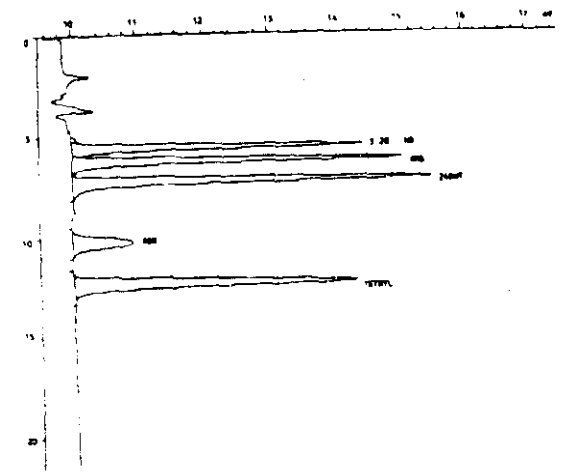
PEAK INFORMATION

Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
1	5.511	901	15143	0.050	NB
2	6.196	1014	18244	0.039	DNB
3	7.209	1095	21289	0.049	24DNT
4	10.236	179	6640	0.046	RDX
5	12.236	873	21909	0.088	TETRYL
Total		4061	63236	0.273	
Residual		0	0	0.000	

INJECTION REPORT

Injection F: <MC3> 2 2CH0124.9.1

Acquired on 24-Jan-95 at 17:03:34
Modified on 26-Jan-95 at 08:37:04
Reported on 26-Jan-95 at 08:43:28
INST ID: IH12, COL. TYPE: LC-CN, ID: LC-CN-03, INJ:100, UV



INJECTION REPORT

Injection F: <MC3> 2 2CH0124.9.1

Acquired on 24-Jan-95 at 17:03:34
Modified on 26-Jan-95 at 08:37:04
Reported on 26-Jan-95 at 08:41:10

ANALYSIS INFORMATION

Analysis ID..... EXP, GC 200
Analyst name..... SS
Comment..... INST ID: IH12, COL. TYPE: LC-CN, ID: LC-CN-03, INJ:100, UV
Number of samples.: 28
Calibration file..: 2CH0124 — Last modified on 26-Jan-95 at 08:37:04
Method file..... LOCK — Last modified on 26-Jan-95 at 08:39:50
Method title..... SW846-8330

SAMPLE INFORMATION

Sample name..... EXPLOSIVE L28 STANDARD
Sample ID..... 10-305-9: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..... Initialize

Dilution factor summary:
PERCENT MOISTURE..... 0.000

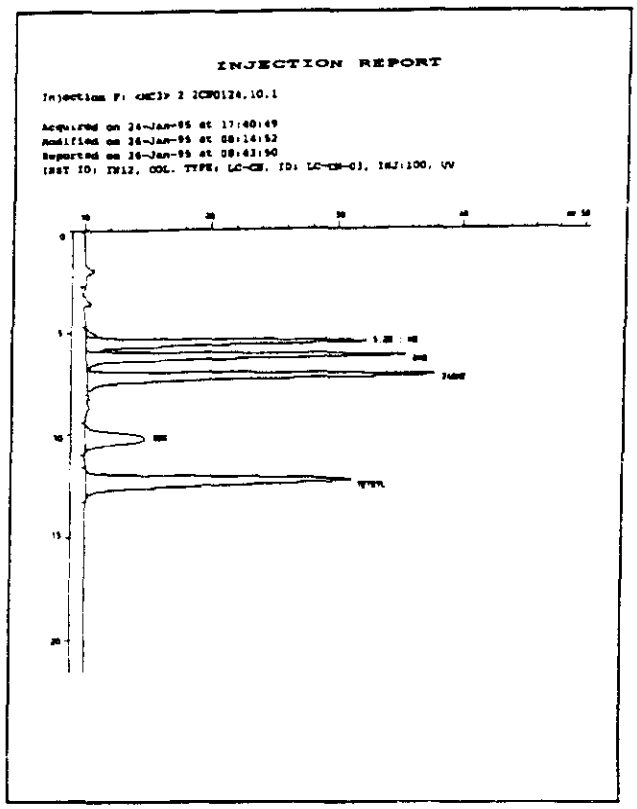
User factors:
Volume (mL)..... 1.000
Dilution..... 1.000
Amount (g or mL)..... 1.000

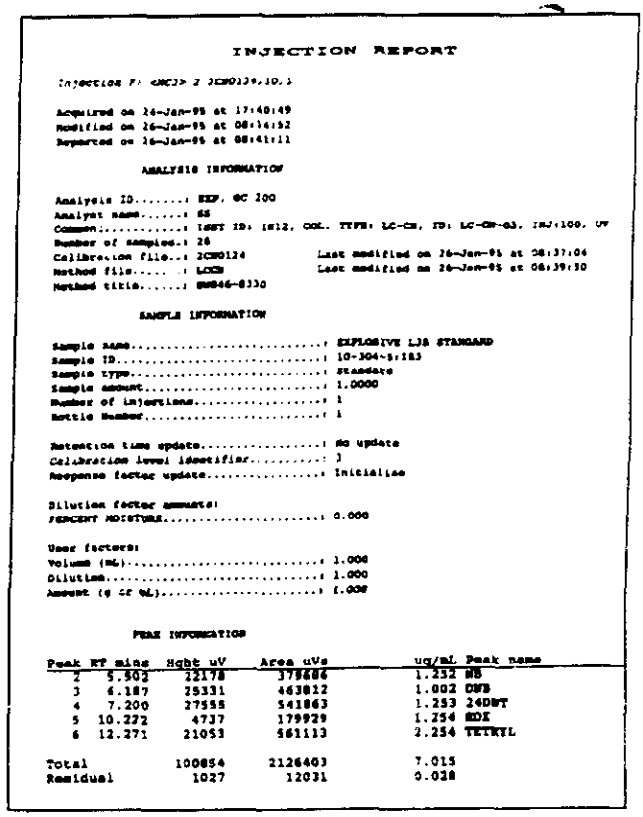
PEAK INFORMATION

Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
2	5.520	4453	72175	0.238	NB
3	6.204	5059	87679	0.189	DNB
4	7.222	5511	103207	0.236	24DNT
5	10.182	935	33319	0.232	RDX
6	12.262	4319	107462	0.432	TETRYL
Total		20276	402835	1.328	
Residual		110	881	2.337E-003	

APPENDIX B

(continued)





INJECTION REPORT

Injection F: <HC3> 2 2CH0124.10.1

Acquired on 24-Jan-95 at 17:40:49
Modified on 26-Jan-95 at 08:14:52
Reported on 26-Jan-95 at 08:41:11

ANALYSIS INFORMATION

Analysis ID.....: EXP. GC 200
Analyst name.....: SS
Comment.....: INST ID: IN12, COL. TYPE: LC-CH, ID: LC-CH-03, INJ:100, UV
Number of samples.: 26
Calibration file..: 2CH0124 — Last modified on 26-Jan-95 at 08:37:04
Method file.....: LCCH — Last modified on 26-Jan-95 at 08:39:50
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: EXPLOSIVE LJS STANDARD
Sample ID.....: 10-304-5:183
Sample type.....: standard
Sample amount.....: 1.0000
Number of injections.....: 1
Bottle Number.....: 1

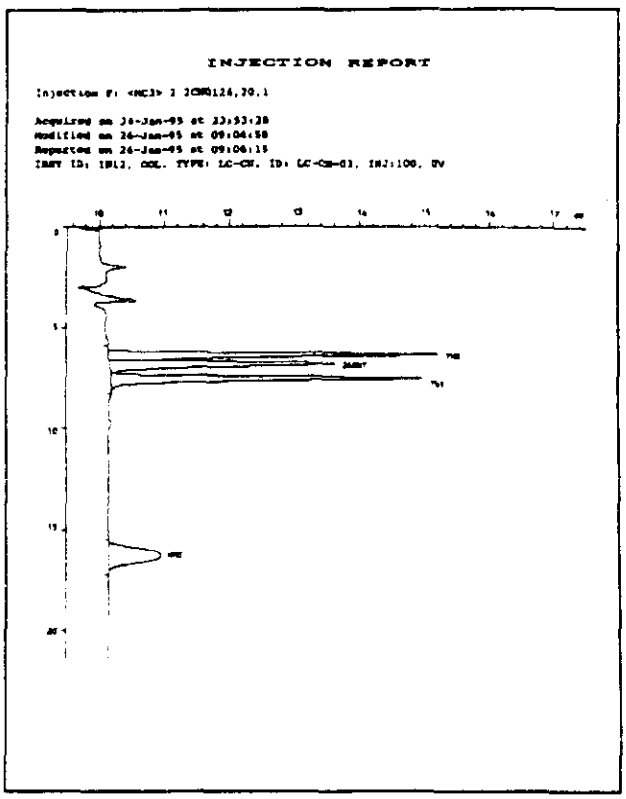
Retention time update.....: No update
Calibration level identifier.....: 3
Response factor update.....: Initialize

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
2	5.502	22178	379686	1.232	NB
3	6.187	25331	463812	1.002	DNB
4	7.200	27555	541863	1.253	24DNT
5	10.272	4737	179929	1.254	HMX
6	12.271	21053	561113	2.254	TETRYL
Total		100854	2126403	7.015	
Residual		1027	12031	0.028	



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SOUTHWEST LABORATORY OF OKLAHOMA, INC.

STANDARD OPERATING PROCEDURE FOR THE ANALYSIS OF METALS BY SW846 METHOD 6010A INDUCTIVELY COUPLED ARGON PLASMA (ICP)

REV 1.0 — 2/22/96

Document Control# SWL-MT913B-_____

APPROVALS BY:

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Signature: _____

Name: *Chuck Hoover* _____ *Title:* *QA/QC Officer* _____

Signature: _____

F-96

Table of Contents

INDUCTIVELY COUPLED ARGON PLASMA FOR TJA 61 AND TJA61E TRACE 1

I. INTRODUCTION 1

II. APPARATUS 1

III. INSTRUMENT OPERATING PROCEDURE 1

IV. REAGENTS AND STANDARDS 2

V. SAMPLE PREPARATION 3

VI. ANALYSIS 3

VII. INTERFERENCES 3

VIII. CALCULATION .....

IX. QUALITY CONTROL 4

Table 1. Elements Analyzed by ICAP61 And ICAP 61E Trace and Their Corresponding Wavelengths By Instrument 8

Table 2. High Stand (S, Std1) Curently Used by ICAP61 and ICAP61E Trace and Their Concentrations 9

Table 3 10

Table 4 11

INDUCTIVELY COUPLED ARGON PLASMA FOR TJA 61 AND TJA61E TRACE

I. INTRODUCTION

A. This method is used for the determination of dissolved, suspended or total metals in a variety of matrices by SW846 Method 6010A. Table I lists elements for which this method applies with current wavelengths used on each of the ICP units. Elements and wavelengths will be added as new methods are developed or new instruments are acquired.

B. This method describes the technique for the simultaneous multi-element determination of trace metals in solution using a Thermal-Jarrel Ash ICAP-61 and ICAP61E Trace. The basis of this 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 generated inductively coupled plasma. The spectra are dispersed by a grating spectrometer and the intensities of the lines are monitored by photomultiplier tubes. The photo currents from the photomultiplier tubes are processed and controlled by the Thermo-Spec Software via the computer system.

II. APPARATUS

A. Thermal-Jarrel Ash ICAP-61 and ICAP-61E Trace optical emission spectrophotometer.

B. IBM PC/AT compatible computer with ThermoSPEC operating software. Automated background correction and interelement corrections.

C. Radio frequency power supply with 2500 watts capacity.

D. Liquid argon supply, 4300 cu.ft.

E. Thermo Jarrell Ash Autosampler with 300 sample positions and 19 QA/QC position.

III. INSTRUMENT OPERATING PROCEDURE

A. Start-Up

1. Check that the argon tank main valve is open and the pressure is set to at least 60 psi.

2. Check that the drain tube is inserted into a plastic water bottle containing at least 8 inches of water.

F-88

3. Replace old pump tubing if necessary.

8. Continue as per manufacturers startup procedure for that instrument.

9. Turn on the computer with ThermoSpec software.

10. Replace old pump tubing if necessary.

B. Shutdown

1. Shutdown instrument as per manufacturers procedure for that instrument.

IV. REAGENTS AND STANDARDS

A. Nitric Acid, conc.: High purity such as Baker Instra-analyzed for trace metal Analysis or Mallincrodt AR select.

B. Standard Stock Solutions

1. Spex Industries, Inc., High Purity Standards, Inc. Inorganic Ventures, Inc. or other traceable source- prepared from 99.995% - 99.999% pure elements assayed by wet chemical metho dissolved in 18 mega-ohm, double deionized water and electronic grade acids. Date soluti are received and opened are posted on each bottle. All standards should be traceable to national standards such as NIST.

C. Blank Solutions

1. Calibration blanks (S0, STD0, ICB and CCB)- dilute 5.0 mL of HNO_3 (conc.) to 100 mL with ASTM Type II water.

2. Preparation Blank (PBW and PBS)- contains all the reagents and in the same volumes as used in the processing of the samples. The preparation blank is carried through the complete digestion procedures and contains the same acid concentration in the final solution as the sample solution.

D. Calibration Solutions

1. Calibration Solution (S, STD1....STDn)- Are prepared in a 5% HNO_3 solution at concentrations recomended by the instrumentation manufacturer or as specified by the client contract and brought to volume with ASTM Type II water.

E. Calibration Check Standards (ICV, ICSA, ICSAB, CRI and CCV):

1. Initial Calibration Verification standard (ICV)-Prepared from a set of standard solutions other than those used for the calibration solutions and preferably from a different vendor.

2. Intercheck Standards A and AB (ICSA, ICSAB)-The intercheck solutions are prepared at the concentrations listed in Table 3.

F-89

3. Contract Requied Analysis (CRI)-The CRI solution is prepared at the PQL.

4. Continuing Calibration Verification solution (CCV)- The CCV is prepared from the same solutions used in the preparation of the calibration standard(s) but at one half the concentration of the highest standard. This is also used as STD2 for clients requiring multi-standard calibration.

5. STD3- The STD3 calibrating solution is prepared from the same solutions used in the preparation of the calibration standard(s) but at one quarter the concentration of the highest standard.

V. SAMPLE PREPARATION

A. Separate operating procedures are written for the digestion of samples to be analyzed by the ICP depending on the matrix of the sample. (See SOP #SWL-MT600B.)

VI. ANALYSIS

A. Set up the instrument with proper operating parameters already established. The instrument must be allowed to become thermally stable before beginning. This usually requires at least 30 minutes of operation prior to calibration.

B. Initiate appropriate method file configuration on computer.

C. Setup the analytical sequence on the ICP autosampler table, following the sequence on Table 4.

D. Load samples into the autosampler according to the autosample table.

E. Start the analysis of the analytical sequence.

VII. INTERFERENCES

A. Spectral Interferences

1. Overlap of a spectral line from another element compensated by interelement correction by instrument.

2. Unresolved overlap of molecular band spectra may require selection of an alternate wavelength.

3. Background contribution from continuous or recombination phenomena.

4. Background contribution from stray light from the line emission of high concentration elements.

Note: Number 3 and 4 effects can usually be compensated by a background correction adjacent to the analyte line.

B. Physical Interferences - affects associated with the sample nebulization and transport processes. Dilution may be necessary.

VIII. CALCULATION

A. If dilutions were performed, the appropriate factor must be applied to sample values.

B. Data will be reported in ug/L off the raw data.

Soils: $$\frac{C \times V}{W} = mg/kg$$

Where:

C = *Concentration in ug/l*

V = Volume of digestate in Liters

W = Weight of sample in grams

Report significant figures as follows:

Result <10 to 2 significant figures

Result >10 to 3 significant figures

Report all results to the MDL. Results between the MDL and PQL will be flagged with a "B"

IX. QUALITY CONTROL

A. Calibration is required every 24 hours.

B. High Standard

1. Immediately after the standardization for the run, for non-CLP cases, a solution or solutions containing the highest concentration of analytes used will be run prior to initiation of the run. If the measurements exceed the control limits of 95 - 105% of the true value, the analysis will not be valid for those analytes.

C. Initial Calibration Verification(ICV)

1. Immediately after the ICP has been calibrated, the accuracy of the initial calibration is verified and documented for every analyte by the analysis of Initial Calibration Verification Solution at each wavelength used for analysis. If the measurements exceed the control limits of 90 - 110% of the true value, the analysis must be terminated, the problem corrected, the instru recalibrated, and the calibration reverified for those analytes.

F-91

D. Continuing Calibration Verification (CCV)

1. To ensure calibration verification during the analysis run, a continuing calibration verification standard is analyzed for every analyte at each wavelength at a frequency of at least every 10 samples or every 2 hours during an analysis run, whichever is more frequent. The standard is also analyzed for every wavelength used for analysis at the beginning of the run and after the last analytical sample. The same continuing calibration standard must be used throughout the analysis runs for a case of samples received. If the measurements exceed the control limits of 90-110% of the true value, the instrument must be recalibrated and all of the samples since the last successful CRI, ICSA, ICSAB, CCV, CCB set analyzed must be reanalyzed.

E. Initial and Continuing Calibration Blank (ICB and CCB)

1. A calibration blank is analyzed at each wavelength used for analysis immediately after each every initial and continuing calibration verification, at a frequency of 10% or every 2 hours during the run, whichever is more frequent. If the absolute value of the blank exceeds the PQL, terminate analysis, correct the problem, recalibrate and reanalyze all analytical samples for those analytes.

F. Interference Check Sample (ICS)

1. To verify interelement and background correction factors an ICP interference check sample is analyzed at the beginning and end of each analysis run or a minimum of twice per 8 hour working shift, whichever is more frequent. The ICS is obtained from EPA and consists of two solutions. Solution A consists of the interferants and solution AB consists of the analytes mixed with the interferants. An ICS analysis consists of analyzing both solutions consecutively for all wavelengths used. Results for the analyses of solution AB must fall within the control limits of ± 20% of the true value for the analytes. (For the ICSA use a control limit of ±2 x PQL for any element with a PQL of ≤10. If not, terminate the analysis, correct the problem, recalibrate and reanalyze the analytical samples analyzed since the last good ICS.

G. Contract Required Analysis standard (CRI)

1. To verify linearity near the lower end of the curve an ICP standard at the PQL is analyzed at the beginning and end of each sample analysis, or a minimum of every 8 hours, whichever is more frequent. Control limits are 50-150 % recovery.

H. Preparation Blank (PBW or PBS)

1. At least one preparation blank must be prepared and analyzed with every Sample Delivery Group (SDG), or with each batch of samples digested, whichever is more frequent. This blank is used to ascertain whether sample concentrations reflect contamination in the following manner:

   a. If the absolute value of the blank is less than the PQL no corrective action is necessary.

   b. If any analyte concentration in the blank is above the PQL the lowest concentration of that analyte in the associated samples must be 10x the blank concentration. If not, all samples

- less than 10x the blank concentration and above the PQL must be redigested and reanalyzed for that analyte.

c. If the concentration of the blank is below the -PQL, all samples associated with the blank must be redigested and reanalyzed if they are not greater than 10x the absolute value of the preparation blank.

I. *Laboratory Control Sample (LCS)*

1. Aqueous and solid LCS are analyzed for each analyte using the same sample preparations, analytical methods and QA/QC procedures employed for the samples. One LCS is prepared and analyzed for every group of similar matrix samples in a batch of samples digested. If the percent recovery for the LCS falls outside the control limits of 80-120% the analyses must be terminated, the problem corrected, and the samples associated with that LCS redigested and reanalyzed.

J. *ICP Serial Dilution*

1. The ICP Serial Dilution analysis is performed on each batch of samples of a similar matrix type and concentration. This sample should be flagged with an L.

2. Samples identified as field blanks cannot be used for serial dilution analysis.

3. If the analyte concentration is at least a factor of 50 above the instrument detection limit in the original sample, an analysis of a 5 fold dilution must agree within 10% of the original determination.

K. Matrix Spike/Matrix Spike Duplicate Sample

1. The spike solution is added before digestion to provide information about the effect of the sample matrix on the digestion and measurement methodology.

2. Samples identified as field blanks cannot be used for spiked sample analysis.

3. If the spike recovery is not within the limits of 75-125%, the data of all samples received associated with that spike sample and determined by the same analytical method will be flagged with the letter "N". A Post Digestion Spike will be done for that element using the sample that was originally associated with the sample for the matrix spike. An exception to this is when the sample concentration exceeds the spike concentration by a factor of four or more.

L. Post Digested Spike

1. When the pre-digested spike recovery falls outside the control limits and the sample result does not exceed 4x the spike added, a post-digested spike is performed. The sample ID should be flagged with an A.

2. Spike the unspiked aliquot of the sample at 2x the indigenous level or 2x CRDL whichever is greater.

M. Duplicate Sample

1. A duplicate sample is analyzed only when requested by the client.

2. Samples identified as field blanks cannot be used for duplicate sample analysis.

3. A control limit of 20% for relative percent difference (RPD) is used for original and duplicate sample values greater than or equal to 5x PQL. A control limit of ±PQL is used for sample values less than 5x PQL. Samples not meeting this criteria will be flagged.

N. Method Detection Limits

1. Method Detection limits for the ICP are determined yearly. The detection limits are determined by using the following protocol:

   a. Seven low level standards, at three to five times the estimated MDL, is digested in the same manner as samples. The digested samples are then analyzed and the standard deviation is taken.

   b. The standard deviation is multiplied by 3.14. This is the new MDL.

O. Interelement Corrections

1. Interelement corrections are determined quarterly by analyzing standards at or near the linear range of the interferants and determining the effect on the analytes. Required at a minimum yearly basis.

P. Linear Range

1. A linear range check standard is analyzed quarterly for each element analyzed by ICP. The analytically determined concentration must be within ± 5% of the true value. This concentration is the upper limit of the ICP linear range beyond which results cannot be reported without dilution of the analytical sample.

F-94

TABLE 1. ELEMENTS ANALYZED BY ICAP61 AND ICAP 61E TRACE AND THEIR CORRESPONDING WAVELENGTHS BY INSTRUMENT

Element	Trace 1 Wavelength (nm)	Trace 2 Wavelength(nm)	ICAP61 Wavelength(nm)
Antimony	206.838	206.831 *	217.581
Antimony	206.839	206.832 *	n/a
Aluminum	308.22	308.2	308.215
Arsenic	189.04	189.04	193.606
Barium	493.41	493.41	493.409
Berylium	313.04	313.04	313.042
Bismuth	223.06	n/a	n/a
Boron	249.68	249.68	249.678
Cadmium	226.50	226.50	228.802
Calcium	317.93	317.93	317.933
Chromium	267.72	267.72	267.716
Cobalt	228.62	228.61	228.616
Copper	324.75	324.75	324.754
Iron	271.44	271.44	259.940
Lead	220.354	220.351 *	220.353
Lead	220.355	220.352 *	n/a
Magnesium	279.08	279.08	279.079
Manganese	257.61	257.61	257.610
Molybdenum	202.03	n/a	202.030
Nickel	231.60	231.60	231.604
Potassium	766.49	766.49	766.491
Potassium	404.72***		
Scandium	361.38	361.38	
Selenium	196.027	196.021 *	196.026
Selenium	196.028	196.022 *	n/a
Silicon	288.1[illegible]	n/a	288.158
Silver	328.07	328.07	328.068
Sodium	588.99	588.99	588.995
Sodium	330.23***		
Strontium	421.55	421.55	n/a
Thallium	190.87	190.86	190.864
Tin	189.99	n/a	189.989
Titanium	334.94	n/a	334.941
Vanadium	292.40	292.40	292.402
Yttrium	371.03	371.03 **	n/a
Zinc	213.86	213.86	213.856

* Dual channel required for these elements on trace
** Used only as an internal standard when needed
*** Alternate line (less sensitive)
n/a Not applicable for this instrument

TABLE 2. HIGH STAND (S, STD1) CURENTLY USED BY ICAP61 AND ICAP61E TRACE AND THEIR CONCENTRATIONS

Element	Trace 1 Concentration(ppb)	Trace 2 Concentration(ppb)	ICAP61 Concentration(ppb)
Antimony	1000	1000	4000
Aluninum	1000	1000	4000
Arsenic	1000	1000	4000
Barium	1000	1000	4000
Berylium	1000	1000	4000
Bismuth	1000	n/a	n/a
Boron	1000	1000	4000
Cadmium	1000	1000	4000
Calcium	10000	10000	40000
Chromium	1000	1000	4000
Cobalt	1000	1000	4000
Copper	1000	1000	4000
Iron	1000	1000	4000
Lead	1000	1000	4000
Magnesium	10000	10000	40000
Manganese	1000	1000	4000
Molybdenum	1000	1000	4000
Nickel	1000	1000	4000
Potassium	10000	10000	40000
Potassium	50000***		
Scandium	1000	1000	n/a
Selenium	1000	1000	4000
Silicon	1000	1000	4000
Silver	1000	1000	4000
Sodium	10000	10000	40000
Sodium	50000***		
Strontium	1000	1000	n/a
Thallium	1000	1000	4000
Tin	1000	n/a	4000
Titanium	1000	n/a	4000
Vanadium	1000	1000	4000
Yttrium	1000	1000	n/a
Zinc	1000	1000	4000

*** Alternate line calibration standard

F-96

TABLE3

	TJA61 ICSA µg/L	TJA61 ICSAB µg/L	TRACE ICSA µg/L	TRACE ICSAB µg/L
Aluminum	500,000	500,000	500,000	500,000
Antimony	0	500	0	600
Arsenic	0	500	0	100
Barium	0	500	0	500
Beryllium	0	500	0	500
Cadmium	0	1,000	0	1,000
Calcium	500,000	500,000	500,000	500,000
Chromium	0	500	0	500
Cobalt	0	500	0	500
Copper	0	500	0	500
Iron	200,000	200,000	200,000	200,000
Lead	0	1,000	0	50
Magnesium	500,000	500,000	500,000	500,000
Manganese	0	500	0	500
Nickel	0	1,000	0	1,000
Potassium	0	0	0	0
Selenium	0	500	0	50
Silver	0	1,000	0	200
Sodium	0	0	0	0
Thallium	0	500	0	100
Vanadium	0	500	0	500
Zinc	0	1,000	0	1,000
Boron	0	500	0	500
Molybdenum	0	500	0	500
Scandium	0	500	0	500
Silicon	0	1,000	0	1,000
Tin	0	1,000	0	1,000
Strontium	0	500	0	500
Titanium	0	500	0	500

TABLE 4

ILM03.0	
1	SO
2	S
3	ICV
4	ICB
5	ICSA
6	ICSAB
7	CRI
8	CCV
9	CCB
10	10 SMPL.
11	CCV
12	CCB
13	10 SMPL.
14	CCV
15	CCB
16	ICSA
17	ICSAB
18	CRI
19	CCV
20	CCB

NOTE: Repeat steps 10-15 until the 8 hour time limit (for the ICSA/AB and CRI) is (for the ICSA/AB and) is up.

F-98

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STANDARD OPERATING PROCEDURE FOR THE SW846 METHODS 7470A/7471A ANALYSIS OF MERCURY BY COLD VAPOR

MT310B

REV 1.0 — 4/15/96

Document Control #SWL-MT310B-_____

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F-99

Table of Contents

I. Purpose 2
II. Reagents and Standards 2
III. Procedure 4
IV. Analysis 5

F-100

I. Purpose

A. This SOP will give the SW846 methods 7470A/7471A digestion and analysis procedures for water and soil matrices. These procedures will be used for the preparation and analysis for mercury.

B. The digestion supervisor will designate the proper method to be used per batch of samples. If the method is not clearly identified on the internal custody forms, check with the Project Officer responsible for the samples before digestion is started.

II. Reagents and Standards

A. Acids used in the preparation of standards and for sample processing must conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society (ACS) and be of the Mallinckrodt ArSelect grade or the equivalent.

B. Reagents

1. Reagent water: ASTM Type II (millipore) water. All references to water in this method refer to reagent water unless otherwise specified.
2. Sulfuric acid, conc.: low for trace metals analysis grade.
3. Sulfuric acid, 0.5 N: Dilute 14.0 mL of conc. sulfuric acid to 1.0 liter.
4. Nitric acid, conc.: low for trace metals analysis grade.
5. Hydrochloric acid, conc.: low for trace metals analysis grade.
6. Stannous chloride: Add 25 grams stannous chloride to 500 mL 25% HCl. Filter solution through a Grade 4, 20-25 um retention filter paper.
7. Sodium chloride - hydroxylamine hydrochloride solution: Dissolve 12 grams of NaCl and 12 grams hydroxylamine hydrochloride in reagent water and dilute to 100 mL.
8. Potassium permanganate, 5% solution, w/v: Dissolve 5 grams of potassium permanganate in 100 mL of reagent water.
9. Potassium persulfate, 5% solution, w/v: Dissolve 5 grams of potassium persulfate in 100 mL of reagent water.

C. Standard Preparation

1. Stock solutions for mercury are prepared solutions from NIST approved standards containing 1,000 mg/l mercury.
2. An intermediate stock solution of 10.0 mg/l mercury is prepared by diluting 1.0 mL of stock solution and 1.0 mL of conc. HNO_3 to 100 mL with ASTM Type II water. This standard is prepared weekly.

3. A second intermediate solution of 0.1 mg/l mercury is prepared each day of analysis by diluting 1.0 mL of the 10.0 mg/l intermediate stock solution and 1.0 mL of conc. HNO3 to 100 mL with ASTM Type II water. Make fresh daily.

4. Calibration standards are prepared by diluting the 0.1 mg/l intermediate standard to 100 mL with ASTM Type II water according to the following schedule:

Standard Calibration ug/l	mL of 0.1 mg/l Intermediate Standard
0.0	0.0
0.2	0.2
0.5	0.5
1.0	1.0
5.0	5.0
10.0	10.0

5. Working calibration standards are prepared fresh each day of analysis.

6. Initial Calibration Verification (ICV): This standard is made from the standard obtained from a NIST certified standard that has a different source than the calibration standards. The true value of this standard should be within the calibration range of the system.

7. CRA standard: use the 0.2 ug/l calibration standard.

8. Continuing Calibration Verification (CCV): use the 5.0 ug/l calibration standard.

9. Initial and Continuing Calibration Blanks (ICB and CCB): use 0.0 ug/l calibration standard.

D. Digestion QA/QC Requirements:

1. The following QA/QC samples will be designated as XXXYYMMDDHGZ.XXX designates the type of sample (PBS, LCS, or LDS). YY is the last two digits in the current year, MMDD is the month and day, HG designates a mercury digestion. Z is the batch letter done that day (if located in batch YYMMDDHG1-Z = "A; if located in batch YYMMDDHG4-Z="D").

   a. Preparation Blanks (PBW or PBS) - prepare by using the following volumes of ASTM type water:

      PBW = 100ml

      PBS = 10ml

      Result must be between the current -PQL and PQL.

b. Laboratory Control Sample (LCW, LCS) is prepared from the following:

LCW samples: spike 99 ml of ASTM Type II water with 1.0 ml of 0.1 mg/l mercury standard (see Section II.C.3.)

LCS samples: add 1.0 ml of 0.1 mg/l mercury standard and proceed with digestion procedure.

c. Laboratory Control Sample Duplicate (LDW, LDS) is prepared from the following:

LDW samples: Same as the LCW.

LDS samples: Same as the LCS.

Note Control limits for the Laboratory Control Samples are 80-120 % recovery, unless stated otherwise by the Inorganic Manager.*

2. Matrix Spike & Matrix Spike Duplicates: This is made from digesting the sample spiked with an known amount and then comparing it with the original sample. This is designated to provide information about the effect of the sample matrix on the digestion and/or measurement methodolgy, and has a control limt of 75-125 % recovery.

Spike the water or soil sample with 1.0 ml of 0.1 mg/l mercury standard (see Sec II.C.3.)

C. Matrix Duplicate: This sample is made by digesting a second aliquot of the same sample to determine the precision of the method, and has a control limit of < 20 % RPD.

D. Frequency Requirements:

1. A PBS, LCS, and LDS are required for every batch of samples (maximum number of samples in a batch is 20 samples). The matrix duplicate, spike, and spike duplicate will be done on request per client basis.

2. Each day of sample preparation, the standards in Sec II.C.4-9 will be brought through the water digestion precedure.

E. Digestion QC failures: If the Preparation Blank or Laboration Control Sample do not meet the required control limits, the samples must be redigested.

III. Procedure

A. Aqueous Preparation

1. Transfer 100 mL of well mixed sample to a 250 mL glass bottle.

2. Add 5 mL of conc. sulfuric acid and 2.5 mL of conc. nitric acid to each bottle.

3. Add 15 mL of KMnO4 solution to each bottle and allow to stand at le. 15 minutes.
4. Add 8 mL of potassium persulfate to each bottle.
5. Cap with Septa Lid.
6. Heat for 2 hours in a water bath at 90-95°C.
7. Cool and add 6 mL of sodium chloride-hydroxylamine sulfate solution to reduce the excess permanganate.

B. Soil Preparation

1. Weigh a representative 0.6 g portion (three 0.2 g of sample in different areas) of wet sample in a 250 ml glass bottle.
2. Add 10 ml of ASTM type II water.
3. Add 5 ml of concentrated sulfuric acid and 2.5 ml of concentrated nitric acid, mixing after dilution.
4. Heat 2 minutes in a water bath at 95°C.
5. Cool and add 50 ml of ASTM type II water.
6. Add 15 ml KMnO4 and 8 ml of potassium persulfate solution.
7. Mix thoroughly and heat at 95°C for 30 minutes in the water bath.
8. Cool and add 6 ml of sodium chloride-hydroxylamine sulfate solution to reduce the excess permanganate.
9. Add 50 ml of ASTM type II water.

IV. Analysis

A. Instrumentation

1. Leeman Labs PS200 Automated Mercury Analyzer.
2. Zero-grade argon.

B. Parameters

1. Wavelength: 254.0 nm
2. ApproximateRinse Time:≈50 seconds (instrument varies rinse time according to mercury level in sample)
3. Pump Speed: ≈5mL/min.
4. Readout mode: Absorbance
5. Integration time: ≈10.0 seconds
6. Gas Flow: ≈0.3L/min.

F-104

C. Calibration

1. Instrument Calibration
   a. Set up instrument according to manufacturers recommendations.
   b. Attach the stannous chloride to the Pump Assembly.
   c. Fill water bath with 5% H2SO4 and 2.5% HNO3.
   d. Rinse system using macro "warmstart" for a minimum of 40 minutes before starting calibration.
   e. Check aperature reading. If > +.100, adjust, wait 10 minutes, and recheck.
   f. Place fresh drying tube containing Magnesium Perchlorate into system.
   g. Place calibration standards into cups, then rack.
   h. Press F2 macro. Type STANDARD, then press enter key.
2. Analysis of Calibration Data
   a. Tabulate the calibration standard concentration versus absorbance response for each calibration standard.
   b. Analyze data by linear regression. The correlation coefficient must be greater than 0.995 before proceeding with analysis.
   c. If data is acceptable, press "A" to accept calibration curve.
3. Calibration Checks
   a. Initial Calibration Verification
      1. After the system has been calibrated, the accuracy of the initial calibration shall be verified and documented by the analysis of the ICV.
      2. The measurement must be within + 20% of the true value or the analysis must be terminated, the problem corrected, the instrument recalibrated and the calibration reverified.
   b. Initial Calibration Blank
      1. Immediately after the analysis of the ICV the ICB must be analyzed.
      2. If the absolute result is greater than the current PQL, terminate analysis, correct the problem and recalibrate.
   c. CRA Standard
      1. To assure linearity of calibration at the low end of the range, the low standard which was carried through the digestion procedure, shall be run after the ICB.

2. The acceptable deviation of the CRDL is 50 - 150% recovery of the true value.

d. Continuing Calibration Verification

1. To assure calibration accuracy during each analysis run, an independently prepared standard solution carried through the digestion procedure will be analyzed at the beginning, a frequency of 10% during the run, and after the last analytical sample.

2. The mercury concentrations will be at the mid-range level of the calibration curve.

3. If the deviation of the continuing calibration verification is not within 80-120 % recovery, the instrument will be recalibrated and the proceeding samples since the last calibration verification will be reanalyzed.

e. Continuing Calibration Blank

1. A calibration blank is analyzed immediately after every CCV.

2. Blanks are to be reported down to the instrument detection limit.

3. If the blank result is greater than the current PQL, terminate analysis, correct the problem and recalibrate.

D. Instrument Analysis

1. Set up instrument parameters as discussed in Section IV.B.

2. Calibrate instrument as discussed in Section IV.C.1.

3. Add samples to the autosampler and press F8 sample to start analysis run.

4. Report mercury concentrations as follows:

a. Below 10 ug/l = two significant figures

b. Above 10 ug/l = three significant figures

5. After analysis remove drying tube, rinse with deionized water, then 1:1 HNO3, then deionized water again. Place in drying oven overnight.

6. Soak water bath in 1:1 HCl overnight.

7. Convert concentrations to ug/g by the following:

(C X V) / W = ug/kg of Hg.

F-106

Where:

C = concentration , ug/l

V = final volume in liters after sample preparation

W = weight in grams of wet sample in preparation

F-107

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SOUTHWEST LABORATORY OF OKLAHOMA, INC.

STANDARD OPERATING PROCEDURE FOR THE DETERMINATION OF TOTAL ORGANIC CARBON IN SOIL/SEDIMENT

IN045S

REV 2.1 —10/17/95

Document Control# SWL-IN045S-_____

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F-114

Table of ContentsContents

I. SCOPE AND APPLICATION 1

II. SUMMARY OF METHOD 1

III. SAMPLE HANDLING AND PRESERVATION 1

IV. INTERFERENCES 1

V. APPARATUS 1

VI. REAGENTS 2

VII. PROCEDURE 2

IX. DATA REDUCTION 4

X. DATA REPORTING REQUIREMENTS 4

XI. REFERENCES 4

I. SCOPE AND APPLICATION

A. This method describes protocols for the determination of organic carbon in soil or sediment.

B. The applicable range of this analysis is 100 to 999,900 mg/Kg organic carbon. The Southwest Laboratory of Oklahoma reporting limit is 100 mg/Kg.

C. Data is reported in mg/Kg on a dry weight basis.

II. SUMMARY OF METHOD

A. Inorganic carbon from carbonates and bicarbonates is removed by acid treatment.

B. The organic compounds are decomposed by pyrolysis in the presence of oxygen.

C. The carbon dioxide that is formed is determined by direct non-dispersive infrared detection.

D. Water content is determined on a separate portion of sediment.

III. SAMPLE HANDLING AND PRESERVATION

A. Collect sediments in glass jars with teflon or aluminum foil lined lids. Cool and maintain 4°C.

IV. INTERFERENCES

A. Volatile organics in the soil/sediment samples may be lost in the process of air drying samples as well as decarbonation resulting in a low bias.

B. Bacterial decomposition and volatilization of the organic compounds are miinimized by maintaining the sample at 4°C, analyzing within the specified holding time, and analyzing the wet sample.

V. APPARATUS

A. Drying oven maintained at 103° to 105°C.

B. Analytical Instrument: Leco Model CR-12 Carbon determinator with chlorinetrap and non-dispersive infrared detector.

C. Carrier Gas: High Purity Oxygen.

D. Integral Alphanumeric Printer.

VI. REAGENTS

A. Distilled water used in preparation of standards and for dilution of samples should be ultra pure to reduce the carbon concentration of the blank.

B. Potassium hydrogen phthalate certified A.C.S. grade: 47.05% carbon for instrument calibration.

C. Calcium carbonate certified A.C.S. grade: 12% carbon for instrument calibration check.

D. Hydrochloric acid solution, 1:1 by volume.

VII. PROCEDURE

A. Sample Preparation

1. Take 0.50 grams of well mixed sample and place in a quartz combustion boat.
2. Air dry this sample in desiccator for minimum of 24 hours.
3. Add 1:1 HCl solution dropwise until the effervescence stops.
4. Dry the samples in a drying oven at 75°C. The sample is now ready for analysis.

B. Calibration

1. Select the channel to be calibrated.
2. Add a combustion boat to the built-in analytical balance.
3. Press tare. Weigh approximately 0.2 grams of potassium hydrogen phthalate in combustion boat.
4. After the balance has stabilized press enter.
5. Press analyze and wait for the digital readout to read "Load Furnace'. Slide in calibration standard and wait for the result to print-out.
6. Repeat steps 2 through 5 a minimum of three more times.
7. Press the SYSTEM UPDATE key, then the 1 key. The message center will display: CALIBRATE SYSTEM YES/NO
8. Press the YES key. The message will display: CALIBRATE, BY STD YES/NO
9. Press the YES key. The printer will print the answer stack for the previous 10 runs.
10. The message center will display: CARBON STD - % ??? MOD BY KBD enter the percent carbon contained in the standard sample (47.05%).
11. Enter the percent carbon obtained in the standard sample (47.05%).
12. The printer will print the standard entered and the old calibration.
13. The message will display: SELECT FROM LAST xx RESULTS

F-117

14. Enter the total number of calibration runs to be included in the calibration calculation. After a number is entered, the message center will display one-at-a-time the results for the number of results entered. Answer YES or NO to each prompt as it appears. The printer will print each answer as the YES key is pressed.

15. After the operator responds to the last prompt, the printer will print the new calibration and the answer stack, recalculated according to the new calibration value.

16. Analyze the 12% calcium carbonate standard and a duplicate to verify the calibration. The percent recovery must be within ± 10% and RPD <20 or recalibration will be necessary.

17. Analyze a blank combustion boat. The result must be below 0.01% or recalibration will be necessary.

18. The 12% calcium carbonate standard and blank is analyzed after every 10 samples and must meet the above criteria or recalibration and reanalysis of the previous 10 samples will be necessary.

C. Sample Analysis

1. Press the ID CODE key. Enter the sample number to be analyzed.

2. Enter the weight used in the sample preparation and press enter.

3. Press the ANALYZE key. The PURGE flow will start immediately. The LANCE flow will start later in the analysis cycle.

4. Wait until the message center display "LOAD FURNACE" and then slide the combustion boat. Immediately after inserting the combustion boat, press the ANALYZE key again since the amount of carbon released will be insufficient to start analysis automatically.

5. When the analysis is complete, the CARBON display will indicate the results and the printer will print the value.

6. Remove the expended combustion boat from the furnace.

VIII. QUALITY ASSURANCE/QUALITY CONTROL

A. With each batch of samples prepared an empty combustion boat will be treated with 1:1 HCl and carried through the drying procedure with the samples. The result of the method blank must be below 0.01%. If all results are below the Reporting Detection Limit (RDL) or above 10x the blank value the results may be used. If not, the samples must be reprepared and analyzed.

B. Due to the nature of the analysis and the high levels of organic carbon found in soils an sediments a matrix spike is not required to establish method accuracy. Accuracy of the analysis is determined by two sources. The calcium carbonate standard used for the initial calibration verification and continuing calibration verification was discussed in Section VII. Accuracy will also be determined using a privately prepared sample of Chatanooga Shal containing 3.6% total

organic carbon. The recovery requirements for this sample will be ±15%. If the recov not within control limits, the sample will be reanalyzed once more. If the recovery is still not within control limits the instrument will be recalibrated and analysis restarted.

C. As stated in Section VII. B. a continuing calibration standard and blank will be analyzed every ten samples.

IX. DATA REDUCTION

A. The concentration printed out in % organic carbon shall be converted to mg/Kg organic carbon by the following formula:

Total organic carbon, mg/Kg:

% Total Organic Carbon (air dried) x 10,000

X. DATA REPORTING REQUIREMENTS

A. Total organic carbon results will be reported in a tabular format in mg/Kg dry weight.

B. The results of the method blank, initial and continuing calibration, and duplicate analysis assoc with each batch will also be reported in a tabular format.

XI. REFERENCES

A. LECO Corp., CR-12 Carbon System Instruction Manual, 1987

B., SW-846, Method 9060, Rev. 2, June, 1990

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SOUTHWEST LABORATORY OF OKLAHOMA, INC.

STANDARD OPERATING PROCEDURE FOR THE DETERMINATION OF TKN (TOTAL KJELDAHL NITROGEN)

IN170
REV 2.1 — 2/6/96

Document Control # SWL-IN170-_____

APPROVALS BY:

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F-129

Table of Contents

I. Scope and Application 1

II. Summary of Method 1

III. Sample Handling and Preservation 1

IV. Interference 1

V. Reference 1

VI. Reagents 1

VII. Procedure 2

VIII. QA/QC Requirements 2

IX. Calculation 3

X. Reference 3

Appendix 1

MODIFICATION TO PROCEDURE FOR: URS Consultants—Navy Clean Project # CTO-0039 4

I. SCOPE AND APPLICATION

A. This method covers the determination of total Kjeldahl nitrogen in drinking, surface and saline waters, domestic and industrial wastes. The procedure converts nitrogen components of biological origin to ammonia.

B. After distillation: the ammonia is determined by the titrimetric method which is applicable to concentrations above 1 mg N/liter.

II. SUMMARY OF METHOD

A. The sample is heated in the presence of conc. sulfuric acid, K_2SO_4 and $HgSO_4$ and evaporated until SO_3 fumes are obtained and the solution becomes colorless or pale yellow. The residue is cooled, diluted, and is treated and made alkaline with a hydroxide-thiosulfate solution. The ammonia is distilled and determined after distillation by titration.

III. SAMPLE HANDLING AND PRESERVATION

A. Samples may be preserved by addition of 2 ml of conc. H_2SO_4 per liter and stored at 4°C. Even when preserved in this manner, conversion of organic nitrogen to ammonia may occur. Preserved samples should be analyzed as soon as possible.

IV. INTERFERENCE

A. High nitrate concentrations (10X or more than the TKN level) result in low TKN values. The reaction between nitrate and ammonia can be prevented by the use of an anion exchange resin (chloride form) to remove the nitrate prior to the TKN analysis.

V. REFERENCE

A. *Digestion apparatus:* A Kjeldahl digestion operates with 250 ml Erlenmeyer flasks.

B. Hot Plate

C. 500 ml double-neck boiling flasks with side-arm inlet tubes.

D. Distillation rack.

VI. REAGENTS

A. Distilled water free of ammonia. Such water is prepared by the passage of distilled water through an ion exchange column containing a strongly acidic cation exchange resin mixed with a strongly basic anion exchange resin.

NOTE 1: All solutions must be made with ammonia-free water.

B. Mercuric sulfate solution: Dissolve 8 grams red mercuric oxide (HgO) in 50 ml of 1:4 sulfuric acid (10.0 ml conc. H_2SO_4:40 ml distilled water) or $6NH_2SO_4$.

C. Sulfuric acid-mercuric sulfate-potassium sulfate solution: Dissolve 267 grams K_2SO_4 in 1300 ml distilled water and 400 ml conc. H_2SO_4. Add 50 ml mercuric sulfate solution (see VII.B) and dilute to 2 liters with distilled water.

D. Sodium hydroxide-sodium thiosulfate solution: Dissolve 500 grams NaOH and 25 g $Na_2S_2O_3 \cdot 5H_2O$ in distilled water and dilute to 1 liter.

E. Mixed indicator: Mix 2 volumes of 0.2% methyl red in 95% ethanol with 1 volume of 0.2% methylene blue in ethanol. Prepare fresh every 30 days.

F. Boric acid solution: Dissolve 20 grams boric acid, H_3BO_3 in water, add 10 ml indicator (see VI.F), and dilute to 1 liter with distilled water.

G. Sulfuric acid, standard solution: (0.02 N) 1 ml = 0.28 mg NH_3-N. Prepare a stock solution of approximately 0.1 N acid by diluting 3 ml of conc. H_2SO_4 (sp. gr. 1.84) to 1 liter with CO_2-free distilled water. Dilute 200 ml of this solution to 1 liter with CO_2 free distilled water. Standardize the approximately 0.02 N acid so prepared against 0.0200 N Na_2CO_3 solution. This last solution is prepared by dissolving 1.0000 gram anhydrous Na_2CO_3 oven-dried at 140°C, and diluting to 1 liter with CO_2-free distilled water. By weight, 4.08 grams conc. H_2SO_4 diluted to 4000 ml = 0.02 N H_2SO_4.

H. Ammonium chloride, stock solution: 1.0 ml = 1.0 mg NH_3-N. Dissolve 3.819 grams NH_4Cl in water and make up to 1 liter in a volumetric flask with distilled water. (Final concentration of 1000 mg/L.)

VII. PROCEDURE

A. The distillation apparatus should be precleaned before use by distilling a 9:1 mixture of distilled water and sodium hydroxide-sodium thiosulfate solution (see VI.D) until the distillate is ammonia-free. This operation should be repeated each time the apparatus is out of service long enough to accumulate ammonia (usually 4 hours or more).

B. *Macro Kjeldahl system:*

1. Place 100 ml of sample (or an aliquot diluted to 100 ml) into a 250 ml Erlenmeyer flask and add 50 ml sulfuric acid-mercuric sulfate-potassium sulfate solution. Evaporate the mixture in the Kjeldahl apparatus until SO_3 fumes are given off and the solution turns colorless or pale yellow. Cool the residue and add 30 ml distilled water. Transfer to a 500 ml boiling flask and dilute to approximately 300 ml.

2. For Soils: Weigh 1.00 gram sample; add 50 ml H_2O; add reagent.

3. Connect the boiling flask to the condenser with an extension of the condenser tip below the level of the boric acid solution in the 500 ml receiving flask.

4. Make the digestate alkaline by careful addition of 50 ml of sodium hydroxide-thiosulfate solution. Add through the side-arm tube. Mix well.

5. Steam distill 100 ml to 150 ml at the rate of 6-10 ml/min., into 50 ml of 2% indicating boric acid solution (see VI.F). Begin at "8" setting. After boiling begins, set to "10".

C. Determination of ammonia in distillate by titration.

1. *Titrimetric determination:* Titrate the ammonia with 0.02 N H_2SO_4 (see VI.G), matching the endpoint against a blank containing the same volume of distilled water and H_3BO_3 solution.

VIII. QA/QC REQUIREMENTS

A. An ICV will be digested with each batch of samples. The percent recovery limits will be ± 15%.

F-132

B. A preparation blank, Laboratory Control Sample (LCS), and Laboratory Control Sample Duplicate (LCSD) will be analyzed with every batch of samples. Pipet 2.00 mls of 1000 mg/L NH_4Cl into 100 mls DI water for LCS/LCSD. The percent recovery control limits for the LCS/LCSD are ± 15% and 0-20% RPD.

C. A Matrix Spike (MS) and Matrix Spike Duplicate (MSD) will be performed if it is requested by the client. The control limits and spiking procedure are the same as the LCS/LDS.

IX. CALCULATION

A. Calculate the Total Kjeldahl Nitrogen, in mg, in the original sample as follows:

$$TKN\ (mg) = A - B)N \times F \times 1{,}000$$

where:

A = milliliters of standard 0.020 N H_2SO_4 solution used in titrating sample.

B = milliliters of standard 0.020 N H_2SO_4 solution used in titrating blank.

N = normality of sulfuric acid solution.

F = milliequivalent weight of nitrogen (14 mg).

B. To calculate the Total Kjeldahl Nitrogen in mg/l:

$$TKN\ (mg/l) = \frac{TKN\ (mg) \times 1000}{V}$$

Where:

V = milliliters of sample digested.

C. To calculate the Total lKjeldahl Mitrogen in mg/kg:

$$TKN\ (mg/kg) = \frac{TKN\ (mg) \times 100}{g}$$

Where:

g = grams of sample digested

NOTES:

If sample plus digestion mixture turns black, dilution will probably be necessary. Do not allow digestion to go to dryness or fall below 10 ml before turning pale.

Soils may need to use 0.10 gram of sample if they are oily or black.

X. REFERENCE

A. EPA 351.3

B. Standard Methods 4500-Norg

APPENDIX 1

MODIFICATION TO PROCEDURE FOR: URS CONSULTANTS—NAVY CLEAN PROJECT # CTO-0039

Perform a 1:5 soil to water extraction stirring constantly for one hour. Allow extract to settle, and filter through whatman number one filter. Proceed with method as written. Back calculate results to original soil sample weight (dry weight basis).

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SOUTHWEST LABORATORY OF OKLAHOMA, INC.

STANDARD OPERATING PROCEDURE FOR THE DETERMINATION OF TOTAL SUSPENDED SOLIDS (TSS)

IN275

REV 1.1 — 8/01/96

Document Control #SWL-IN275-_____

APPROVALS BY:

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Name: Chuck Hoover Title: QA/QC Officer

Signature: Chuck Hoover

F-229

Table of Contents

I. SCOPE AND APPLICATION 2

II. SUMMARY OF METHOD 2

III. SAMPLE HANDLING AND PRESERVATION 2

IV. INTERFERENCES 2

V. APPARATUS 2

VI. PROCEDURE 3

VII. CALCULATION 3

VIII. QUALITY CONTROL 3

IX. REFERENCES 4

I. Scope and Application

A. This method is applicable for drinking, surface, and saline waters, domestic and industrial wastes.

B. The practical range of the determination is 4 mg/l to 20,000 mg/l.

II. Summary of Method

A. A well-mixed sample is filtered through a glass fiber filter, and the residue retained on the filter is dried to constant weight at 103° C to 105° C.

B. The filtrate from this method may be used for total dissolved solids.

III. Sample Handling and Preservation

A. Non-representative particulates such an leaves, sticks, fish and lumps of fecal matter should be excluded from the sample.

B. Preservation of the sample is not practical; analysis should begin as soon as possible. Refrigeration to 4° C minimizes microbiological decompostion of the solids.

C. Holding time is 7 days from sample collection.

IV. Interferences

A. Filtration apparatus, filter material, prewashing, post-washing, and drying temperature are specified because these variables have been shown to affect the results.

B. Samples high in dissolved solids such as saline waters, brines and some waters, may be subject to a positive interference. Care must be taken in selecting the filtering apparatus so that washing of the filter and any dissolved solids in the filter minimizes this potential interference.

V. Apparatus

A. Glass fiber filters, 2.5 cm

B. Filter supports Gooch Crucible and holder

C. Vacuum flask, 1L

D. Drying Oven, 103° C to 105° C.

E. Desiccator

F. Analytical balances Mettler H80 or S/P 180

VI. Procedure

A. Preparation of glass fiber filter discs:

1. Insert the glass fiber filter into the bottom of the Gooch Crucible with wrinkled surface up, while vacuum is applied.
2. Wash the disc with three successive 20ml volumes of distilled water. Remove all traces of water by continuing to apply vacuum after water has passed through.
3. Remove crucible and filter and dry in oven at 103° C to 105° C for at least one hour.
4. Remove to desiccator and store until needed. Weigh immediately before use. After weighing, handle the crucible with forceps or tongs only.

B. Analysis:

1. Weigh the crucible and filter on an analytical scale and record weight.
2. Assemble the filtering apparatus and begin suction. Wet the filter with a small volume of distilled water to seat it against the fritted support.
3. Shake the sample vigorously and transfer 100 ml of the sample to the filter using a graduated cylinder. If the sample has alot of particulate matter the analyst should use their judgement on how small of volume to use. Remove all traces of water by continuing to apply vacuum after sample has passed through.
4. With suction on, wash the graduated cylinder and Gooch Crucible wall with thre portions of distilled water allowing complete drainage between washings. Remove all traces of water by continuing to apply vacuum after water has passed through.
5. Using tongs, remove the crucible and filter and place in a drying oven at 103° C to 105° C for at least four hours. Cool in a desiccator and weigh.

VII. Calculation

A. Calculate non-filterable residue as follows:

$$\text{Total Suspended Solids, mg/l} = \frac{(A - B) \times 1000}{C}$$

Where: A = Weight of filter and crucible + residue in mg

B = Weight of filter and crucible in mg

C = ml of sample filtered

VIII. Quality Control

A. The following QC samples will be required for each batch of samples:

1. Preparation Blank - consisting of ASTM Type II DI water must be below the reporting limit; if not reprep batch.
2. Laboratory Control Sample - consisting of a known value from a verified source, the recovery must be between 80-120% R.
3. Matrix Duplicate Sample - Select one sample and analyze a duplicate for precision.

IX. References

A. U.S. Environmental Protection Agency, Methods for the Determination of Inorganic Substances in Environmental Samples, August 1993, Method 160.2

B. Standard Methods for the Examination of Water and Wastewater, 18th Edition, APHA-AWWA-WPCF, Method 2540-D, pp. 2-56

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SOUTHWEST LABORATORY OF OKLAHOMA, INC.

STANDARD OPERATING PROCEDURE FOR THE ANALYSIS OF CHEMICAL OXYGEN DEMAND (COLORIMETRIC WITH HACH TUBES, LOW LEVEL 0-150 PPM, DETECTION LIMIT 5 PPM)

IN090D

REV 2.0 — 7/22/96

Document Control #SWL-IN090D- 005

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Signature: Chuck Hoover

F-234

Table of Contents

I. SCOPE AND APPLICATION 2

II. SUMMARY OF METHOD 2

III. SAMPLE HANDLING AND PRESERVATION 2

IV. INTERFERENCES 2

V. APPARATUS 2

VI. REAGENTS 2

VII. STANDARDS 3

VIII. PROCEDURE 3

IX. CALCULATION 4

X. QUALITY CONTROL 4

XI. PRECISION AND ACCURACY 4

XII. BIBLIOGRAPHY 4

F-235

I. Scope and application

A. This method covers the determination of COD in surface waters, domestic and industrial wastes.

B. The applicable range of this method is 5-150 ppm, samples above 150 ppm may be diluted, then run using same standard curve, or may be run using the mid-high range method; 10-1500 ppm.

II. Summary of method

A. Sample, blanks and standards in sealed Hach tubes are heated in a block digestor at 150° C for a minimum of two hours. After heating period, the tubes are removed from the digestor, cooled and measured spectrophotometrically at 600nm.

III. Sample Handling and preservation

A. Collect the samples in glass bottles if possible. Use of plastic containers is permissible if it is known that no organic contaminants are present in the containers.

B. Samples should be preserved with sulfuric acid to a pH <2 and maintained at 4° C until analysis.

C. Holding Time: 28 days from collection, if preserved. Non-preserved samples should be run as soon as possible from collection date.

IV. Interferences

A. Chlorides are quantitatively oxidized by dichromate and represent a positive interference. Mercuric sulfate has been added to the Hach tubes to complex the chlorides.

V. Apparatus

A. Block digestor, 150° C

B. Hach COD tubes 0-150 ppm.

C. 5.0ml x 1/10 Disposable Serological pipets.

E. Spectrophotometer.

VI. Reagents

A. Hach COD tubes 0-150 ppm.

B. Each tube (0-150 ppm) contains:

F-236

1. H_2SO_4 98% concentration
2. Mercuric sulfate
3. Chromic acid
4. Ag_2SO_4
5. DI water

VII. Standards

A. Stock potassium acid phthalate (KHP): Dissolve 1.700g of dried (2 hours at 120oC) KHP in 800 mls of distilled water and dilute to 1 liter. 1ml = 2mg of COD; 2000mg/l COD.

1. Prepare six standard solutions, i.e., 5, 25, 50, 100, and 125 mg/l, by diluting appropriate volumes of stock standard.
2. Analyze DI water as a reagent blank with standard curve.

VIII. Procedure

A. Turn on both block digestor and spectrophotometer, set wavelength to 600 nm.

B. Mix both unknowns and standards by inversion.

C. Add with 5.0 ml disposable serological pipet 2.0 mls of sample to each tube. Use same volume for both standards and unknowns.

D. Use a standard (ICV) with the source different than the calibration standards to verify the calibration standards. Concentration should in the range of 50-100 ppm.

E. Use 50 ppm standard as a blank spike (LCW and LDW) with each run of unknowns.

F. Use DI water for a reagent blank with each run of unknowns to set zero absorbance on spectrophotometer.

G. Cap tightly and mix tubes by inversion to blend both phases.

H. Place tubes in block digestor at 150° C ± 5° C for two hours.

I. Remove tubes from digestor, place in Nalgene rack, and allow to cool for 10 minutes. Mix each tube by inversion twice and allow any precipitate to settle and for tubes to reach room temperature, about 20 minutes.

J. Measure intensity in spectrophotometer at 600 nm, making sure that the Hach label on the tubes faces to the right in the spectrophotometer.

F-237

IX. Calculation

A. Prepare a standard curve by plotting absorbance against known concentrations of standards.

B. Compute concentration of unknowns by comparing sample response to standard curve.

X. Quality Control

A. The calibration coeffient will be > 0.995.

B. The ICV should be + 10%. If outside your control limits, remake calibration standards and repeat calibration.

C. The Reagent Blank must be below the reporting limit.

D. The LCW and LDW have control limits of 80-120 %R and 20% RPD. If control limits are exceeded - reprep all assoicated samples.

E. Standard KHP kept in the refrigerator should be good for 4 months or until discoloration appears.

F. Every new lot number of tubes must have a new standard curve.

XI. Precision and Accuracy

A. Duplicates will be done on client request.

B. Matrix spikes(MS) and Matrix spike Duplicates(MSD) will be doneon client request. For the MS and MSD spike the sample with 0.05 ml of the 2000 mg/l standard (Sec VII. A). Spike added concentration will be 50 mg/l COD.

C. Duplicates have control limits of 20% RPD. If control limits are exceeded - flag data on control charts.

D. MS & MSD have control limits of 75-125% R and 20% RPD. If control limits are exceeded - flag data on control charts.

XII. BIBLIOGRAPHY

A. US EPA: Methods for Chemical Analysis of Waters and Wastes. (410.2)

B. Hach: Water Analysis Handbook

F-238

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SOUTHWEST LABORATORY OF OKLAHOMA, INC.

STANDARD OPERATING PROCEDURE FOR THE DETERMINATION OF BIOCHEMICAL OXYGEN DEMAND (BOD) IN WATER

Document Control # - IN025-_____

IN025

REV 1.0 — 2/11/93

Clark Hoover

Robert W. Hedrick Lab Mgr.

F-244

I. SCOPE & APPLICATION

A. This method is an empirical determination of the oxygen requirement for the biochemical degradation of organic and inorganic material in water and wastewater.

B. The method is host applicable to measurement of biochemical oxygen demand above 10 mg/l.

II. SUMMARY OF METHOD

A. A series of dilutions of sample are incubated for 5 days in airtight bottles. The dissolved oxygen content of each bottle is measured prior to and after incubation. The difference in dissolved oxygen demand is calculated.

III. SAMPLE HANDLING & PREPARATION

A. Samples should be stored at 4°C until time of analysis.

B. Holding times are 48 hours from sample collection.

IV. INTERFERENCES

A. None.

V. APPARATUS

A. Orion D.O. meter

B. 300 ml BOD bottles with stopper and caps

C. Incubator

VI. REAGENTS

A. Distilled water

B. Hach Nutrient/Buffer Pillow

C. Hach Nitrification inhibitor

D. Glucose-glutamic acid solution

E. Seed water

F-245

VII. PROCEDURE

A. Preparation of Dilution Water

1. Fill a 3 liter glass jug with distilled water and add contents of one nutrient pillow, nitrification inhibitor, and 5 ml of seed, and begin to aerate jug with compressed air.

2. Check dilution water before using by incubating a bottle for 5 days. The D.O. uptake should not be more than 0.2 mg/l. If it is over 0.2 mg/l, prepare a new batch of dilution water.

B. Glucose-glutanic Acid Check

1. Take a mixture of 150 mg glucose per liter and 150 mg glutanic acid per liter.

2. Determine the BOD of a 2% dilution of glucose-glutanic acid standard check solution using same procedure as samples.

3. If the BOD value of the check is outside the range of 200 $\pm$ 37, reject any BOD determinations made with the seed and dilution water, and seek the cause of the problem.

C. Sample Preparation

1. Sample should be allowed to come to room temperature, and pH of sample adjusted to 6.5 to 7.0 with sulfuric acid or sodium hydroxide.

2. Begin to aerate sample with compressor air.

D. Set-up of Bottles

1. Arrange numbered bottles and put 1, 5, 50, and 100 mL of aerated sample (or other appropriate quantities) into the bottles.

2. Fill bottles with dilution water to completely fill bottle.

E. Initial D.O. Reading

1. Calibrate instrument per manufacturing instructions

2. Determine initial DO reading with probe for each bottle and record.

3. Seal each bottle with ground glass stopper and plastic cap.

F. Incubation

1. Place filled BOD bottles in incubator and incubate at 20°C for five days. The bottles should remain in the dark during the incubation period.

F-246

G. Post DO Reading

1. Calibrate instrument per manufacturing instructions.

2. After five days, remove bottles and determine DO for each bottle.

H. Calculations

$$BOD\ (mg/l) = (D_1 - D_2) - (B_1 - B_2)\ f/p$$

where: D_1 = initial DO of sample

D_2 = Post DO of sample

p = Decimal fractions of sample used

B_1 = Initial DO of Blank

B_2 = Post DO of Blank

f = Ratio of seed in diluted sample to seed in Blank

Notes: An acceptable dilution will yield at least 2 mg/l. Do consumption and have 9 post DO of no less than 1 mg/l in most cases.

VIII. QUALITY CONTROL

A. Analyze one duplicate sample for every 10 samples.

F-247

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AMERICAN ANALYTICAL & TECHNICAL SERVICES, INC. / SOUTHWEST LABORATORY OF OKLAHOMA, INC.

STANDARD OPERATING PROCEDURE FOR THE ANALYSIS OF OIL & GREASE

IN200, IN205

REV 3.2 — 1/05/96

Document Control# AATSWL-IN200-_____

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F-248

Table of Contents

I. SCOPE AND APPLICATION 1

II. METHOD SUMMARY 1

III. SAMPLE PRESERVATION, CONTAINERS, HANDLING & STORAGE 1

IV. INTERFERENCES AND POTENTIAL PROBLEMS 1

V. EQUIPMENT/APPARATUS 1

VI. REAGENTS AND STANDARDS 2

VII. PROCEDURE 2

VIII. CALCULATIONS 5

IX. QUALITY CONTROL 5

X. DATA VERIFICATION .....

XI. HEALTH AND SAFETY 7

XII. REFERENCES 7

I. SCOPE AND APPLICATION

A. This SOP is applicable to the analysis of solid and liquid samples for oil and grease. Concentrations from 0.5 mg/L and upward for water and 10 mg/kg and upward for solids are obtainable using the methods described herein.

II. METHOD SUMMARY

A. Samples are extracted with trichlorotrifluoroethane (freon) or n-hexane (gravimetric only) to remove petroleum hydrocarbons. Water samples must be extracted and analyzed within 28 days and soils must be extracted within 14 days and analyzed within 40 days of sample collection. Oil and grease in the extract is then determined by infrared analysis according to EPA Method 413.2 or gravimetrically, after driving off the solvent, according to EPA Method 413.1.

III. SAMPLE PRESERVATION, CONTAINERS, HANDLING & STORAGE

A. Soil samples should be collected and stored in airtight glass containers with teflon lined lids. A minimum of 100 grams of sample should be collected. Samples should be maintained at 39°F ± 4° (4°C ± 2°) during shipment and storage prior to analysis. Aqueous samples should be collected in airtight glass containers with teflon lined lids. A minimum of 1 L of sample should be collected. Aqueous samples should be preserved with sulfuric acid to <2 pH. Samples should be maintained at 39°F ± 4° (4°C ± 2°) during shipment and storage prior to analysis.

IV. INTERFERENCES AND POTENTIAL PROBLEMS

A. Solvents, reagents, and processing hardware may yield artifacts leading to misinterpretation of data. The system must be demonstrated to be free from interferences under the conditions of the analysis by running method blanks. Specific selection of reagents and purification of solvents may be required.

V. EQUIPMENT/APPARATUS

A. *Pipets* — 1 mL, 5 mL and 10 mL

B. *Volumetric Flasks* — 100 mL, 1000 mL

C. *Round-bottomed (Florence) flasks* — 500 mL

D. *Three-ball Snyder columns*

E. *Separatory Funnels* — 2000 mL glass with Teflon stopcock

F - 250

F. *Beakers* — 400 mL

G. *Filter Funnels* — Glass with six-inch tip

H. *Filter Paper* — S&S #595 or equivalent

I. *Graduated Cylinder* — 1000 ml

J. *Horn Sonicator* — Tekmar Sonic Disrupter with 3/4 inch horn or equivalent

K. *Separatory Funnel Shaker* — Glas-Col, or equivalent

L. *Spectrophotometer* — Perkin Elmer 1600 FTIR or equivalent

M. *Scintillation Vials* — 20 mL

VI. REAGENTS AND STANDARDS

A. *Anhydrous Sodium Sulfate* — Mallinckrodt #8024KJXE, or equivalent

B. *Freon* — EM TX1165-1, or equivalent

C. *n-Hexane* — Mallinckrodt ChromAR, or equivalent

D. *Iso-octane* — Baker #9335-3, or equivalent

E. *n-Hexadecane* — Fluka AG #CH9470, or equivalent

F. *Chlorobenzene* — Aldrich #28,451-3, or equivalent

VII. PROCEDURE

A. *SAMPLE PREPARATION*

1. *Water Samples — Separatory Funnel Technique*

   a. Sample pH is checked with pH paper. Those samples with pH >2.0 are acidified to <2.0 with sulfuric acid.

   b. The TDS is checked when the sample volume is determined by weighing. This ensures that the accuracy of the weight determination/volume is not effected by the TDS in the samples. Samples with TDS >10,000mg/L should be measured volumetrically.

   c. Weigh to the nearest gram the sample and container. Decant the entire sample into a 2 liter separatory funnel. Add 30 ml solvent to the sample bottle and shake vigorously to remove hydrocarbons remaining on the container. Vent the container frequently to prevent pressure buildup. Add this solvent to the separatory funnel. Weigh the en...

F-251

container and record the difference between the full and empty weights and identify as sample volume. (Alternatively, sample volume may be determined by direct measurement. Mark the water level on the side of the sample container. Decant the entire sample into a 2 liter separatory funnel. Using a one liter graduated cylinder, decant tap water into the sample container to the mark. Measure the amount poured from the graduate and record as the sample volume.)

d. Perform the extraction based on SW846 Method 3510. Vigorously shake the separatory funnel for two minutes to thoroughly mix the two phases, venting as necessary to prevent pressure buildup. Allow the phases to separate (at least ten minutes). Filter the solvent phase through enough sodium sulfate to resolve any emulsion and remove entrained water. Collect in a 120 mL glass container for Infrared (IR) analysis or a round-bottomed flask for gravimetric analysis. Repeat the extraction twice more using 30 additional ml solvent each time.

e. After filtering the last of the extract through the sodium sulfate, rinse the separatory funnel tip and the sodium sulfate twice to remove traces of hydrocarbon. Proceed to the desired analysis step B.1. or B.2.

2. *Soil/Sludge Samples — Sonication Technique*

a. Weigh 15-30 grams of sample into a clean 400 ml beaker. Stir in anhydrous sodium sulfate and mix until the mixture is of uniform consistency and free-flowing.

b. Add sufficient solvent to completely cover the sample and allow immersion of the sonicator probe tip one-fourth inch into the liquid.

c. Perform the extraction according to SW846 Method 3550. Immerse the probe tip into the liquid (1/8 to 1/4 inch). Sonicate three minutes at 50-80% duty cycle and 7-9 output control.

d. Decant the extract through sodium sulfate and collect in the glass extract container or round-bottomed flask. Repeat the extraction steps twice more. Proceed to the desired analysis step.

B. Standardization/Instrument Calibration

1. *Infrared Analysis*

a. *Reference Oil Concentrate Preparation*

(1) Prepare reference oil concentrate by combining 15 mL n-hexadecane, 15 mL isooctane and 10 mL chlorobenzene in a 40 mL VOA vial with teflon liner. Keep tightly sealed and refrigerated when not in use.

b. *Stock Standard Preparation*

(1) Prepare the stock standard by pipetting 1 g of reference oil into a 100 ml volumetric flask and dilute to volume with freon. Prepare monthly or sooner as needed. Record this and all subsequent standards in a standards log book, uniquely identifying each standard. Record this unique identity in the bench notebook each time the standard is used.

c. *Initial Calibration*

(1) From the stock standard, prepare working standards at five concentrations (e.g. 5, 10, 20, 30 & 40 mg/100mL) by diluting with freon.

(2) Prepare a calibration curve by plotting absorbance (at 2930 cm^{-1}) vs concentration

F-252

for the five working standards. The acceptance limit is a minimum correl. coefficient of 0.99. If the acceptance limit is not met, prepare new standards and perform initial calibration again. All standards should be replaced and new initial calibration performed at a minimum of every six months.

d. *Continuing Calibration*

(1) At the beginning of each day, adjust the absorbance to zero with fresh freon. Read and record the absorbance for a mid level standard (e.g. 20 mg/100 mL) daily. If the absorbance is within ±15% of the original value as determined from the initial calibration the system is in calibration and sample analysis can proceed. Otherwise, prepare a fresh mid level standard. If the absorbance of this fresh standard is also out of limits, prepare new working standards and perform initial calibration again.

2. *Gravimetric Analysis*

a. *Primary Spiking Standard Preparation*

(1) Prepare primary spiking standard by weighing 1.0 g #2 fuel oil and 1.0 g corn oil into a 100 mL volumetric flask. Dilute to volume with the solvent. Prepare the working spiking standard by measuring 25 mL primary standard into a 1000 mL volumetric flask and diluting to volume. Replace primary and working standards at a minimum of every 12 months.

C. *SAMPLE ANALYSIS*

1. IR Analysis

a. For IR analysis, bring the extract volume to the 100 ml mark on the extract container. Measure the absorbance at 2930 cm^{-1} and record.

b. Oil and Grease is calculated as described in Section VIII.

2. Gravimetric Analysis

a. For gravimetric analysis, place two boiling chips in the flask and place a three-ball Snyder column atop the flask. Place the flask in a water bath at 70°C and allow the solvent to boil and reflux until the volume of solvent remaining in the flask is about the diameter of a half dollar. Remove the flask from the bath and allow to cool. Decant the remaining extract into a clean, previously weighed scintillation vial. Rinse the flask three times with 1-2 mL each time and decant each rinse into the scintillation vial. Place the open scintillation vial under the nitrogen blow-down apparatus until all solvent is evaporated. Cool the vial in a desiccator one hour and reweigh to a constant weight as determined by two consecutive weighings.

F-253

VIII. CALCULATIONS

A. Oil and Grease by IR is calculated as follows:

$$C = (A_{smp}/A_{std})(C_{std})(100/V)$$

Where:

C = concentration (mg/l or mg/kg)

A_{smp} = absorbance of sample extract

A_{std} = absorbance of standard

C_{std} = concentration of standard

V = sample volume (L) or weight (g)

B. Oil and Grease, Gravimetric is calculated as follows:

$$C = [(W_r - W_t) * 1000]/V$$

Where:

C = concentration (mg/l or mg/kg)

W_r = weight of vial + residue (g)

W_t = tare weight of empty vial

V = sample volume (L) or weight (Kg)

IX. QUALITY CONTROL

A. *Instrument Baseline*

1. For IR analysis the absorbance must be brought to zero before analyses are performed. The baseline is established using pure freon and is checked after each ten sample readings, or fewer if instrument drift is suspected.

B. *Blanks*

1. Prepare one blank per batch of up to 20 samples. Water blanks are prepared using 1000 mL deionized tap water. Soil blanks are prepared using 30 g sodium sulfate. Blanks are taken through the entire extraction process.

C. *Matrix Spikes/Duplicates*

1. Prepare a spike/spike duplicate pair for each batch of 20 or fewer soil samples. For IR analysis, spikes are prepared by adding 2 mL of the stock standard to the already mixed soil/sodium sulfate prior to sonication. For gravimetric analysis, spikes are prepared by adding 20 mL of the working spiking standard to the already mixed soil/sodium sulfate prior to sonication. Analyst discretion is used to determine which soil sample to spike.

2. When sufficient sample volume is provided (i.e. three separate one liter containers), matrix

F-254

spikes/duplicates for waters are prepared by adding 2 mL of the stock standard (IR ana.) or 20 mL of the working standard (gravimetric analysis) to the separatory funnel containing the sample to be spiked.

D. *Laboratory Control Spikes*

1. For soils, provide one laboratory control spike per day by adding 2 mL of the stock standard (IR analysis) or 20 mL of the working standard (gravimetric analysis) to 30 g of sodium sulfate and carry through the extraction procedure.

2. For waters, provide a spike/duplicate pair per batch of 20 or fewer samples by adding 2 mL of stock standard (IR analysis) or 20 mL of the working standard (gravimetric analysis) to one liter of deionized tap water and carry through the extraction process.

E. Control Limits

1. If the soil blank contains >10 mg/kg oil and grease the system is out of control. Extract a new blank and reextract all samples associated with that blank.

2. If a water blank contains >0.5 mg/l oil and grease, so note on the report to the client.

3. The acceptance range for soil spike recoveries is 50-120%. If a spike is out of limits, reextract. If the reextract is also out of limits the system is out of control and the batch of samples associated with that spike must be reextracted with a new spike/duplicate pair. Find and correct the problem before proceeding.

4. The acceptance limit for relative percent difference (RPD) for soil spike/duplicates is If RPD >20%, reextract the spike/duplicate pair, taking care to homogenize the sample. If the RPD is still >20%, evaluate the laboratory control spike (LCS). If the LCS is within control limits the system is in control and a matrix effect has resulted in the out-of-limit spike/spike duplicate RPD. Submit the LCS result with the report and no further action is necessary. If the LCS is out of acceptance limits the system is out of control and the batch of samples associated with that spike/duplicate pair must be reextracted. Ascertain and correct the problem prior to reextracting.

5. The acceptance range for water control spike recoveries is 50-115%. If a spike is out of limits, extract a new spike. If the new extract is out of limits the system is out of control and results are so noted to the client. Find and correct the problem before proceeding.

6. RPD of water control spikes should be <20%. Otherwise, extract a new spike or spike duplicate or both. If the RPD is still >20% the system is out of control and the problem must be ascertained and corrected before more samples are extracted and analyzed.

X. DATA VERIFICATION

A. The analyst checks all calculations and initials the appropriate page in the bench notebook. The supervisor periodically reviews the bench notebook for accuracy and completeness and initials pages reviewed. All reports are reviewed by the supervisor before issuance of the final report.

F-255

XI. HEALTH AND SAFETY

A. Good laboratory practices dictate the careful handling of all laboratory samples and reagents.

XII. REFERENCES

A. EPA Methods 413.1 and 413.2

B. SW-846, 9070

F-256

APPENDIX G

EXAMPLE DATA DELIVERABLE PACKAGE (EXPLOSIVES)

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	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

I= 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

# of MS analyte recoveries out of QC Limits = 0

COMPOUND	SPIKE ADDED (ug/Kg)	MSD CONCENTRATION (ug/Kg)	MSD % REC #	% RPD#	QC LIMITS RPD	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-[illegible]LSA 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	35[illegible]	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 Case No: SDG:
Instrument ID:IN13 GC Column ID: ODS-10

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

-PLOSIVE CALIBRATION DATA SW846-8330

strument 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	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
.T	2641	522	63850	505	139359	549	198749	523	243958	482
24DNT	3041	987	76048	988	165673	1076	236530	1020	289611	658
2NT	2137	486	51452	468	113102	514	164272	498	200211	455
4NT	1861	423	46710	425	102853	468	149318	452	182225	414
3NT	2200	526	54973	527	120305	573	174104	554	212118	507

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
	735	6.15	0.99605	709	0.000
	792	4.91	0.99633	779	0.000
	516	4.76	0.99667	505	0.000
26DNT	946	17.42	0.94747	810	0.000
24DNT	484	4.85	0.99652	477	0.000
2NT	436	5.18	0.99658	434	0.000
4NT	538	4.82	0.99662	532	0.000
3NT					

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 : 1EX0711,22
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

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

CONTINUING CALIBRATION DATA
Form 9C

In'tial Calibration Date: 07-11-96
inuing Calibration #: 5
inuing Cal Date : 07-12-96
tinuing 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: 07-11-96
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: 7-11-96
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

EXPLOSIVE CALIBRATION DATA - SW846-8330

Injection 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 DATA - SW846-8330

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 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
	3.54	3.55	3.54	3.54	3.54	3.54	3.52	3.51
X	4.83	4.84	4.82	4.82	4.83	4.83	4.80	4.79
TNB	5.95	5.95	5.93	5.93	5.94	5.94	5.92	5.90
TETRYL	6.71	6.69	6.67	6.66	6.68	6.68	6.65	6.63
DNB	7.34	7.32	7.30	7.28	7.31	7.31	7.28	7.26
TNT	8.10	8.08	8.05	8.04	8.06	8.06	8.04	8.01
NB	8.42	8.40	8.36	8.35	8.38	8.37	8.35	8.32
26DNT	9.50	9.46	9.42	9.40	9.44	9.44	9.41	9.38
24DNT	10.10	10.06	10.01	10.00	10.04	10.04	10.00	9.97
2NT	11.77	11.72	11.65	11.64	11.69	11.68	11.65	11.62
4NT	12.53	12.46	12.40	12.38	12.44	12.44	12.40	12.36
3NT	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

EPA 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

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: 07-1[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

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

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

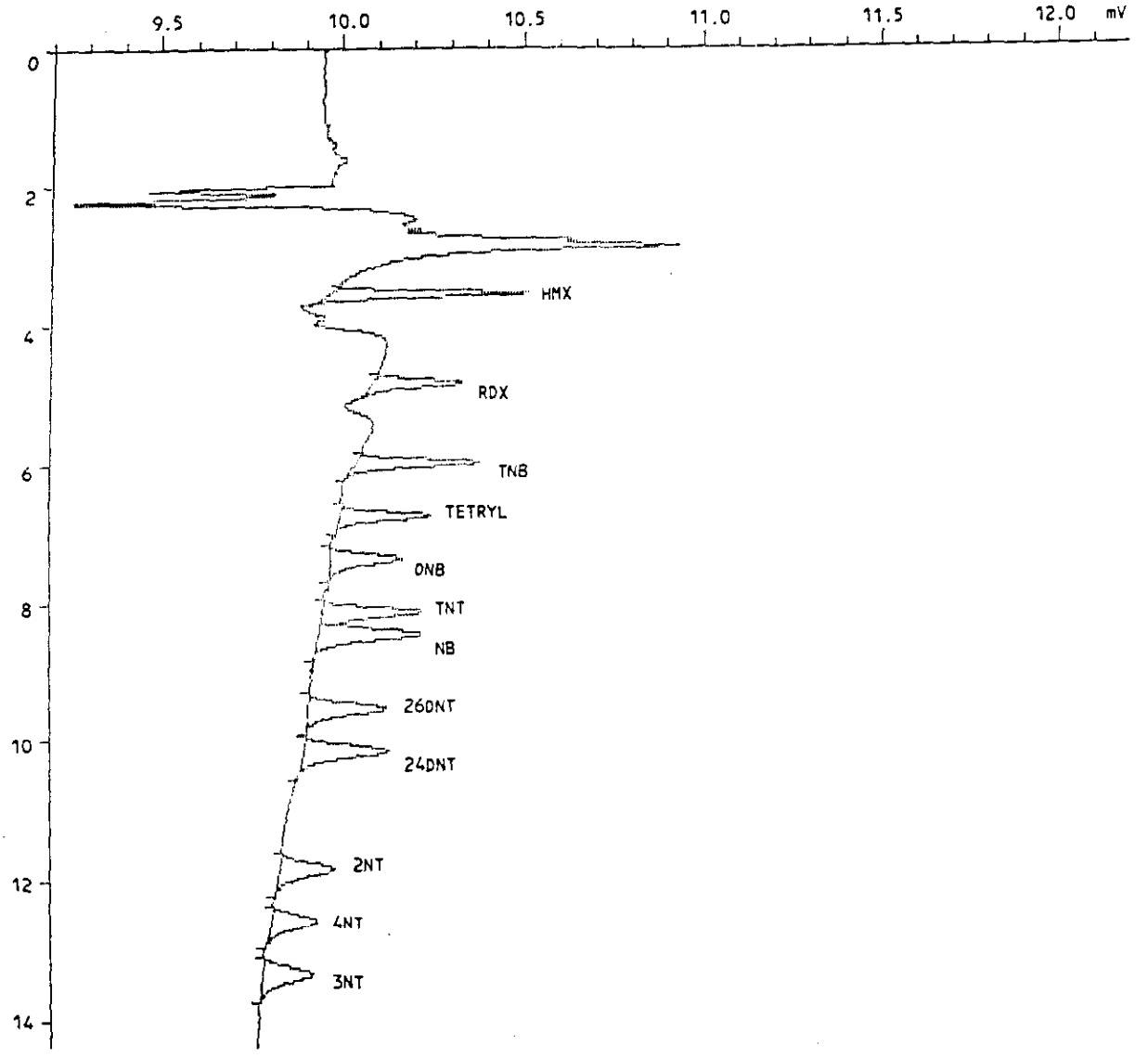
COMPONENT NAME	RT Cont CA1	RT Cont CA2	RT Cont CA3	RT Cont CA4	RT Cont CA5	RT Cont CA6	RT Cont CA7	RT Cont CA8
4ADNT	7.88	7.86	7.81	7.80	7.83	7.84	7.82	7.79
2ADNT	8.26	8.24	8.20	8.18	8.21	8.22	8.20	8.17

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



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

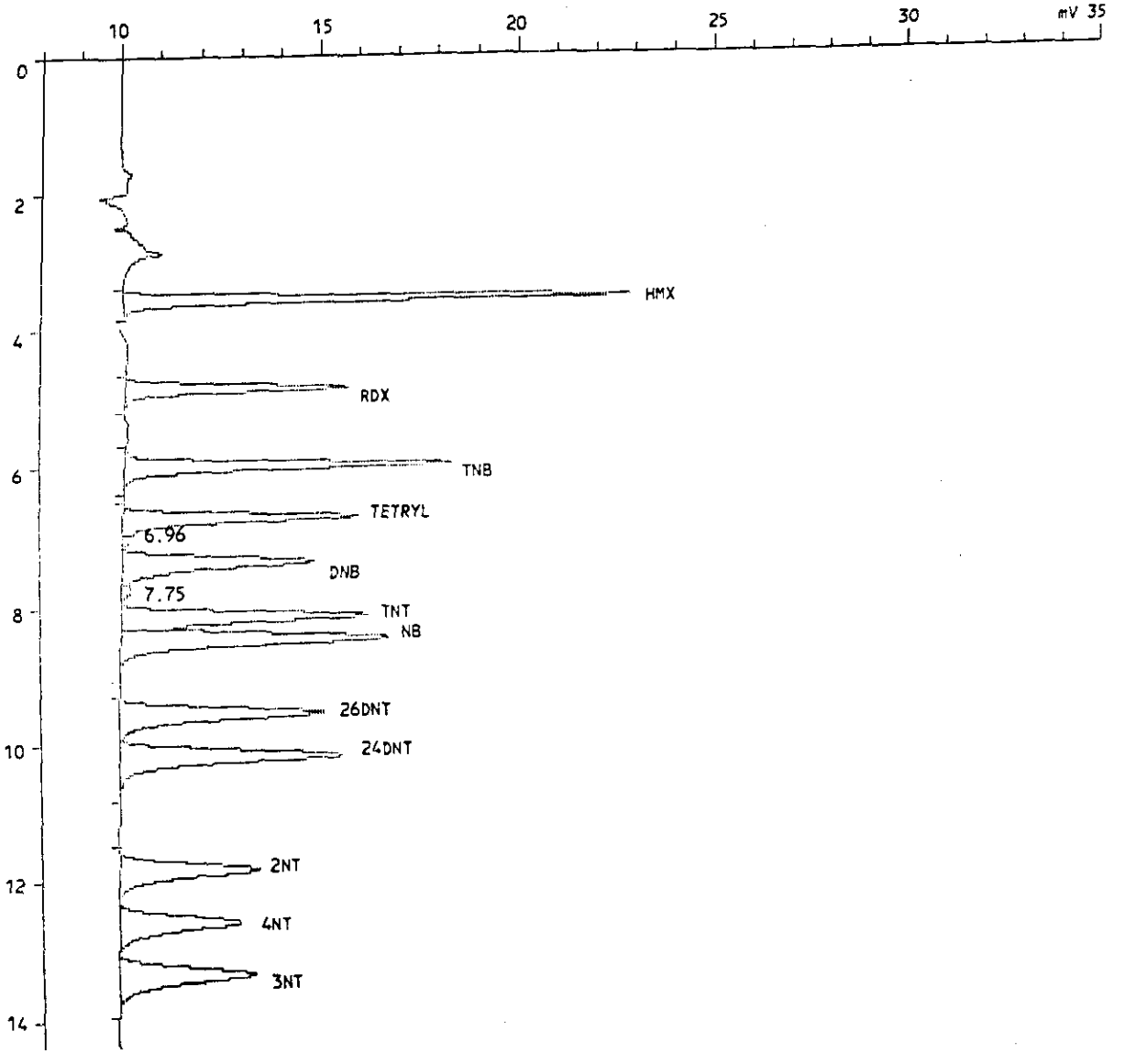
PEAK INFORMATION

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

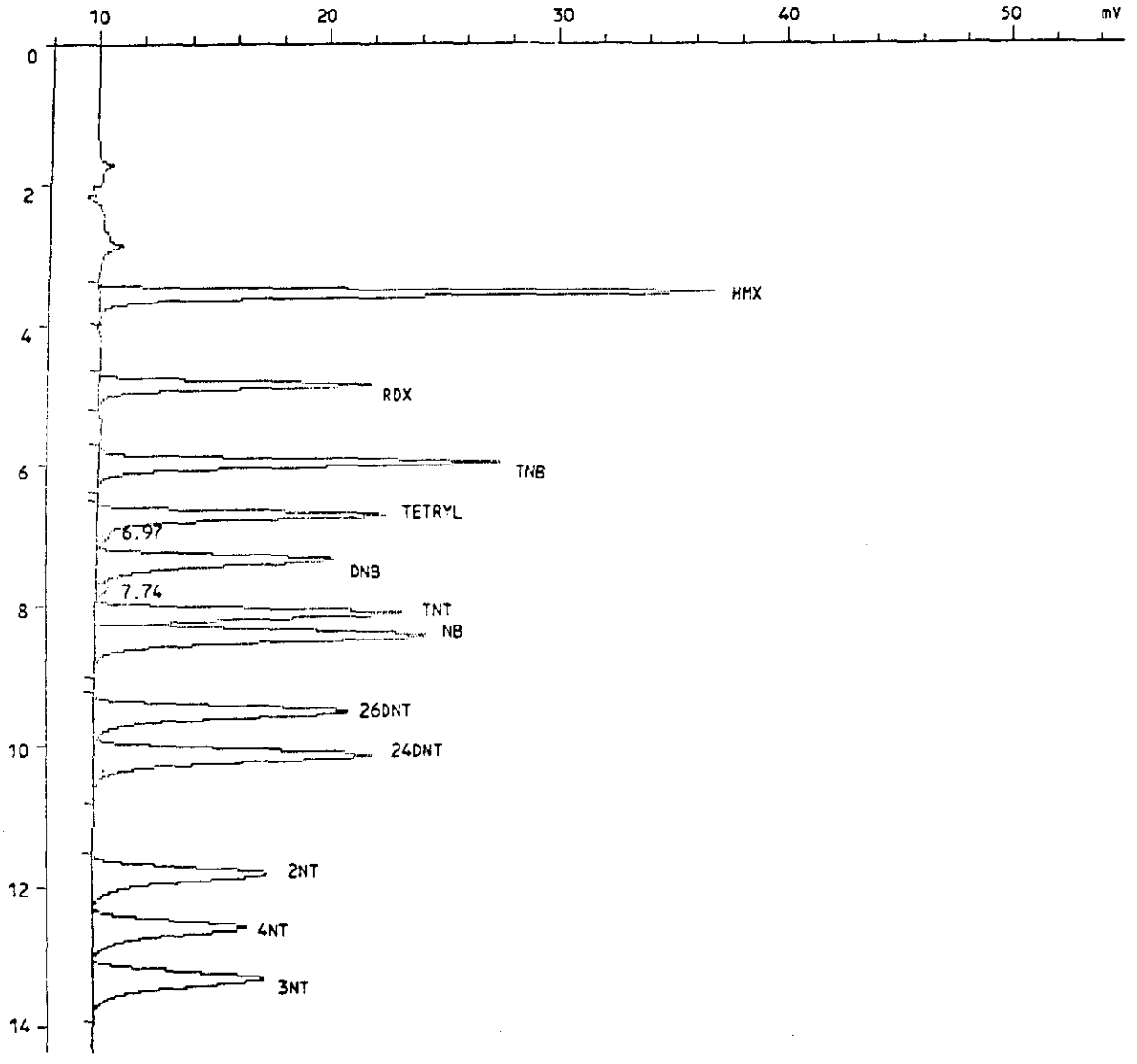
PEAK INFORMATION

Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
1	3.547	12816	84248	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



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

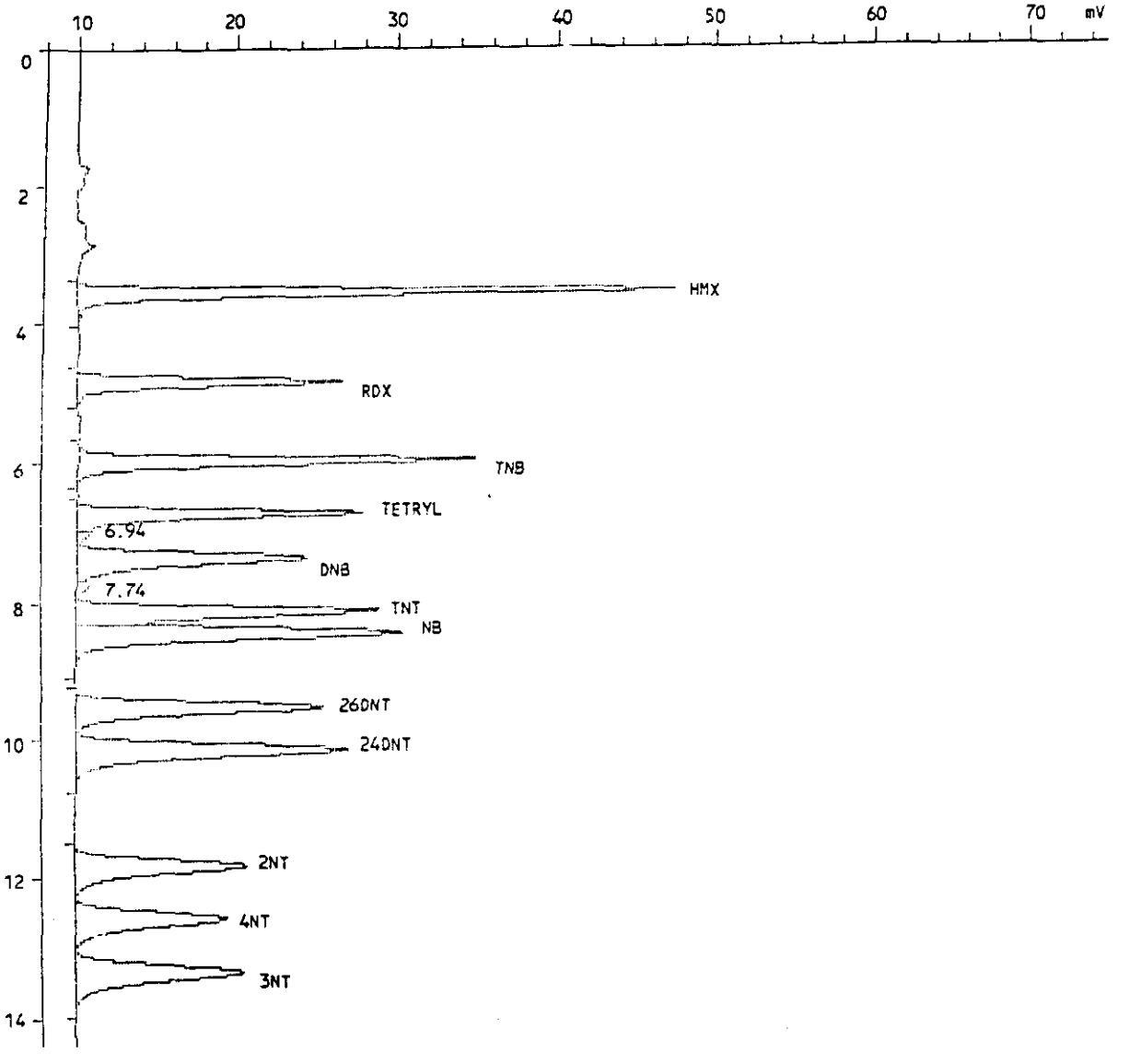
PEAK INFORMATION

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



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

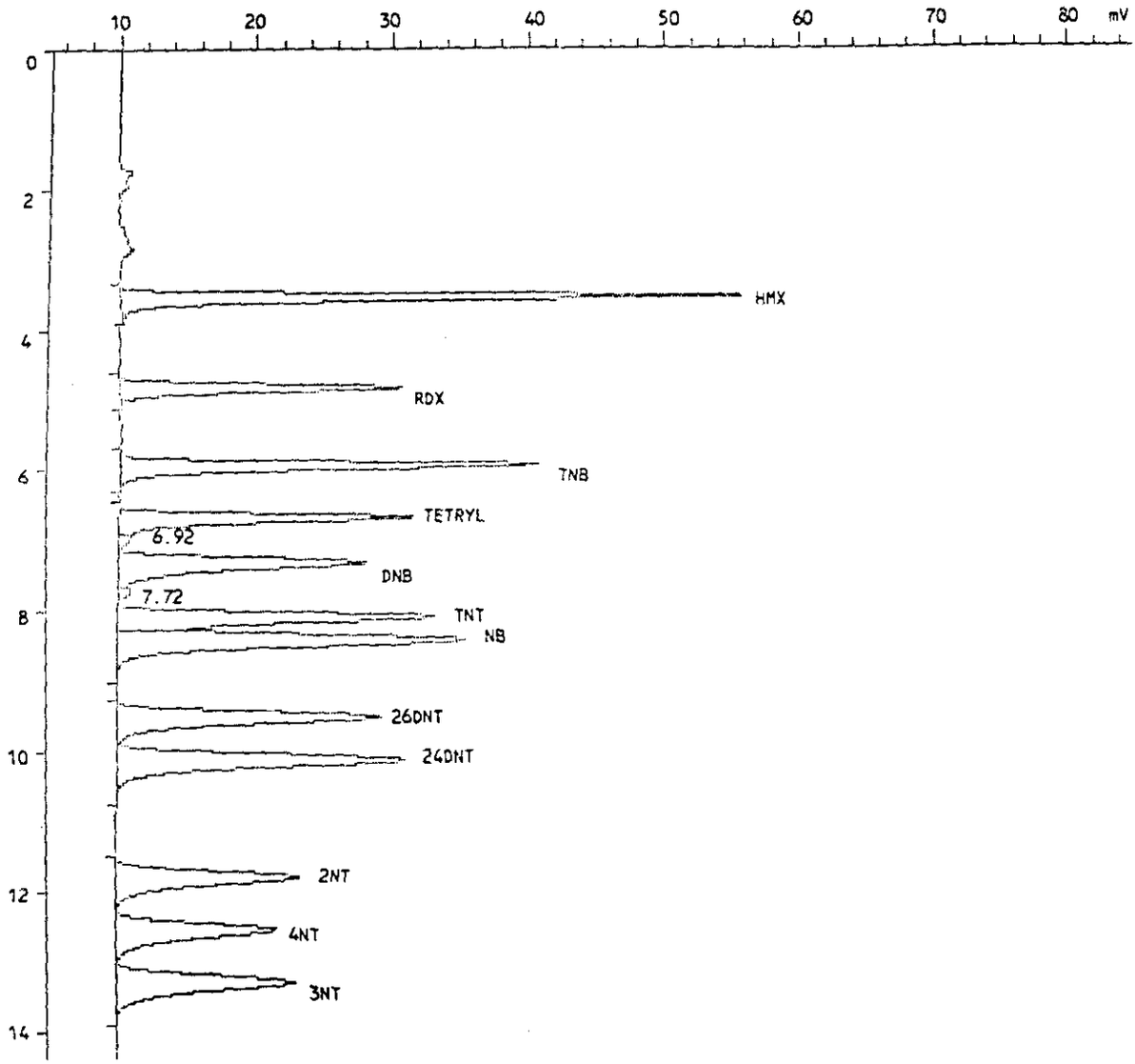
PEAK INFORMATION

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



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 L5A 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

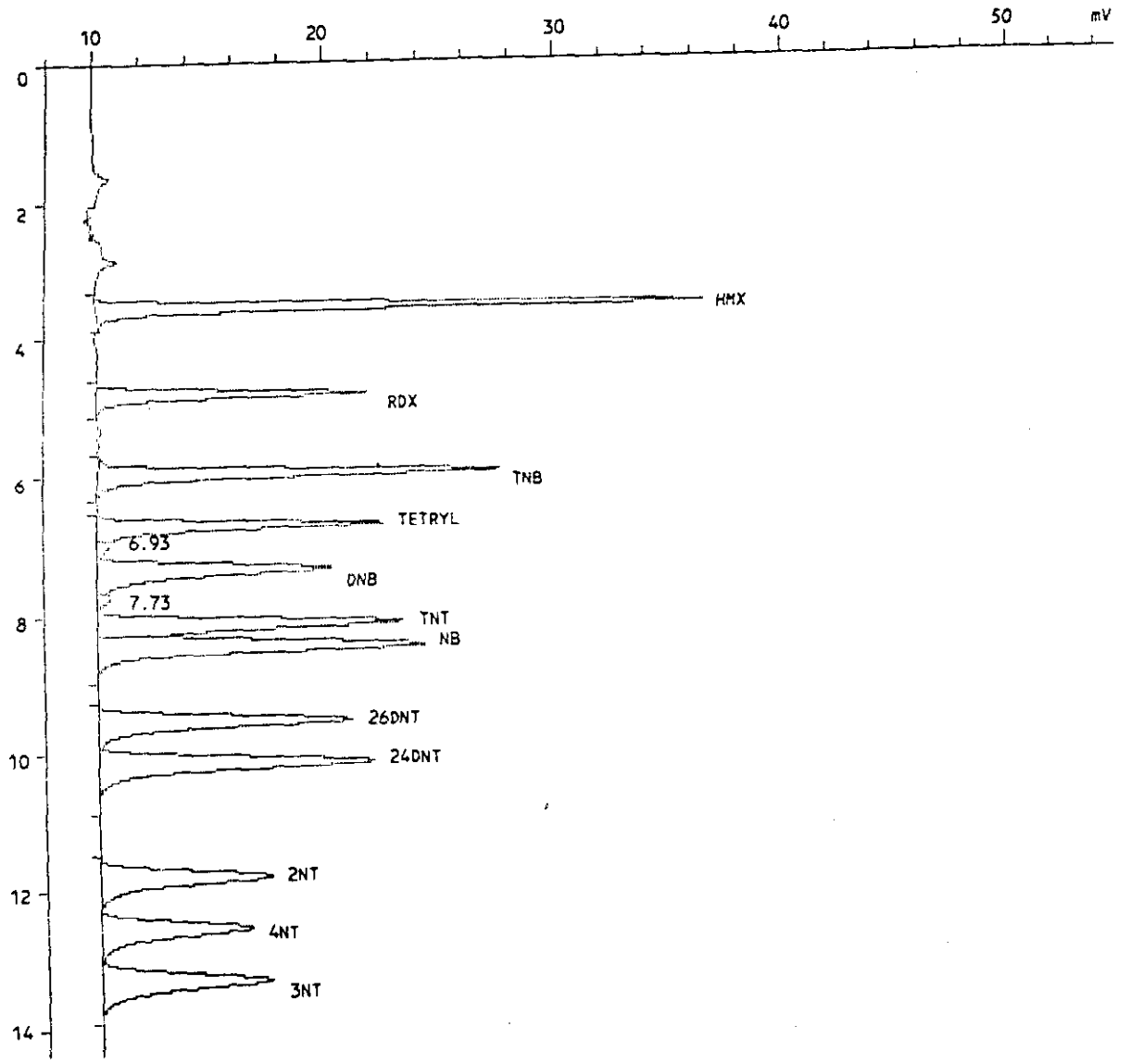
PEAK INFORMATION

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



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

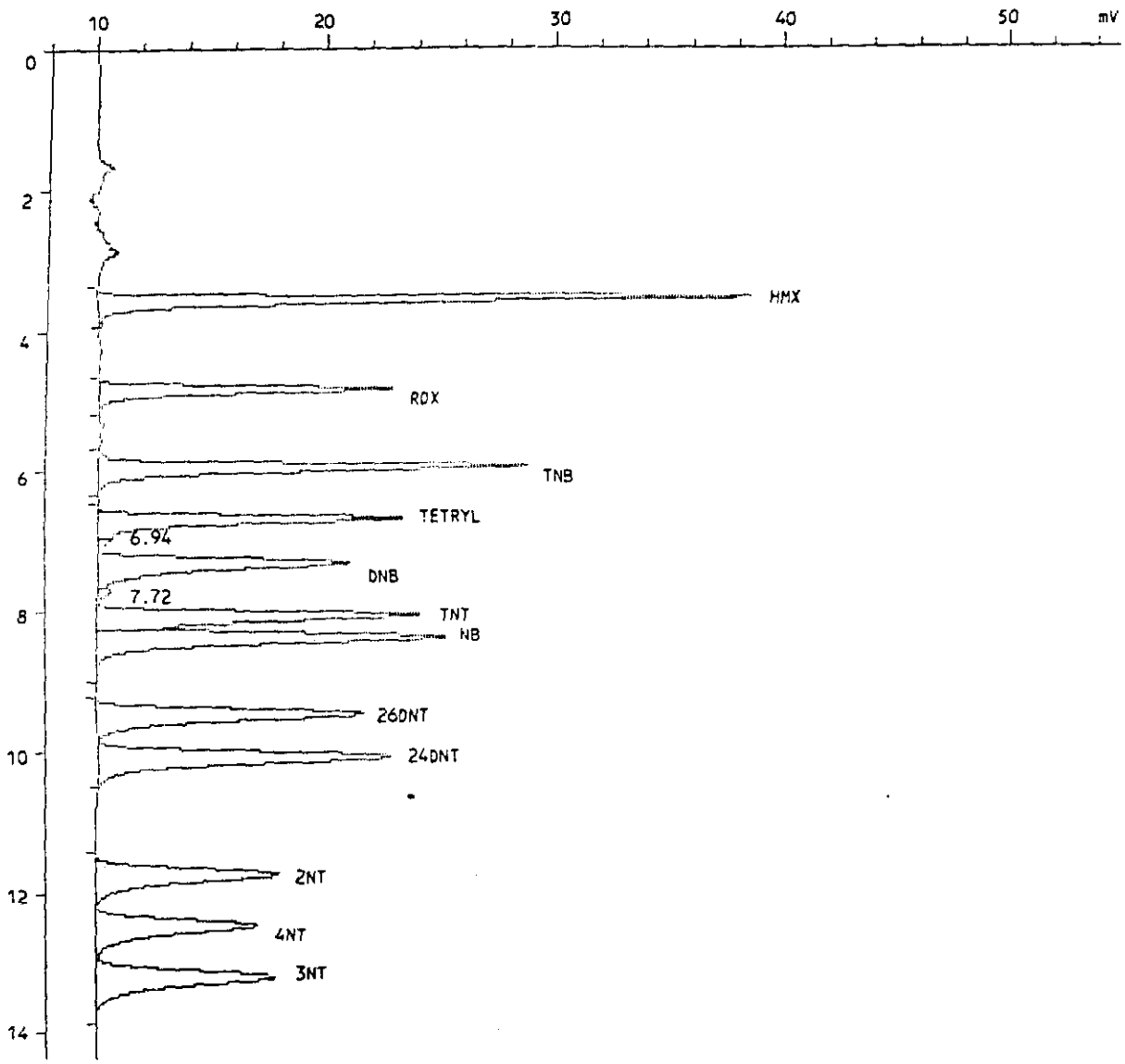
PEAK INFORMATION

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



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-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: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

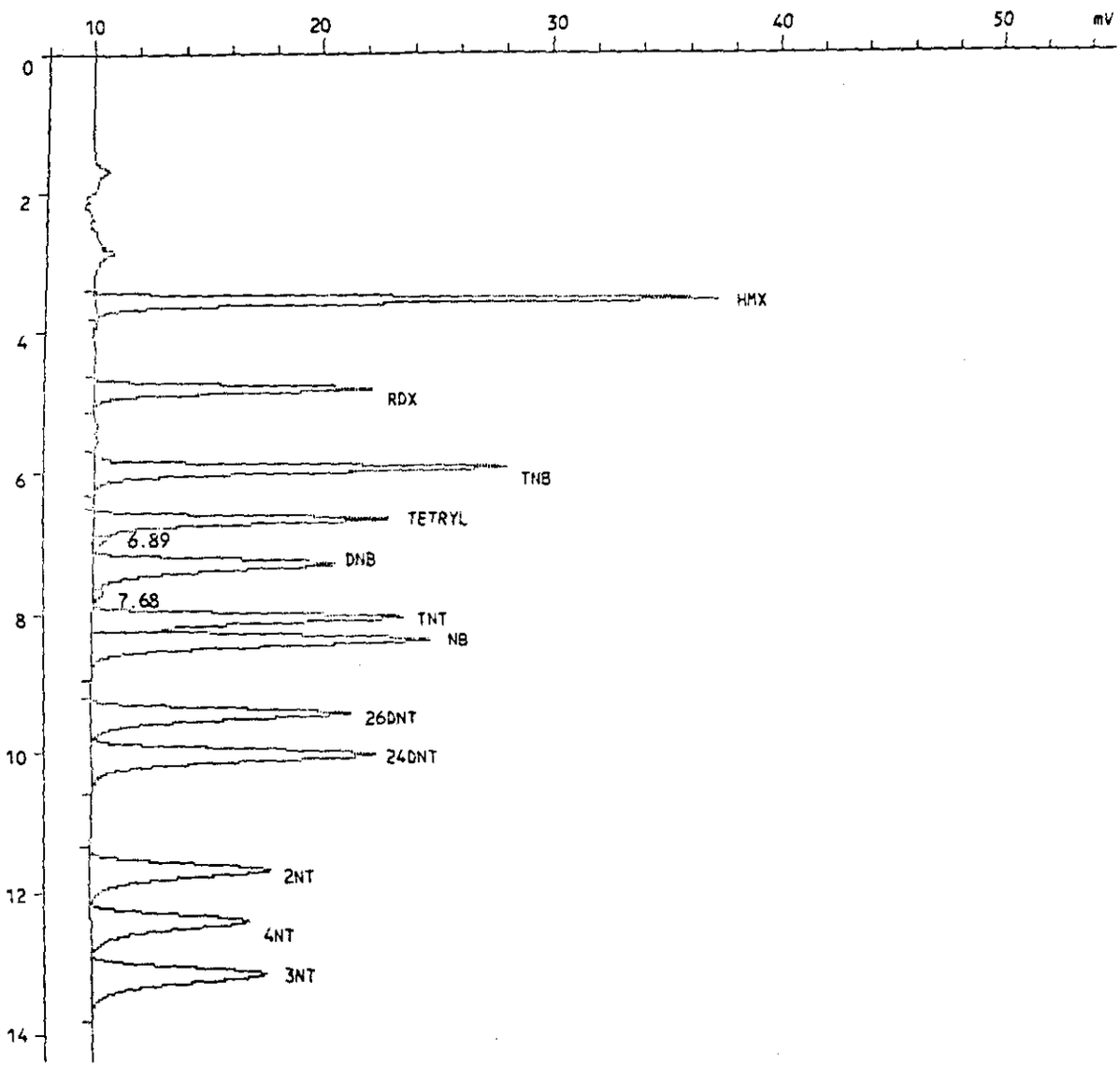
PEAK INFORMATION

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

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



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

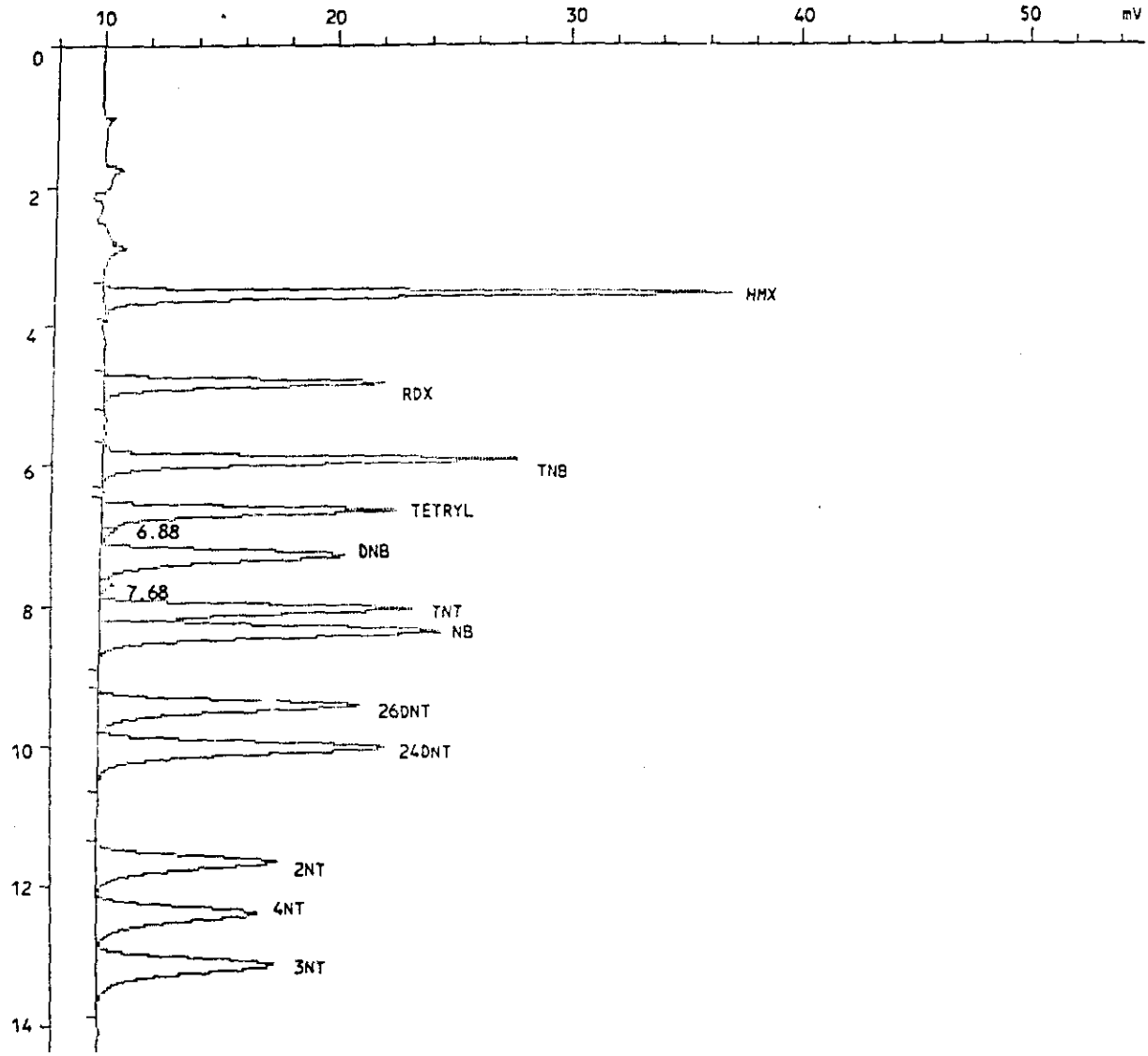
PEAK INFORMATION

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

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



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

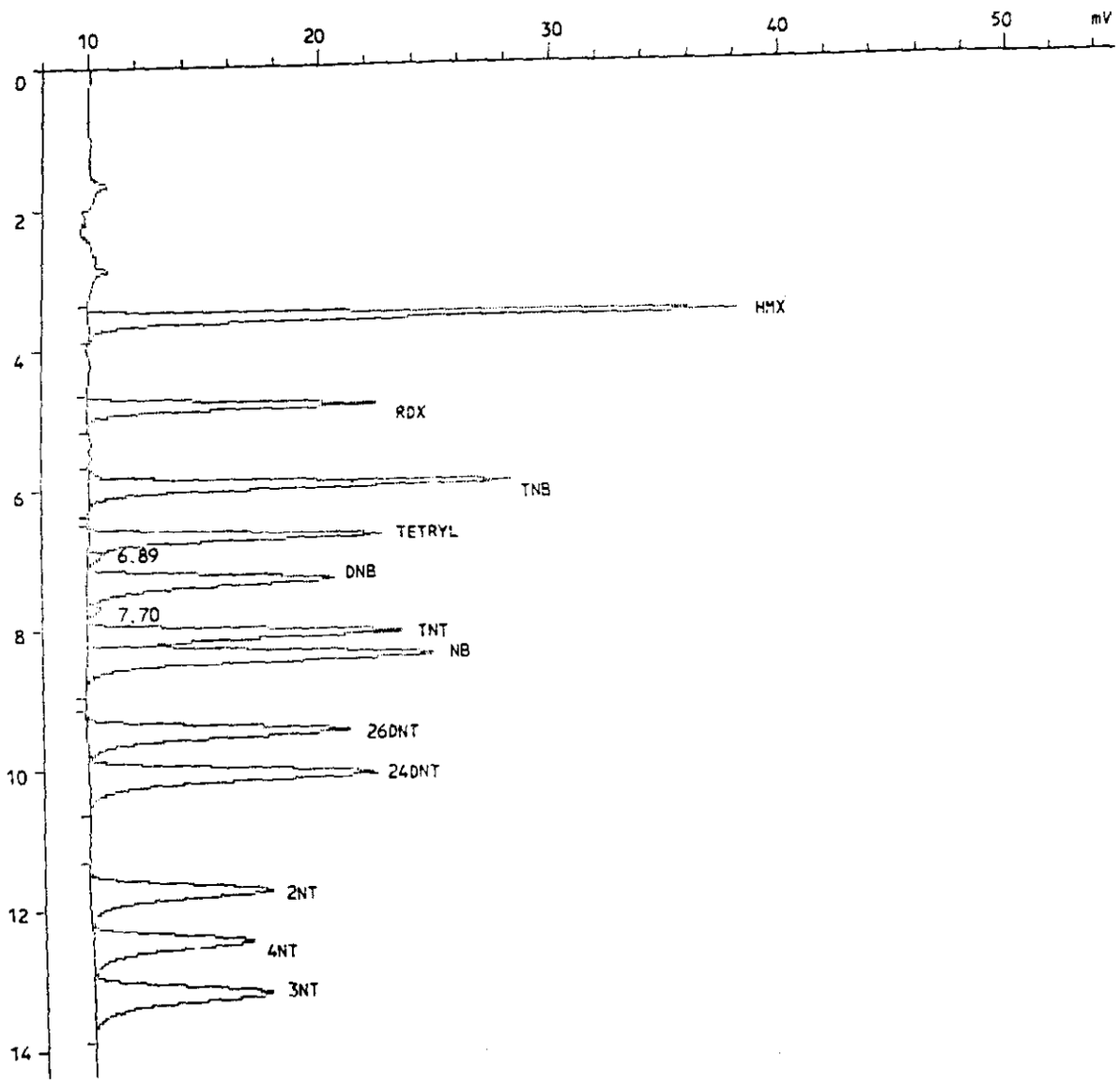
PEAK INFORMATION

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



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

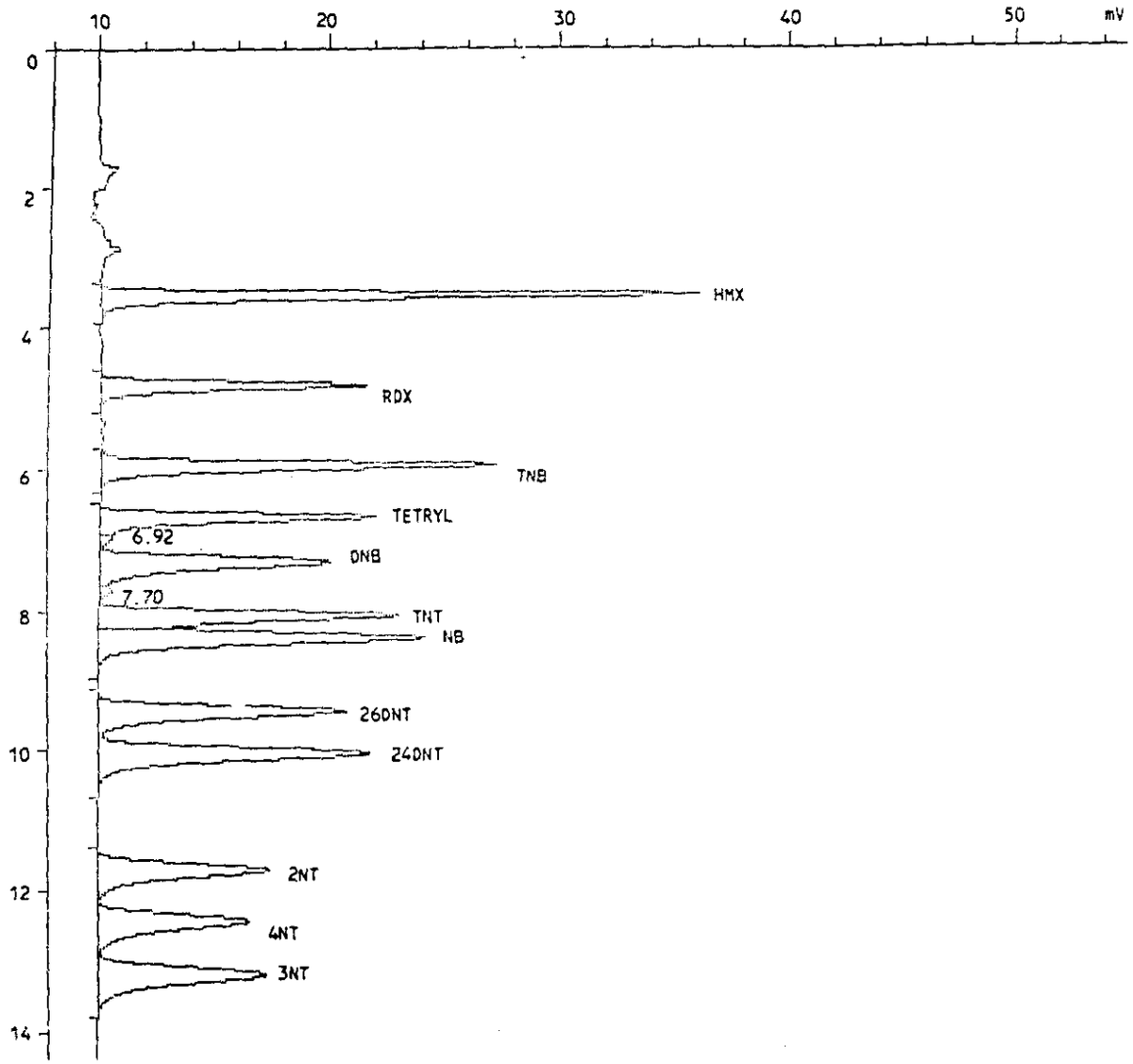
PEAK INFORMATION

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



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 15:08:10

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

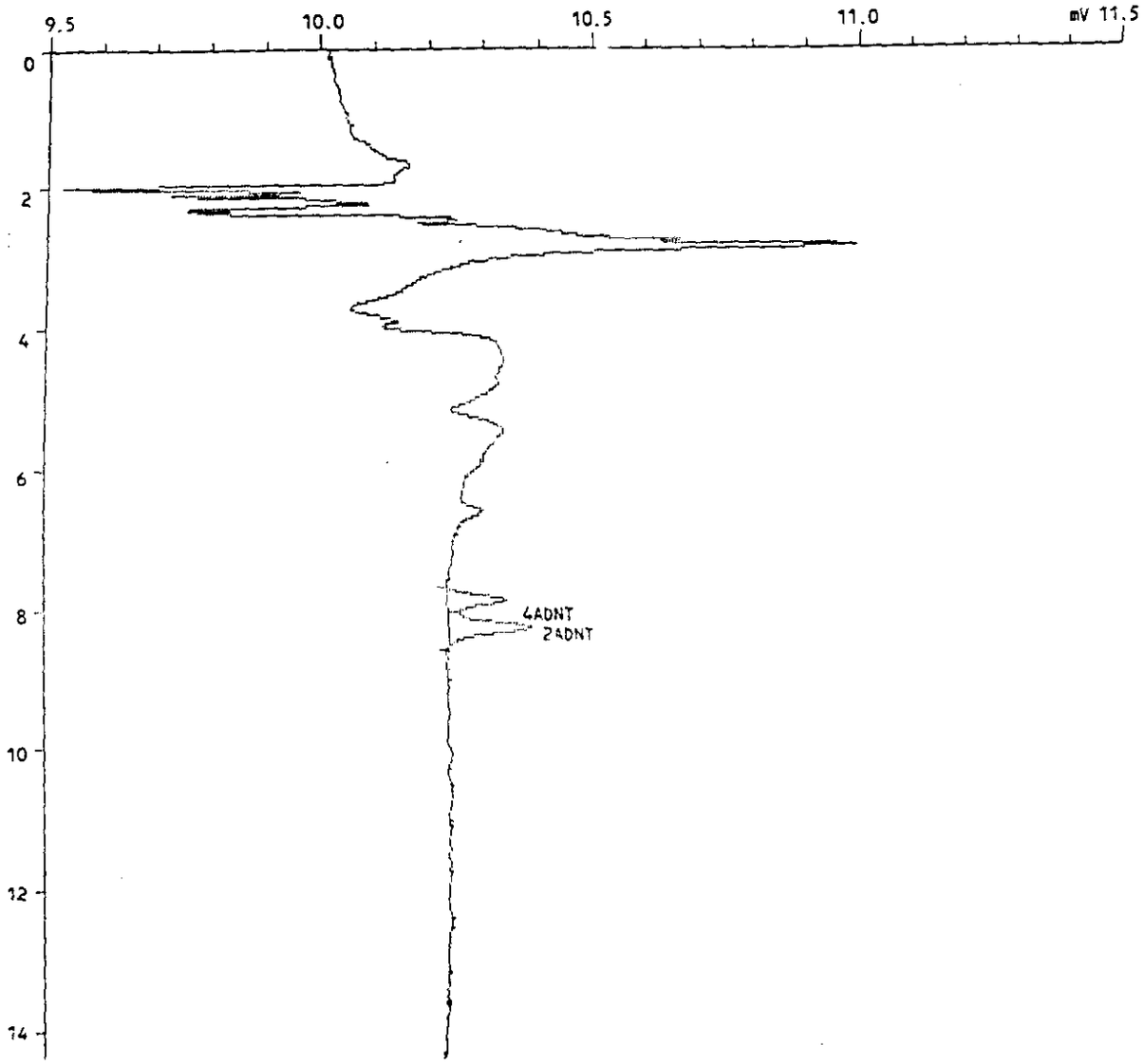
PEAK INFORMATION

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



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

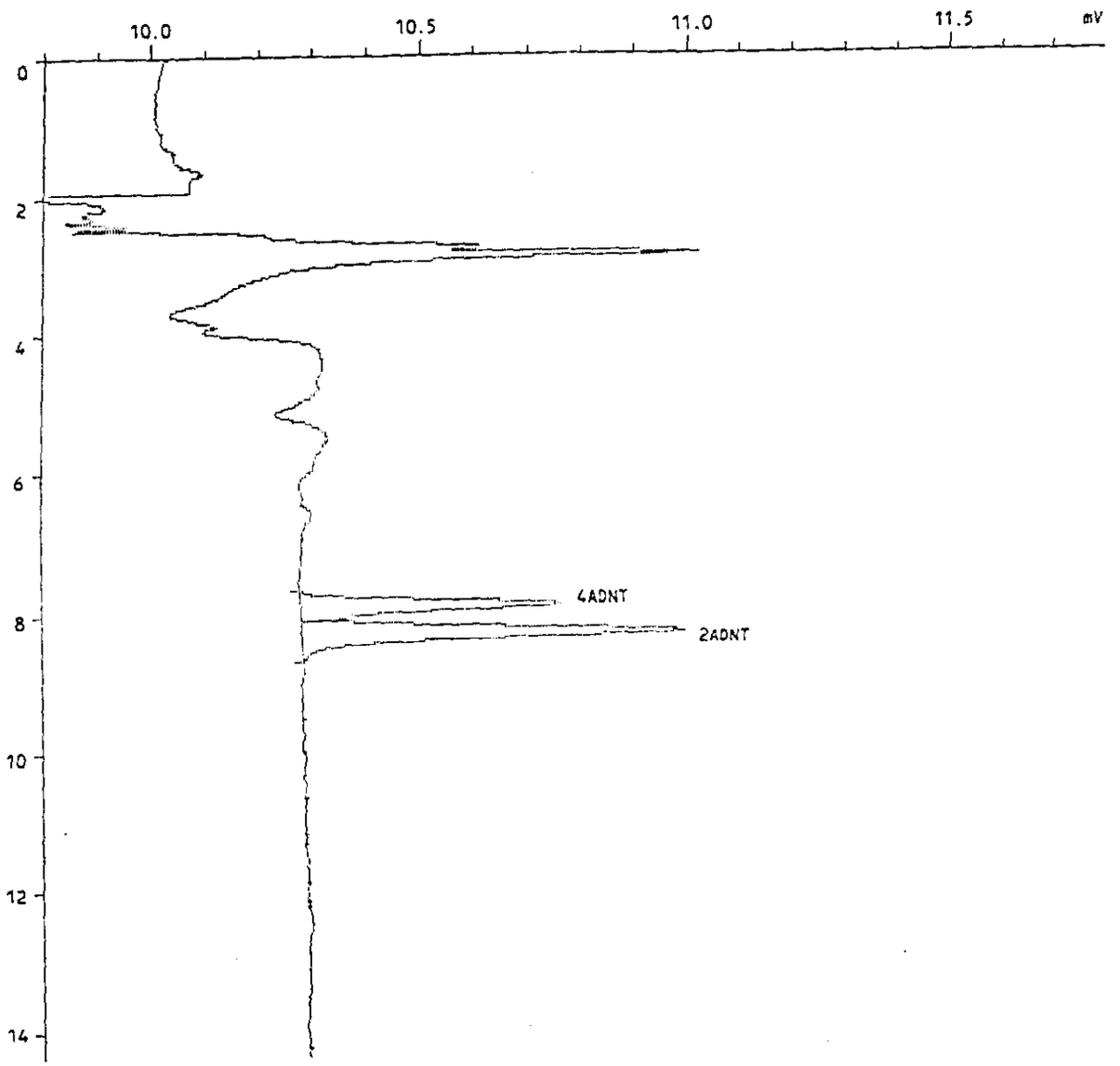
PEAK INFORMATION

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 19-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



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

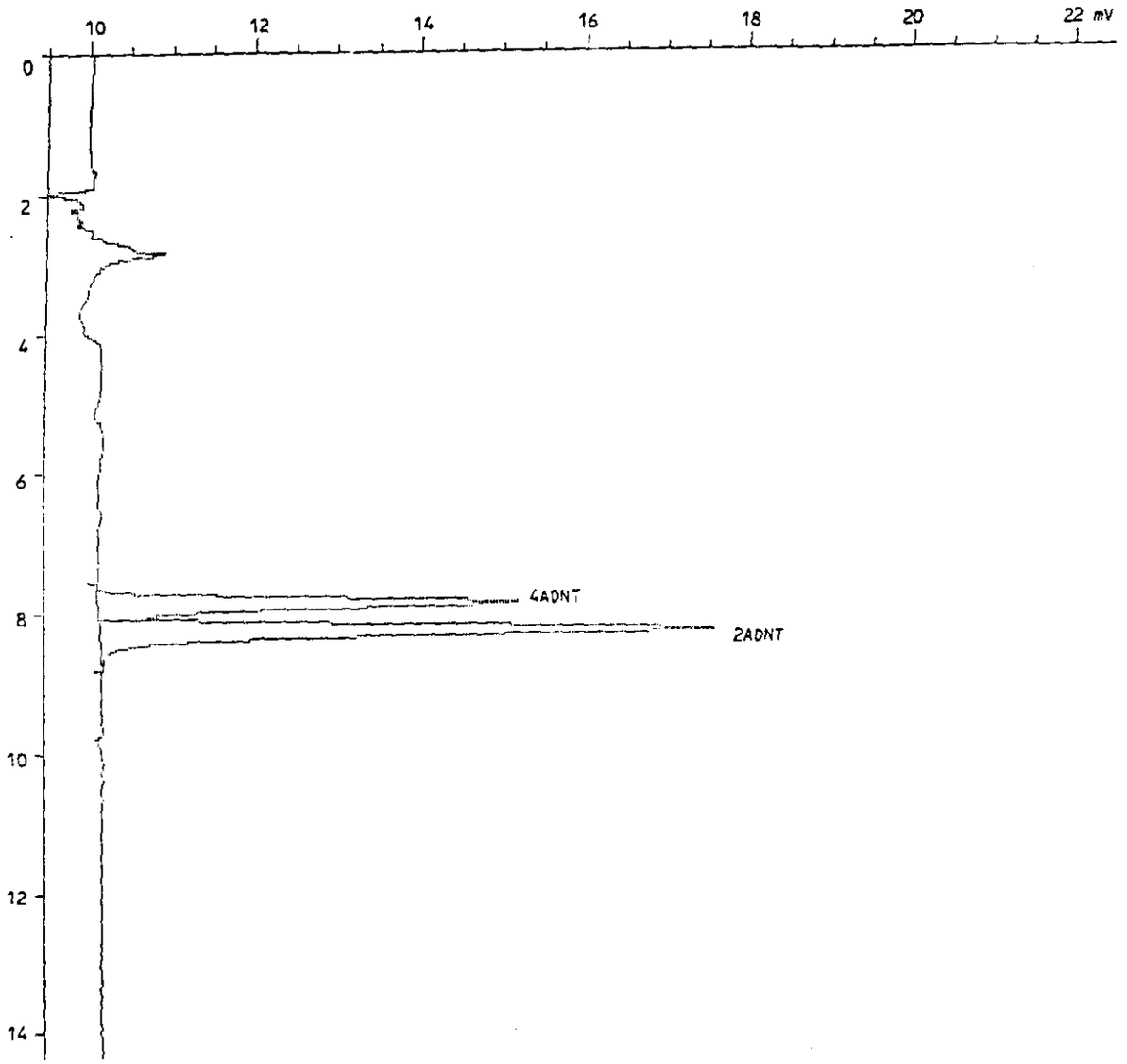
PEAK INFORMATION

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	

INJECTION REPORT

Injection F: <MC3> 1 IEX0711,11,1

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



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

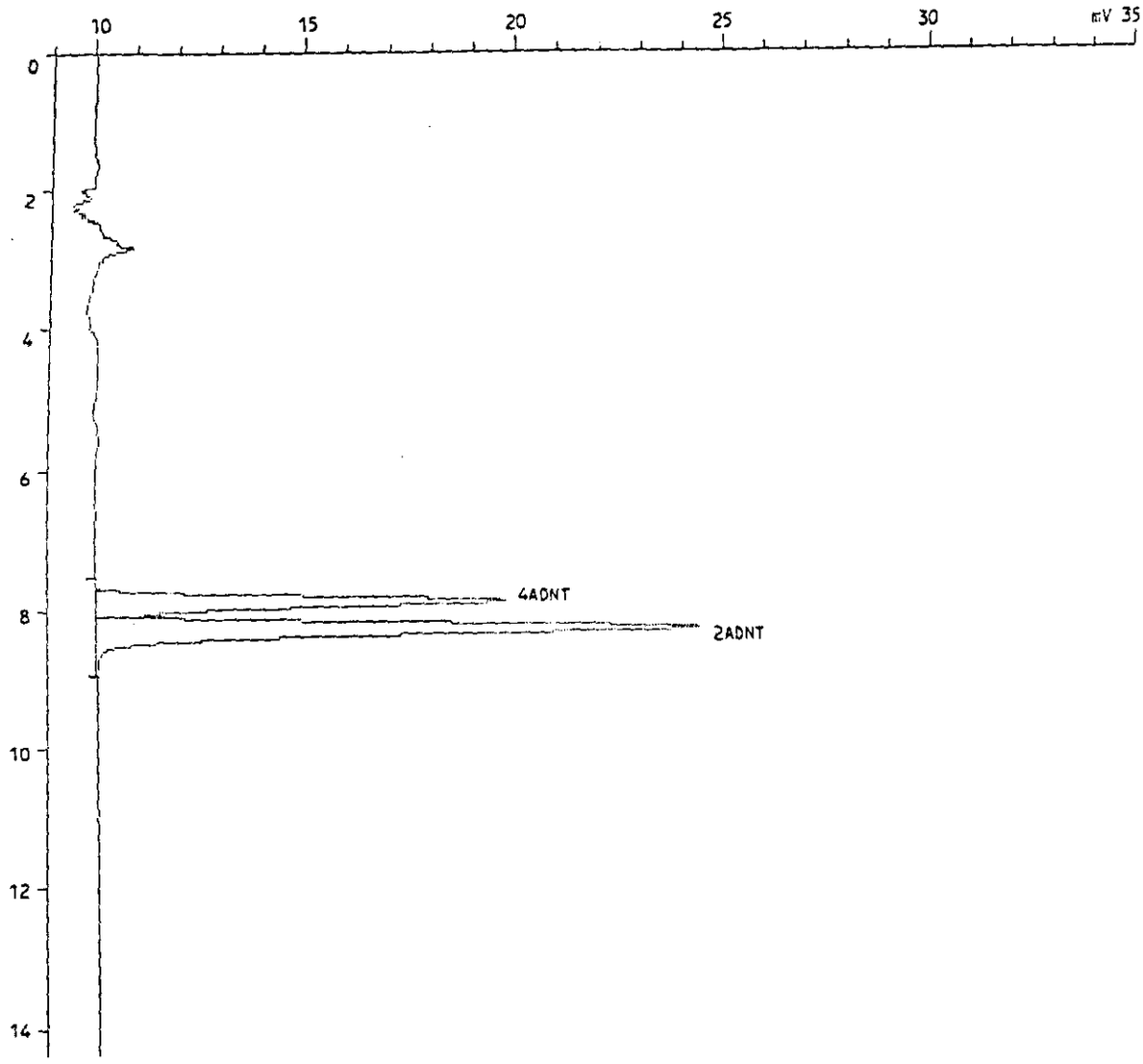
PEAK INFORMATION

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 10-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



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

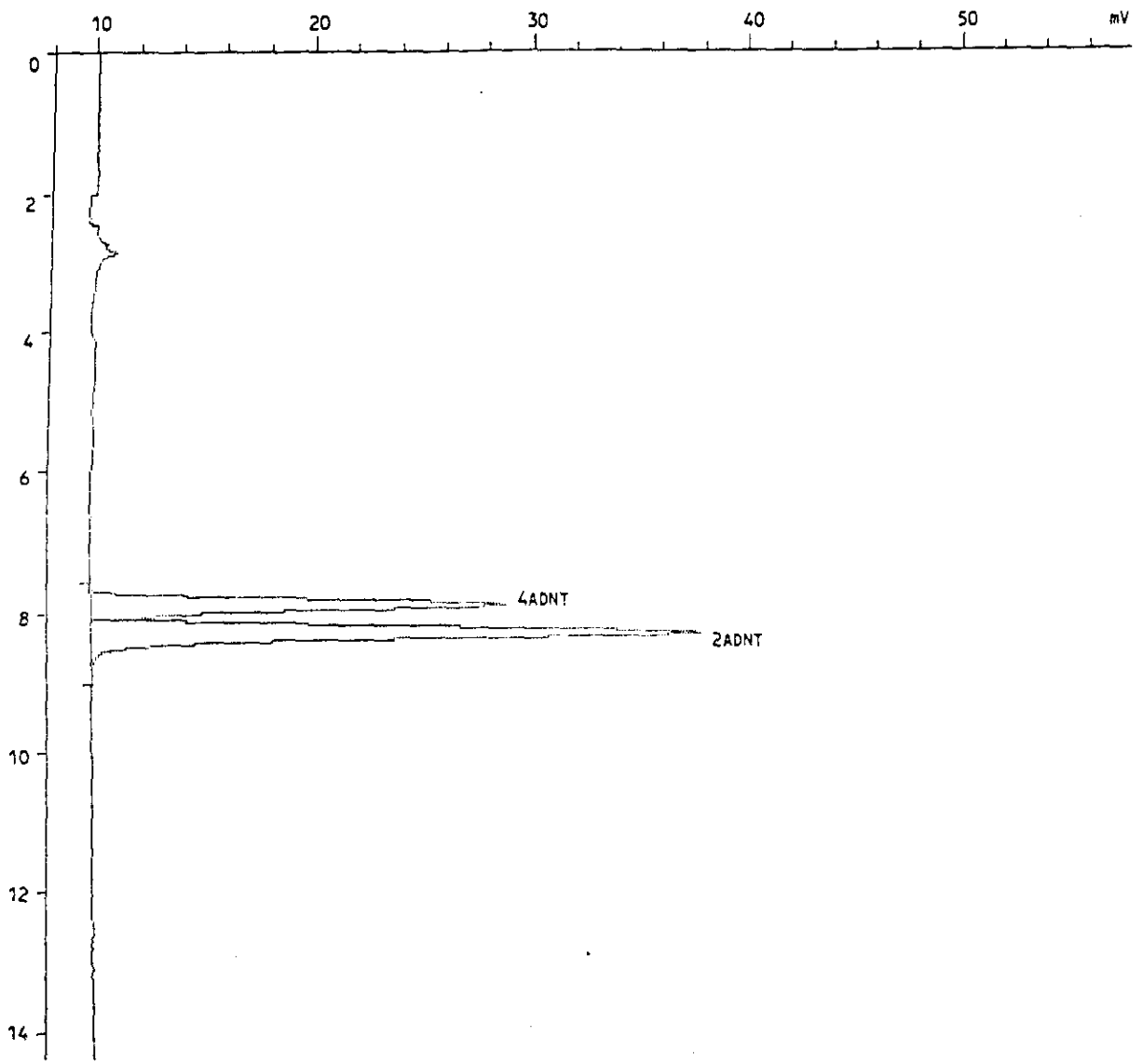
PEAK INFORMATION

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



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

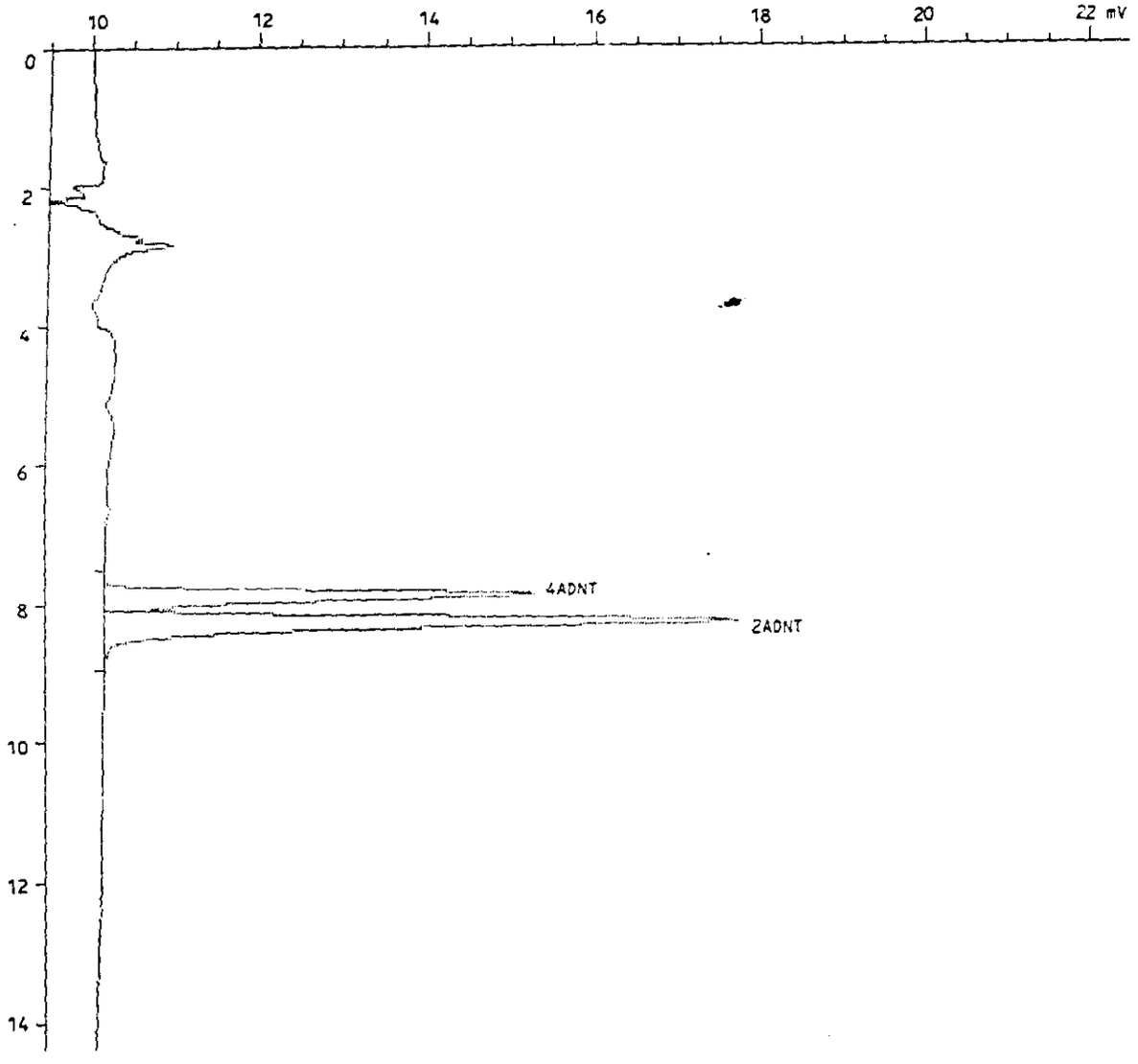
PEAK INFORMATION

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	

INJECTION REPORT

Injection F: <MC3> 1 1EX0711,23,1

Acquired on 12-Jul-96 at 02:14:58
Modified on 16-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



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

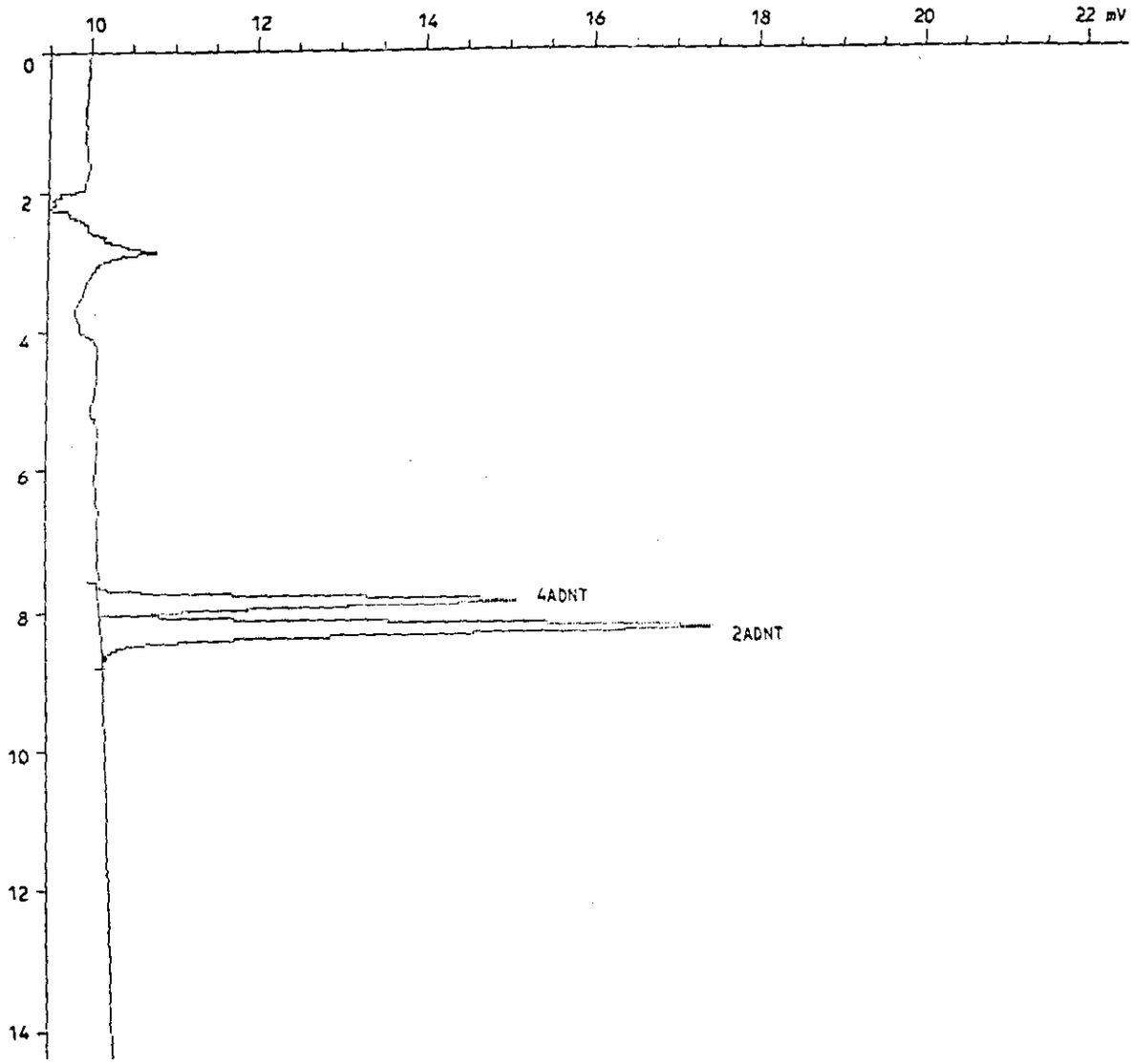
PEAK INFORMATION

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	

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



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

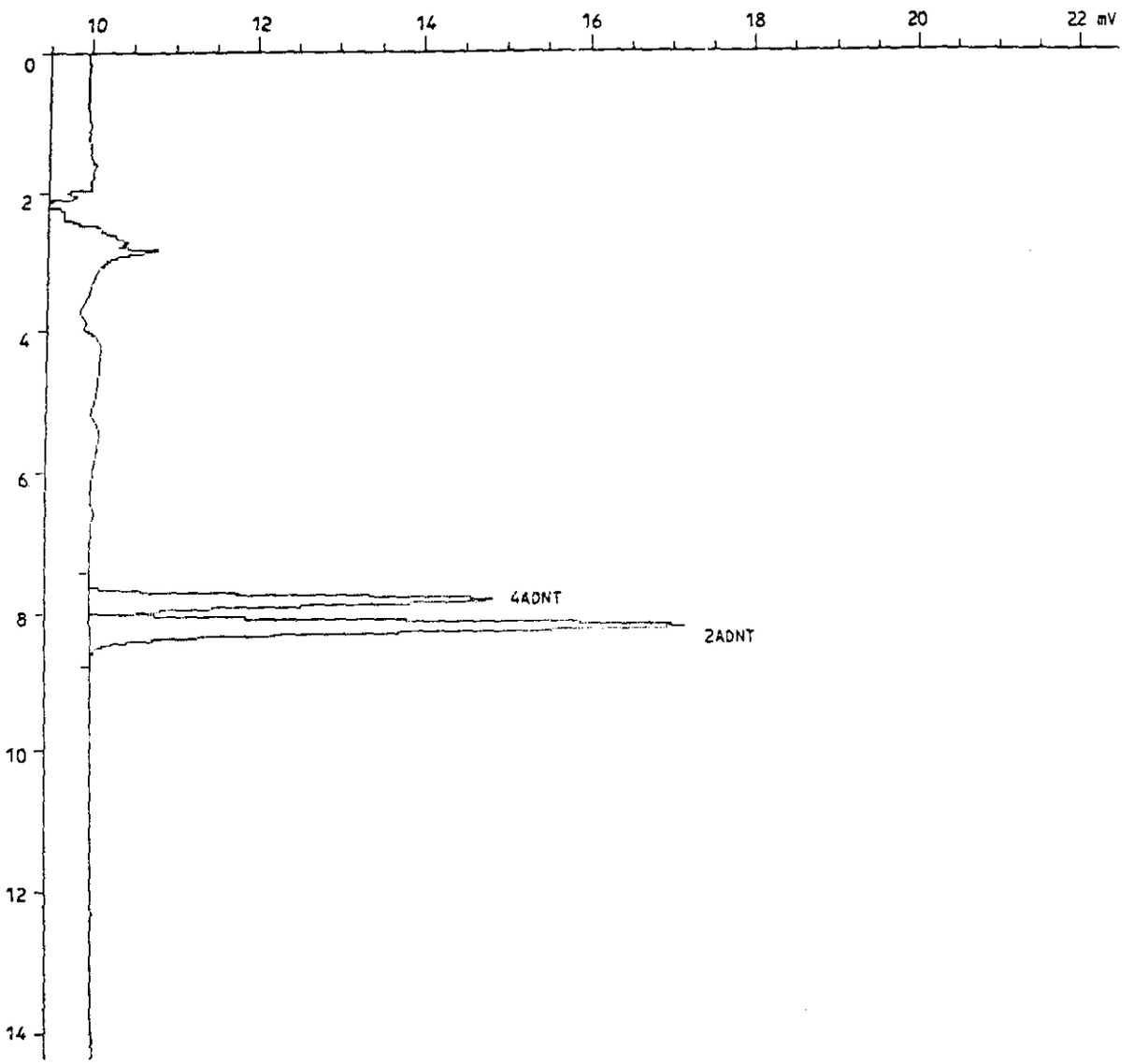
PEAK INFORMATION

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	

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



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

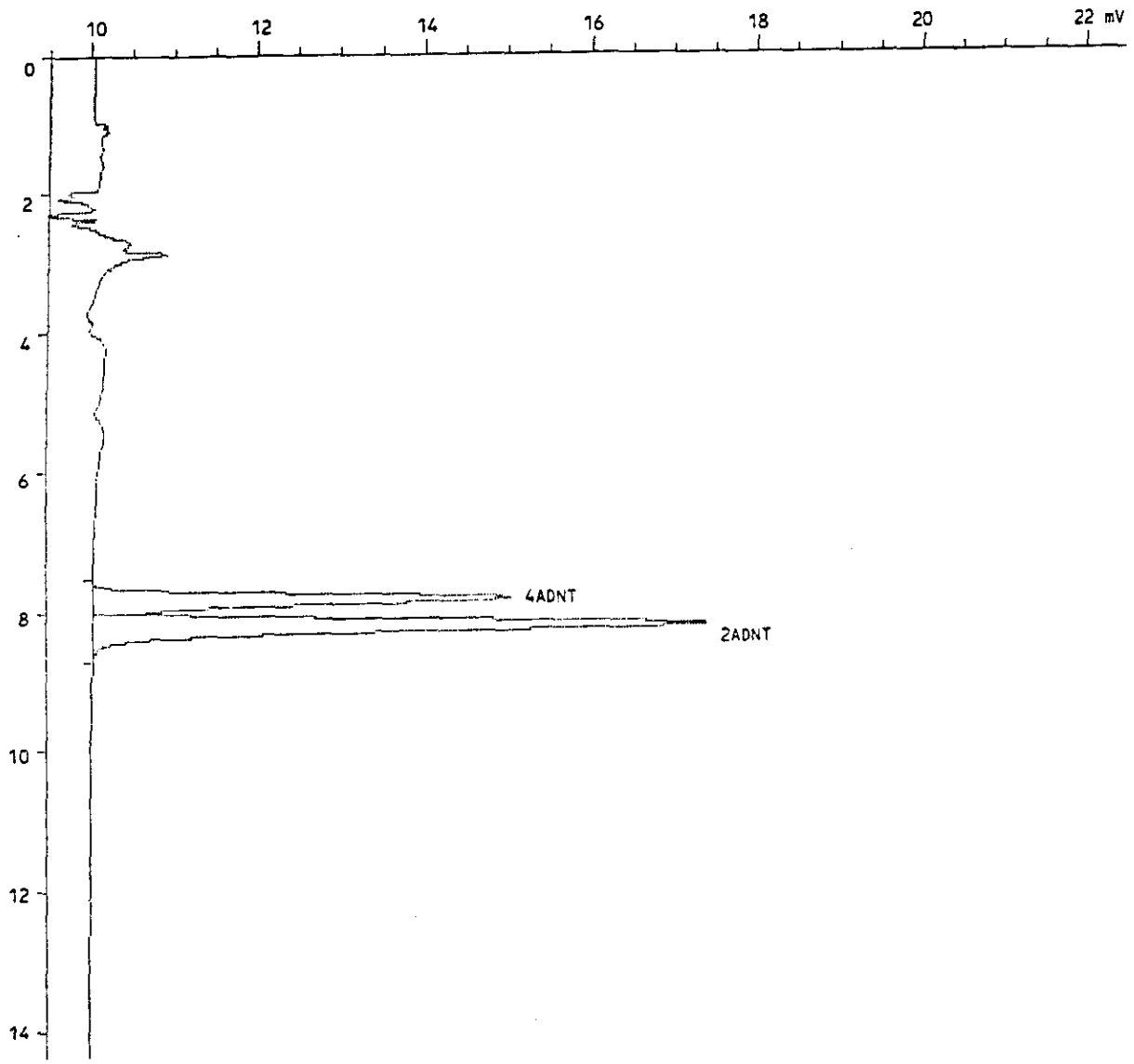
PEAK INFORMATION

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	

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



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 10:25:24

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

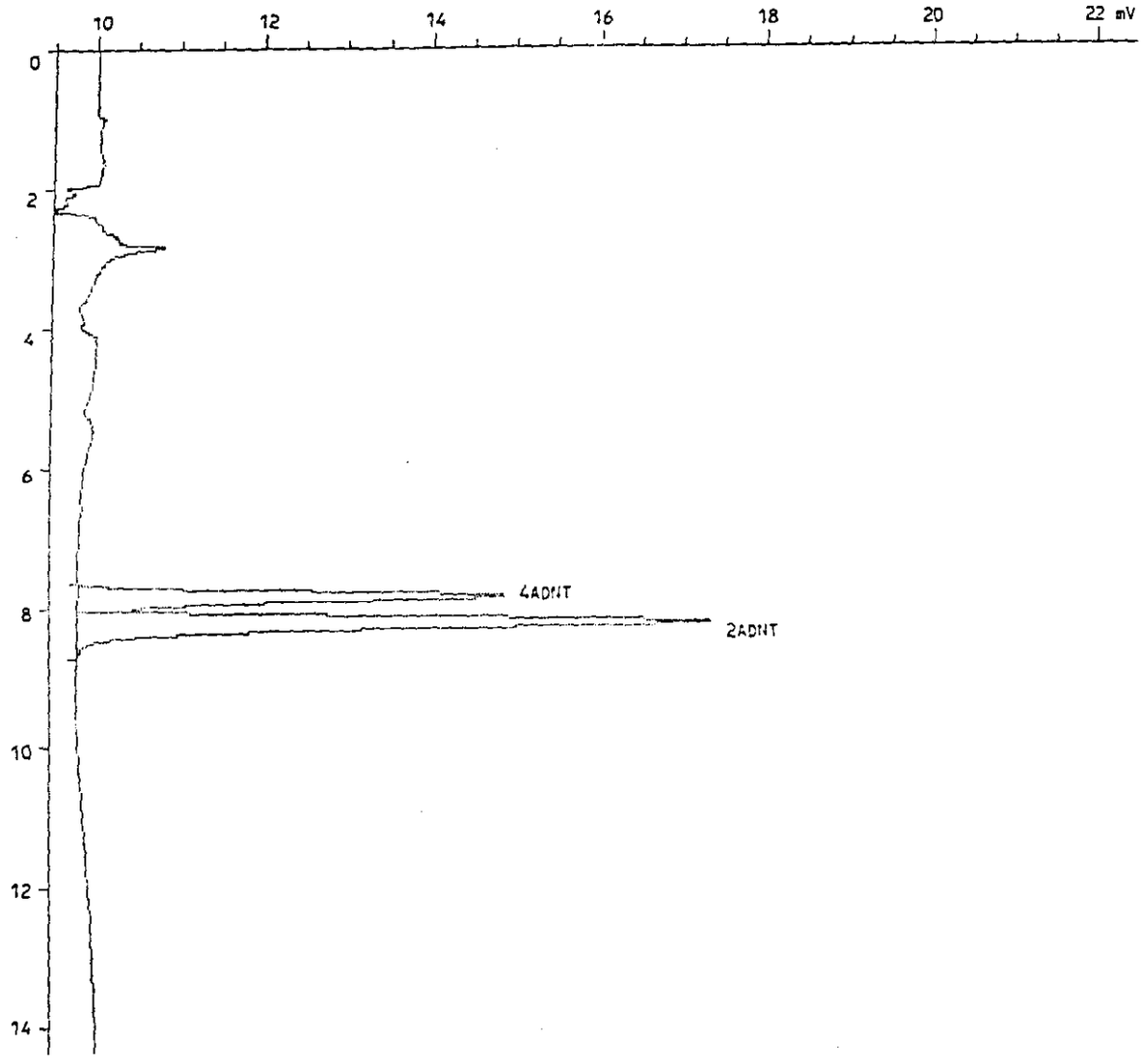
PEAK INFORMATION

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	

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



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:CAS
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

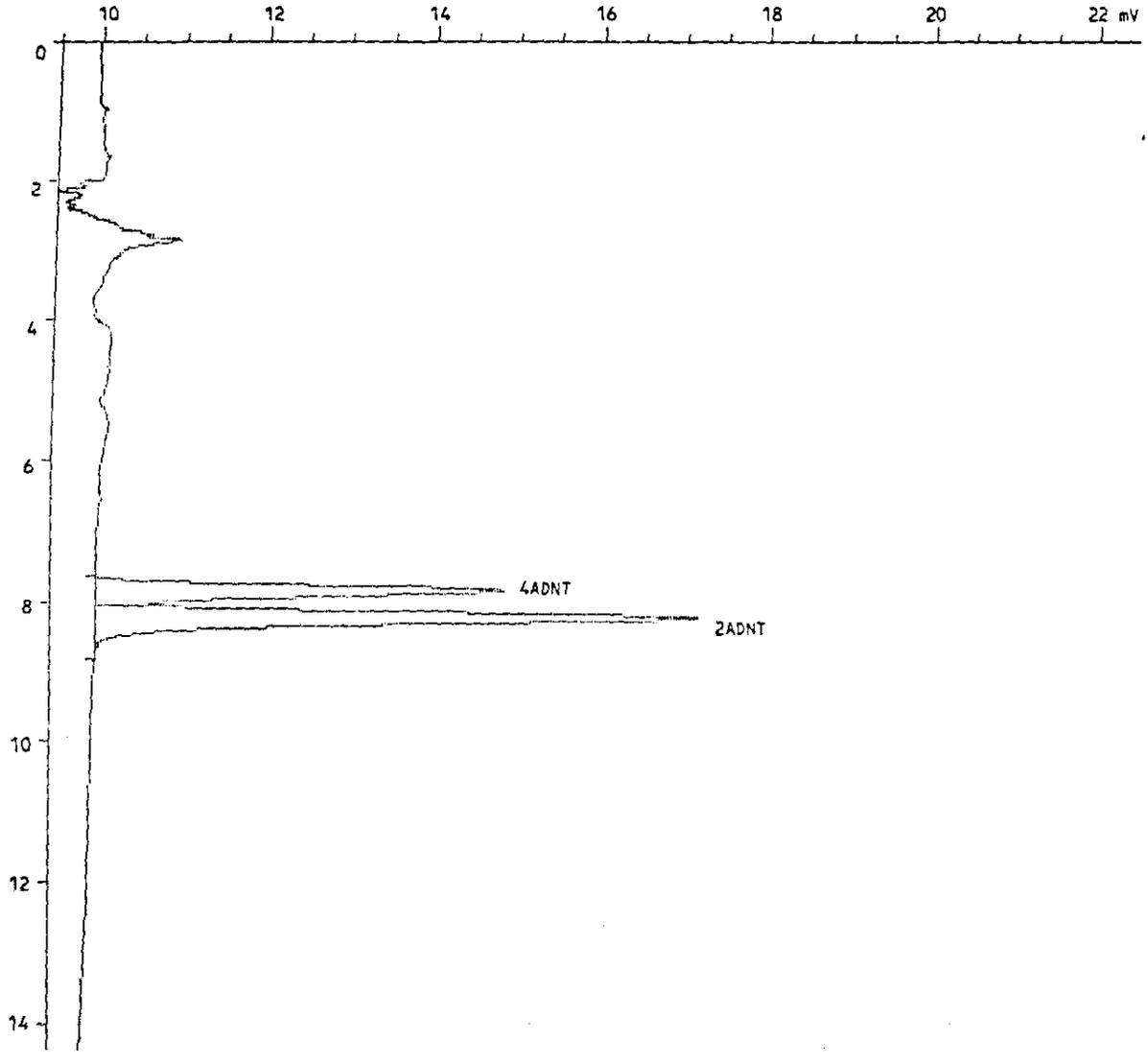
PEAK INFORMATION

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	

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



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	2~~1~~00	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

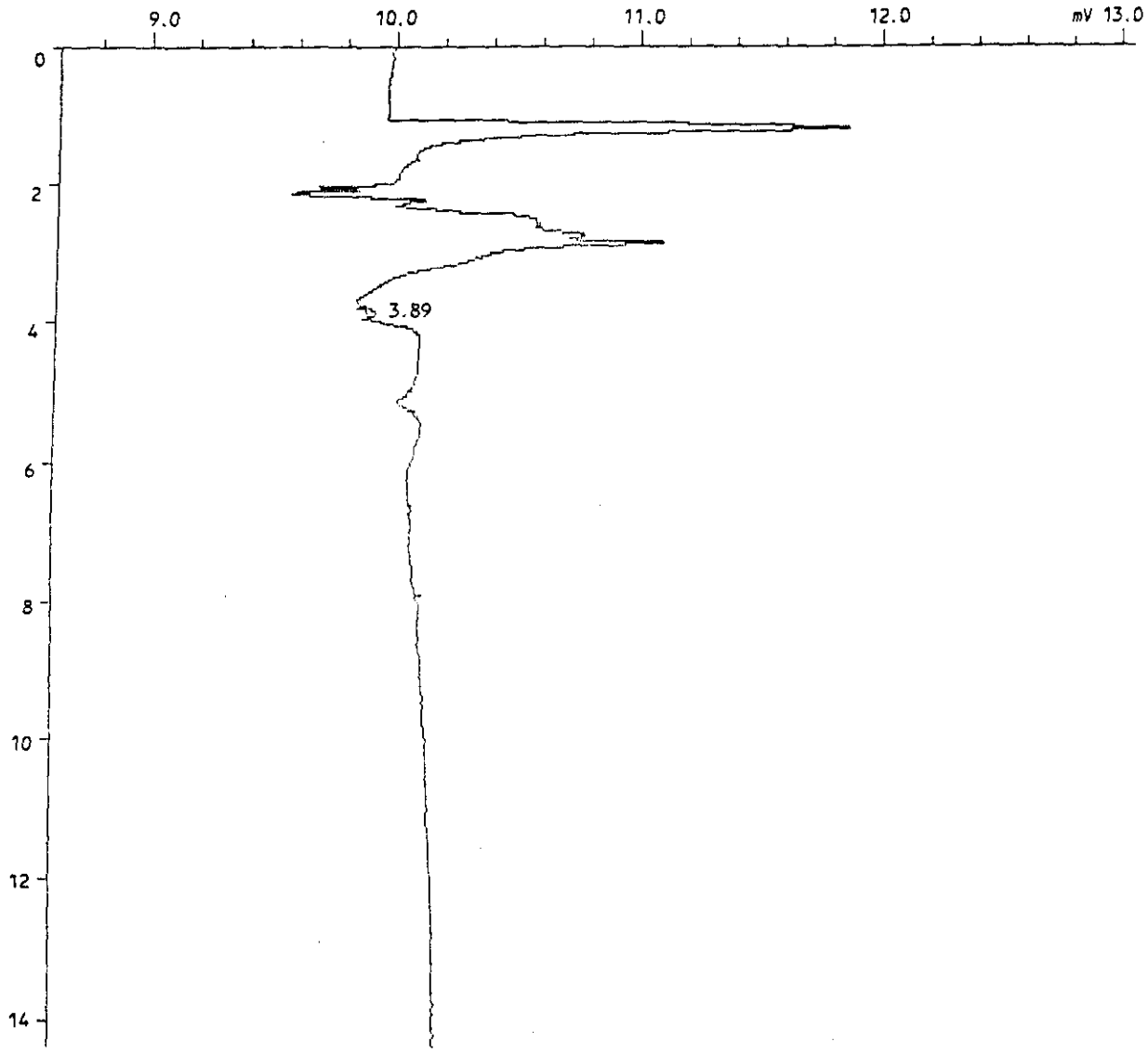
01/07/97

FORM I

INJECTION REPORT

Injection F: <MC3> 1 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



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	

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

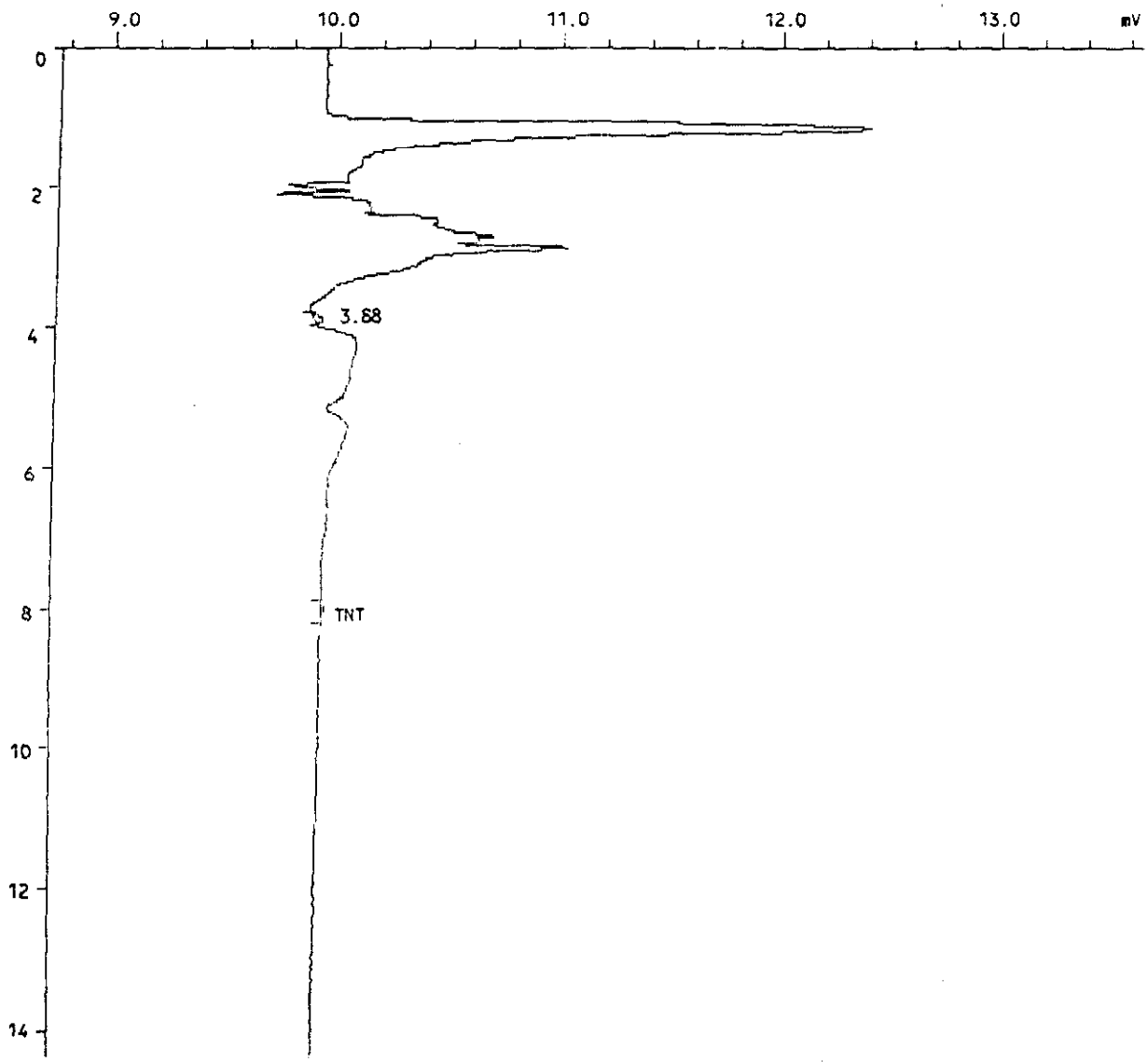
01/07/97

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



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	

APPENDIX H

NEESA 20.2-047B - SAMPLING AND CHEMICAL ANALYSIS 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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SAMPLING AND CHEMICAL ANALYSIS QUALITY ASSURANCE REQUIREMENTS FOR THE NAVY INSTALLATION RESTORATION PROGRAM

NEESA 20.2-047B
Project Manager: Anthony R. Sturtzer

September 1985
First Revision June 1986
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under
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Environmental Restoration Department (Code 112E)
NAVAL ENERGY AND ENVIRONMENTAL SUPPORT ACTIVITY
Port Hueneme, California 93043-5014

H-3

CONTENTS

	Page No.
LIST OF FIGURES	v
LIST OF TABLES	vii
LIST OF ACRONYMS	ix
1. INTRODUCTION	1
1.1 SCOPE	1
1.2 APPROACH	2
1.3 LEVELS OF QC	2
1.3.1 Level D QC	4
1.3.2 Level C QC	4
1.3.3 Level E QC	4
1.4 ROLES AND RESPONSIBILITIES	5
2. APPROVAL PROCESS	9
2.1 OVERVIEW	9
2.1.1 The Laboratory Approval Process	9
2.1.2 Laboratory Reapproval	11
3. SITE-SPECIFIC QC REQUIREMENTS	13
3.1 CONTENTS OF SITE-SPECIFIC QC SECTION	13
3.2 STATE AND REGION REQUIREMENTS FOR SITE QC PLAN	13
3.3 SAMPLING DESIGN	14
3.4 PRESERVATIVES	14
3.5 SAMPLE CONTAINER CLEANING PROCEDURES	14
3.6 ORGANIZATION AND PERSONNEL	18
3.7 FIELD QC SAMPLES	18
3.8 CHAIN OF CUSTODY	20
3.9 SHIPPING REQUIREMENTS	21
3.10 SAMPLE RECEIPT	21
4. LABORATORY QA PLAN REQUIREMENTS	23
4.1 PURPOSE AND SCOPE	23
4.2 ORGANIZATION AND CONTENTS OF PLAN	23
4.3 CONTROL SAMPLES	27
4.4 CONTROL CHARTS	28

CONTENTS (continued)

	Page No.
4.4.1 Method Blank/Spike Control Program	29
4.4.2 Control Sample Quality	29
4.4.3 Minimum Statistical Control Charting	30
4.4.4 Minimum Criteria For An Out-Of-Control Condition	30
4.4.5 Reactions to Out-of-Statistical-Control Conditions on Control Samples	31
4.4.6 Administration of the Control Charts	31
4.4.7 Statistical Quality of the Control Charts	31
4.4.8 Example of Setting Control Limits	32
4.5 OUT-OF-CONTROL EVENTS	33
4.6 CORRECTIVE ACTION REPORT	37
5. PROFICIENCY TESTING	39
5.1 SUBMITTING THE PROFICIENCY SAMPLES	39
5.2 RESULTS OF PROFICIENCY SAMPLES	39
5.3 EVALUATION OF PROFICIENCY SAMPLE RESULTS	39
6. LABORATORY INSPECTION	41
6.1 PURPOSE OF INSPECTION	41
6.2 INSPECTION PROCEDURE	41
7. ANALYTICAL METHODS	43
7.1 QC REQUIREMENTS FOR THE LABORATORY	44
7.2 DELIVERABLES	60
7.3 DATA VALIDATION	60
7.3.1 Level D Validation	60
7.3.2 Level C Data Validation Guidelines	63
8. MAINTAINING LABORATORY APPROVAL	69
8.1 MONTHLY PROGRESS REPORT	69
8.2 FINAL REPORT	70
8.3 FINAL QC DATA REPORT	71
9. DATA FORMAT	73
GLOSSARY	75
BIBLIOGRAPHY	83
KEY CONTACTS FOR THE NAVY INSTALLATION RESTORATION PROGRAM	87

LIST OF FIGURES

Figure No.	Title	Page No.
1.1	QA Organizations	6
2.1	Approval and Review Process	10
2.2	Laboratory Reapproval Process	12
4.1	Control Chart Example Data and Control Value of Table 4.3	35
4.2	Control Chart Example Data and Second Iteration Control Values of Table 4.3	36
9.1	Example of Data Format for Final Report	74

LIST OF TABLES

Table No.	Title	Page No.
1.1	Overall Plan for QC Based on Type of Site	3
3.1	Required Containers, Preservation Techniques, and Holding Times	15
3.2	Preservative and Holding Times for the CLP	16
3.3	Preservative and Holding Times for EPA-Document SW846 (3rd Ed.)	17
3.4	Field QC Samples Per Sampling Event	19
4.1	Typical Methods to be Monitored Through Measurement Control Program	31
4.2	Control Charts Formulae for Water and Soil Control Batch Program	32
4.3	Data and Caculations for Control Chart Example	34
7.1	List of Approved Biological Test Procedures	45
7.2	List of Approved Inorganic Test Procedures	46
7.3	List of Approved Test Procedures for Nonpesticide Organic Compounds	54
7.4	List of Approved Test Procedures for Pesticides	56
7.5	List of Approved Radiological Test Procedures	58
7.6	Data Set Deliverables for Level C QA	61

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

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 HCR.
[3]Includes methods from SW 846, American 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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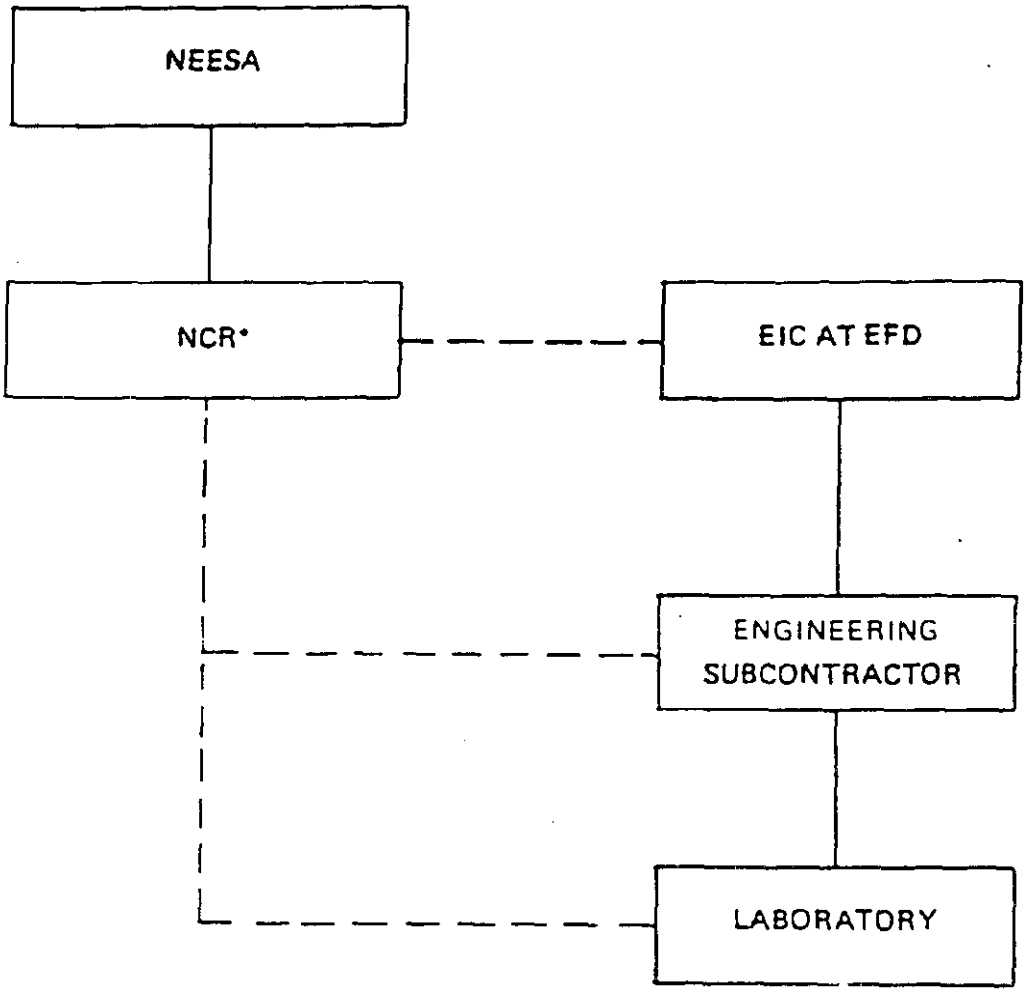

*NCR-MARTIN MARIETTA ENERGY SYSTEMS, INC.

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.

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.

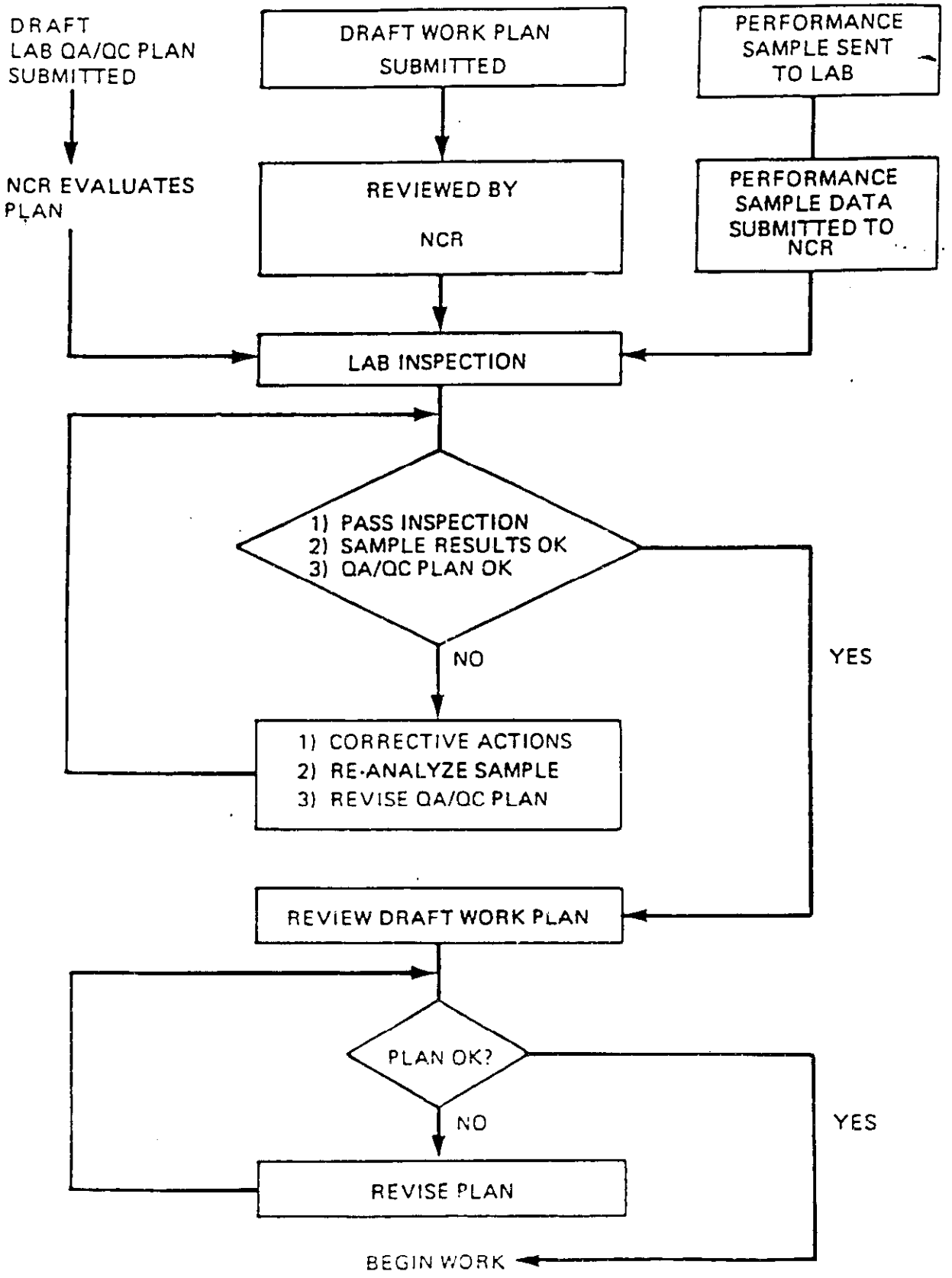

Fig. 2.1. Approval and Review Process

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.

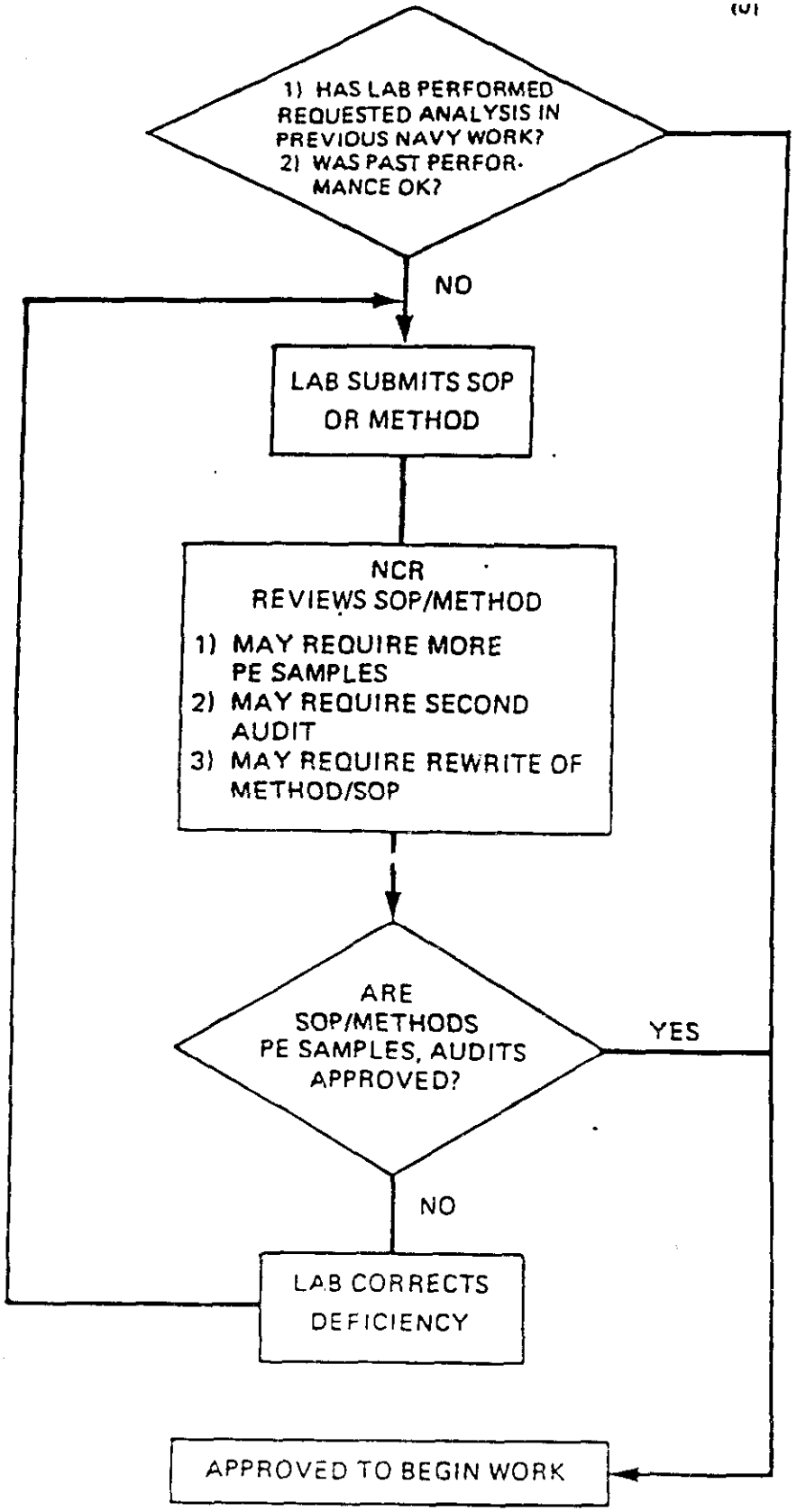

Fig. 2.2. Laboratory Reapproval Process

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]
[illegible]	[illegible]	[illegible]	[illegible]

[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	US
Bacteria					
1 Coliform (fecal) number per 100 ml	MPN, 5 tube, 3 dilution, or, membrane filter (MF) [4], single step	p. 132	908C		
		p. 124	909C		8-0055
2 Coliform (fecal) in presence of chlorine number per 100 ml	MPN, 5 tube, 3 dilution	p.132	908C		
3 Coliform (total), number per 100 ml	MPN, 5 tube, 3 dilution, or, MF [4] single step or two step	p. 114	906A		
		p. 108	906A		8-0025
4 Coliform (total) in presence of chlorine, number per 100 ml	MPN, 5 tube, dilution, or MF [4] with enrichment	p. 114	906A		
		p. 111	909 (A – A.5c)		
5 Fecal streptococci, number per 100 ml	MPN, 5 tube, 3 dilution, MF [4], or, plate count	p. 139	910A		
	p. 136	9108		8005S-77 [5]	
	p. 143	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 on environment.

[5] Incubate only a dilution of the KF Streptococcus Agar (Section 3.1, USGS Method 8-0055-77) a week in a boiling water bath to avoid scorching of the ...

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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 Peduction
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

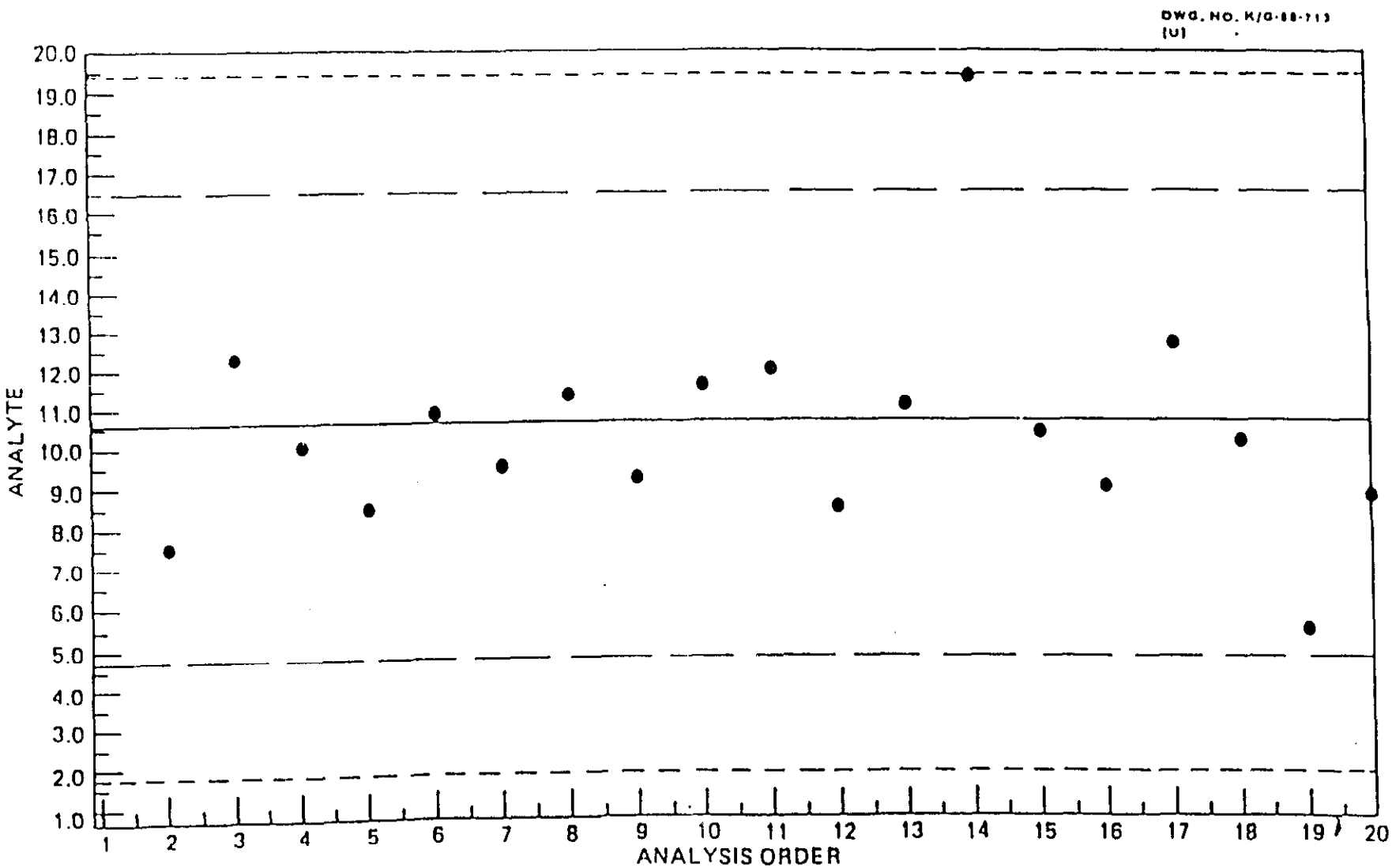

Fig. 4.1. Control Chart Example Data and Control Values of Table 4.3

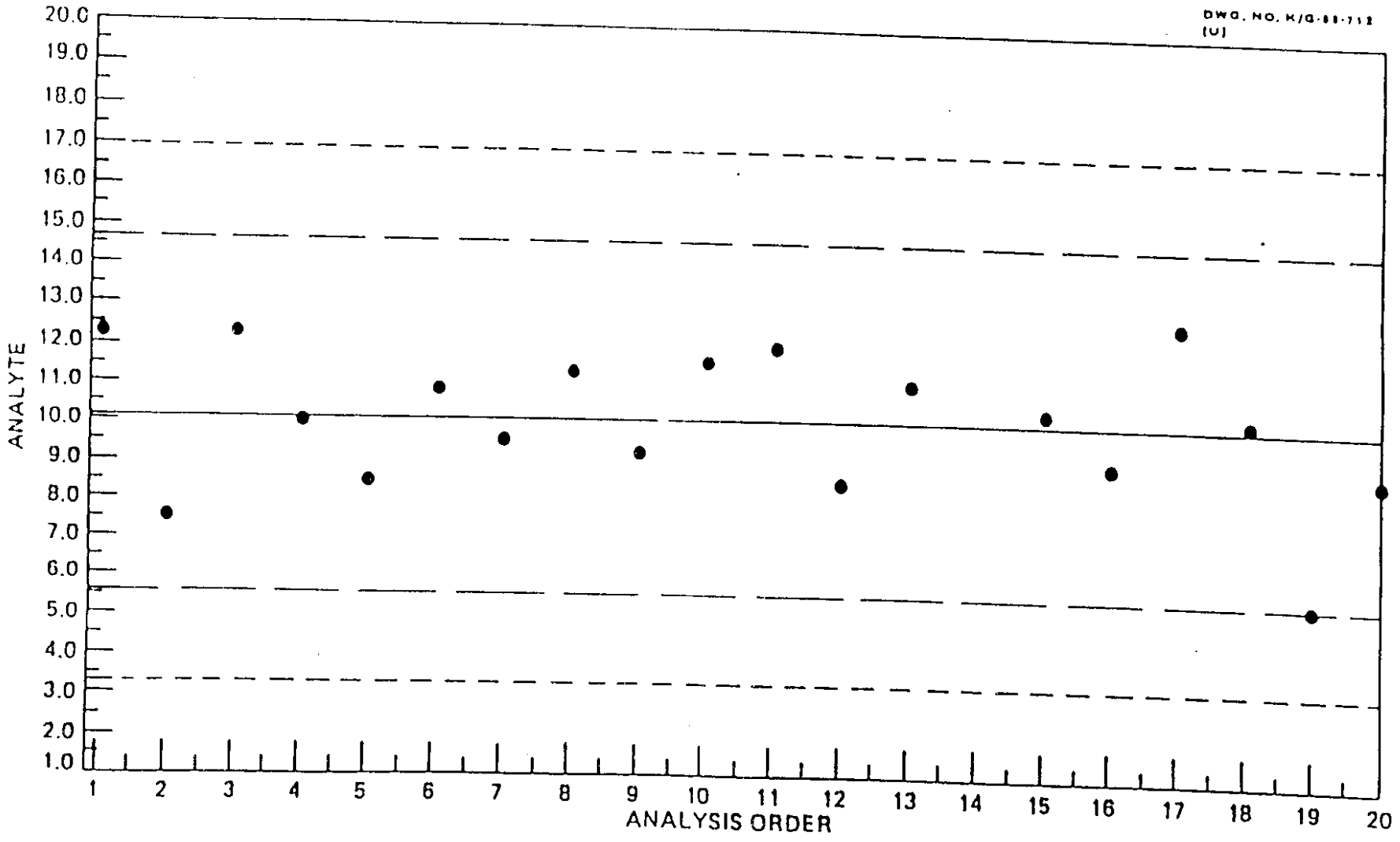

Fig. 4.2. Control Chart Example Data and Second Iteration Control Values of Table 4.3

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.

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]	EPA [1]	Reference (Method Number or Page)		
			Standard Methods 15th Ed	ASTM	USGS
Bacteria					
1. Coliform (fecal) number per 100 ml	MPN, 5 tubes, 3 dilutions, or, membrane filter (MF) [4], single step	p. 132	908C		
		p. 124	908C		8-0055-77
2. Coliform (fecal) in presence of chlorine number per 100 ml	MPN, 5 tubes, 3 dilutions	p. 132	908C		
3. Coliform (total), number per 100 ml	MPN, 5 tubes, 3 dilutions, or, MF [4] single step or two step	p. 114	906A		
		p. 108	906A		8-0025-77
4. Coliform (total) in presence of chlorine, number per 100 ml	MPN, 5 tubes, dilutions, or MF [4] with enrichment	p. 114	906A		
		p. 111	906 (A-A-5c).		
5. Fecal streptococci, number per 100 ml	MPN, 5 tubes, 3 dilutions, MF [4], or, plate count	p. 139	910A		
	p. 136	910B		80055-77 [6]	
	p. 143	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 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 cultivated, 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).	—	3068	—	—	
4 Ammonia (as N), mg/L	Manual distillation (at pH 9.5)[3], followed by	350.2	417A	—	—	33.057[1]
	Nesslerization,	350.2	417B	D1426-79(A)	I-3520-84	33.057[1]
	Titration,	350.2	417D	—	—	
	Electrode,	350.3	417E or F	D1426-79(D)	—	
	Automated phenate or	350.1	417G	D1426-79(C)	I-4523-84	
	Automated electrode.	—	—	—	—	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[1], p.17[3]
10 Boron—Total mg/L	Colorimetric (curcumin), or	212.3	404A	—	I-3112-84	
	Inductively Coupled plasma.	—	—	—	—	200.7[4]
11 Bromide mg/L	Titrimetric.	320.1	—	D1246-82(C)	I-1125-84	p.544[1]

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.e.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[3], 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[3]
	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[11]
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]

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	110.1	204D	—	—	Note 17
	(Platinum cobalt), or	110.2	204A	—	I-1250-84	
	Spectrophotometric.	110.3	204B	—	—	
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[J], p.37[4]
	AA furnace,	220.2	304	—	—	
	Inductively coupled plasma,	—	—	—	—	200.7[4]
	Colorimetric (Neocuproine), or	—	3138	D1688-84(A)	—	
	(Bicinchoninate).	—	—	—	—	Note 18
23. Cyanide—Total, mg/L	Manual distillation with $MgCl_2$ followed by	—	412B	—	—	
	Titrimetric, or	—	412C	—	—	p.22[4]
	Spectrophotometric, manual or	335.2	412D	D2036-82(A)	I-3300-84	
	Automated.[15]	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[4] followed by	—	413A	—	—	
	Electrode, manual or	340.2	413B	D1179-80(B)	—	
	Automated,	—	—	—	I-4327-84	
	Colorimetric (SPADNS) or Automated complexone,	340.1	413C	D1179-80(A)	—	
		340.3	413E	—	—	
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	130.2	3146	D1126-80	I-1338-84	33.082[J]
	Ca plus Mg as their carbonates, by inductively coupled plasma or AA direct aspiration. (See Parameters 13 and 33.)					
28. Hydrogen ion (pH), pH units	Electrometric measurement, or	150.1	423	D1293-84(A or B)	I-1586-84	33.006[J]
	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[J]
	AA furnace,	236.2	304	—	—	
	Inductively coupled plasma, or	—	—	—	—	200.7[4]
	Colorimetric (Phenanthroline).	—	3158	D1068-84(A)	—	Note 21

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	351.3	420A or B	D3590-84(A)	—	
	Titration,	351.3	417D	D3590-84(A)	—	33.051[1]
	Nesslerization,	351.3	417B	D3590-84(A)	—	
	Electrode,	351.3	417E or F	—	—	
	Automated phenate,	351.1	—	—	I-4551-78[7]	
	Semi-automated block digestor, or	351.2	—	D3590-84(A)	—	
	Potentiometric.	351.4	—	D3590-84(A)	—	
32. Lead—Total[2], mg/L	Digestion[3] followed by					
	AA direct aspiration,	239.1	303A or B	D3559-85(A or B)	I-3399-84	33.089[3]
	AA furnace,	239.2	304	—	—	
	Inductively coupled plasma,	—	—	—	—	200.7[4]
	Voltametry[10], or	—	—	D3559-85(C)	—	
	Colorimetric (Dithizone).	—	316B	—	—	
33. Magnesium—Total[2], mg/L	Digestion[3] followed by					
	AA direct aspiration,	242.1	303A	D511-84(B)	I-3447-84	33.089[3]
	Inductively coupled plasma, or	—	—	—	—	200.7[4]
	Gravimetric	—	318B	D511-77(A)	—	
34. Manganese—Total[2], mg/L	Digestion[3] followed by					
	AA direct aspiration,	243.1	303A or B	D858-84(B or C)	I-3454-84	33.089[3]
	AA furnace,	243.2	304	—	—	
	Inductively coupled plasma, or	—	—	—	—	200.7[4]
	Colorimetric (Persulfate), or	—	319[illegible]	D858-84(A)	—	33.126[3]
	(Periodate)	—	—	—	—	Note 22
35. Mercury—Total[2], mg/L	Cold vapor, manual or	245.1	303F	D3223-80	I-3462-84	33.095[3]
	Automated.	245.2	—	—	—	
36. Molybdenum—Total[2], 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[2], 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).	—	321B	—	—	
38. Nitrate (as N), mg/L	Colorimetric (Brucine sulfate), or	352.1	—	D992-71	—	33.063[3], 419D[11], p.28[8]
	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	—	—	—	

Table 7.2. (continued)

Parameter and Units	Method	Reference (Method No. or Page) EPA 1979	Std. Methods 16th Ed.	ASTM	USGS[1]	Other
40. Nitrite (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.4[33]
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[1]	33.028[1]
	Electrode.	360.1	421F	—	I-1576-78[1]	
47. Palladium—Total[3], mg/L	Digestion[3] followed by					
	AA direct aspiration, or	253.1	—	—	—	p.S27[4]
	AA furnace.	253.2	—	—	—	p.S28[4]
48. Phenols, mg/L	Manual distillation[23] followed by	420.1	—	D1783-80(A or B)	—	Note 26
	Colorimetric (4AAP) manual, or	420.1	—	—	—	Note 26
	Automated[13].	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	—	3228	D1428-82(A)	—	
	Colorimetric (Cobaltinitrite).	—	—	—	—	3178[11]

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[2], mg/L	Digestion[3] followed by					
	AA direct aspiration, or	265.1	303A	—	—	
	AA furnace.	265.2	304	—	—	
59. Ruthenium—Total[2], mg/L	Digestion[3] followed by					
	AA direct aspiration, or	267.1	303A	—	—	
	AA furnace.	267.2	304	—	—	
60. Selenium—Total[2], 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 (Molybdosilicate), or	—	—	—	I-2700-84	
	Inductively coupled plasma.	—	—	—	—	200.7[4]
62. Silver—Total[2,4], mg/L	Digestion[3] followed by					
	AA direct aspiration,	272.1	303A or B	—	I-3720-84	33.069[5], p.37[6]
	AA furnace,	272.2	304	—	—	
	Colorimetric (Dithizone), or	—	—	—	—	3198[11]
	Inductively coupled plasma.	—	—	—	—	200.7[4]
63. Sodium—Total[2], mg/L	Digestion[3] followed by					
	AA direct aspiration,	273.1	303A	—	I-3735-84	33.107[5]
	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[5]
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[5]
	Turbidimetric.	375.4	—	D516-82(B)	—	426C[10]

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[4]
	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.

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.

[a]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.

[b]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.

[c]Ammonia, Automated Electrode Method, Industrial Method Number 379-75 WE, dated February 19, 1976, Technicon AutoAnalyzer II, Technicon Industrial Systems, Tarrytown, NY, 10591.

[d]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).

[e]American National Standard on Photographic Processing Effluents, Apr. 2, 1975. Available from ANSI, 1430 Broadway, New York, NY 10018.

[f]"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).

[g]The use of normal and differential pulse voltage ramps to increase sensitivity and resolution is acceptable.

[h]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.

[i]OIC Chemical Oxygen Demand Method, Oceanography International Corporation, 512 West Loop, P.O. Box 2980, College Station, TX 77840.

[j]*Chemical Oxygen Demand, Method 8000, Hach Handbook of Water Analysis*, 1979, Hach Chemical Company, P.O. Box 389, Loveland, CO 80537

[k]The back titration method will be used to resolve controversy.

[l]Orion Research Instruction Manual, Residual Chlorine Electrode Model 97-70, 1977, Orion Research Incorporated, 840 Memorial Drive, Cambridge, MA 02138.

[m]The approved method is that cited in *Standard Methods for the Examination of Water and Wastewater*, 14th Edition, 1976.

[n]National Council of the Paper Industry for Air and Stream Improvement, (Inc.) Technical Bulletin 253, December 1971.

[o]Copper, Bicinchoninate Method, Method 8506, Hach Handbook of Water Analysis, 1979, Hach Chemical Company, P.O. Box 389, Loveland, CO 80537

[p]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.

[q]Hydrogen Ion (pH) Automated Electrode Method, Industrial Method Number 378-75WA, October 1976, Technicon Auto-Analyzer II, Technicon Industrial Systems, Tarrytown, NY 10591.

[r]Iron, 1,10-Phenanthroline Method, Method 8008, 1980, Hach Chemical Company, P.O. Box 389, Loveland, CO 80537.

[s]Manganese, Periodate Oxidation Method, Method 8034, Hach Handbook of Wastewater Analysis, 1979, pages 2-113 and 2-117, Hach Chemical Company, Loveland, CO 80537.

[t]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).

[u]Nitrogen, Nitrite, Method 8507, Hach Chemical Company, P.O. Box 389, Loveland, CO 80537.

[v]Just prior to distillation, adjust the sulfuric-acid-preserved sample to pH 4 with 1 + 9 NaOH.

[w]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.

[x]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.

[y]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.

[z]The approved method is that cited in *Standard Methods for the Examination of Water and Wastewater*, 15th Edition.

[aa]The approved method is that cited in *Standard Methods for the Examination of Water and Wastewater*, 13th Edition.

[bb]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.

[cc]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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ble List of approved test procedures for
onpesticide 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. S102.
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 Dibenz(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	604	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. S102.
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.
2-Methyl-4,6-Dinitrophenol	604	625, 1625		
Naphthalene	610	625, 1625		
Nitrobenzene	609	625, 1625		
2-Nitrophenol	604	625, 1625		
4-Nitrophenol	604	625, 1625		
N-Nitrosodimethylamine	607	625, 1625		
N-Nitrosodi-n-propylamine	607	*625, 1625		
N-Nitrosodiphenylamine	607	*625, 1625		
1,2-Diphenylhydrazine	611	625, 1625		
PCB-1016	608		625.	Note 3, p. 43.
PCB-1221	608		625.	Note 3, p. 43.
71 PCB-1232	608		625.	Note 3, p. 43.
72 PCB-1242	608		625.	Note 3, p. 43.
73 PCB-1248	608		625.	Note 3, p. 43.
PCB-1254	608		625.	Note 3, p. 43.
PCB-1260	608		625.	Note 3, p. 43.
Pentachlorophenol	604	625, 1625		Note 3, p. 140.

Table 7.3. (continued)

Parameter [1]	EPA Method Number [2,3]			Other
	GC	GC/MS	HPLC	
64. Phenanthrene	610	625, 1625	610	
65. Phenol	604	625, 1625		
66. Pyrene	610	625, 1625	610	
67. 2,3,7,8-Tetrachlorodibenzo-p-dioxin		[4] 613		
68. 1,1,2,2-Tetrachloroethane	601	624, 1624		Note 3, p. 130;
69. Tetrachloroethene	601	624, 1624		Note 3, p. 130;
70. Toluene	602	624, 1624		
71. 1,2,4-Trichlorobenzene	612	625, 1625		Note 3, p. 130;
72. 1,1,1-Trichloroethane	601	624, 1624		
73. 1,1,2-Trichloroethane	601	624, 1624		Note 3, p. 130;
74. Trichloroethene	601	624, 1624		
75. Trichlorofluoromethane	601	624,		
76. 2,4,6-Trichlorophenol	604	625, 1625		
77. Vinyl Chloride	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, 1978.

[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 the 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."

Table 7.4. List of approved test procedures for pesticides[1]
(40 CFR, Part 136, July 1, 1987)

Parameter µg/L	Method	EPA[1,1]	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 S64
3. Aminocarb	TLC				Note 3, p 84; Note 6, p S16.
4. Alachlor	GC				Note 3, p 83, Note 6, p S64.
5. Atrazine	GC				Note 3, p 83, Note 6, p S64
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 84; Note 6, p S60
14. Carbofenothion	GC				Note 4, p 30; Note 6, p S73.
15. Chlordane	GC	608	509A	D3086	Note 3, p 7
	GC/MS	625			
16. Chlorpyrifos	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			
	GC				Note 4, p 30, Note 6, p S73
43. Linuron	TLC				Note 3, p 104, Note 6, p S64
44. Malathion	GC		509A		Note 3, p 25, Note 4, p 30 Note 6, p S51
45. Methiocarb	TLC				Note 3, p 84 Note 6 p S60
46. Methoxychlor	GC		509A	D3086	Note 3, p 7, Note 4, p 30
47. Mexacarbate	TLC				Note 3, p 84, Note 6, p S60
48. Mirex	GC		509A		Note 3, p 7
49. Monuron	TLC				Note 3, p 104, Note 6, p S64
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	GC		509A		Note 3, p 25, Note 4, p 30
53. Parathion ethyl	GC		509A		Note 3, p 25
54. PCNB	GC		509A		Note 3, p 7
55. Perthane	GC			D3086	
56. Prometon	GC				Note 3, p 83 Note 6, p S64
57. Prometryn	GC				Note 3, p 83 Note 6, p S64
58. Propazine	GC				Note 3, p 83 Note 6, p S64
59. Propham	TLC				Note 3, p 104, Note 6, p S64
60. Propoxur	TLC				Note 3, p 84 Note 6 p S60
61. Secbumeton	TLC				Note 3, p 83 Note 6, p S64
62. Simazine	TLC				Note 3, p 104 Note 6 p S64
63. Simetryn	GC				Note 3, p 83 Note 6 p S64
64. Swoden	GC		509A		Note 3, p 7

Table 7.4. (continued)

Parameter µg/L	Method	EPA[1]	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[5]
68 Terbuthylazine	GC				Note 3, p 80, 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 7D 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 name.

[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 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, 1978. The 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, β-BHC, endosulfan I, endosulfan II, and endrin. However, when they are known to exist, Method 608 is the preferred method.

[6] "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).

[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) in 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 with 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 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."

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 [3]
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 [3]
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 7.5 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).
[3] 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."

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/semi-volatiles. 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

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.

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

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.

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.

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.

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

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