DEPARTMENT OF THE NAVY
CRANE DIVISION
NAVAL SURFACE WARFARE CENTER
300 HIGHWAY 361
CRANE, INDIANA 47522-5000

IN REPLY REFER TO:

5090
Ser 095/9073

29 MAR 1999

U.S. Environmental Protection Agency, Region V
Waste, Pesticides, & Toxics Division
Waste Management Branch
Illinois, Indiana, and Michigan Section
ATTN: Ms. Carol Witt-Smith (DRP-8J)
77 West Jackson Blvd.
Chicago, IL 60604

Dear Ms. Witt-Smith:

Tetra Tech NUS, Inc. has advised Crane Division, Naval Surface Warfare Center (NAVSURFWARCENDIV Crane) that some pages for the Response to 12/2/98 and 12/24/98 Notice of Deficiency, submitted to your office March 18, 1999, were inadvertently omitted. Enclosure (1) contains the replacement pages and instructions. Enclosure (2) is the required Certification Statement.

NAVSURFWARCENDIV Crane point of contact is Mr. Thomas J. Brent, Code 09510, telephone 812-854-6160.

Sincerely,

Phillip B. Keith, for

for J. M. HUNSICKER
Director, Environmental Protection Department
By direction of
the Commander

Encl:
(1) Change Pages for the March 18, 1999 Submittal
(2) Certification Statement

Copy to:
ADMINISTRATIVE RECORD (w/2 copies)
COMNAVSEASYSCOM (SEA OOT) (w/o encl)
SOUTHNAVFACENGCOM (Code 1864) (w/o encl)
IDEM (Michelle Timmermann) (w/1 copy)
TETRA TECH (Ralph Basinski) (w/o encl)

I certify under penalty of law that this document and all attachments were prepared under my direction or supervision in accordance with a system designed to assure that qualified personnel properly gather and evaluate the information submitted. Based on my inquiry of the person or persons who manage the system, or those persons directly responsible for gathering the information, the information submitted is, to the best of my knowledge and belief, true, accurate, and complete. I am aware that there are significant penalties for submitting false information, including the possibility of fine and imprisonment for knowing violations.

Philip A. Keith, acting

SIGNATURE

JAMES HUNSICKER

DIRECTOR, ENVIRONMENTAL PROTECTION DEPARTMENT

TITLE

29 MAR 1989

DATE

Enclosure (2)

TETRA TECH NUS, INC.
661 Andersen Drive ▪ Pittsburgh, Pennsylvania 15220-2745
(412) 921-7090 ▪ FAX (412) 921-4040 ▪ www.tetratech.com

PITT 03-9-158

March 23, 1999

Project Number 7651

Commanding Officer
Department of the Navy
SOUTHNAVFACENGCOM
Attn: Mr. E. P. Johns (Code 1829)
2155 Eagle Drive
North Charleston, South Carolina 29406

Reference: CLEAN Contract No. N62472-90-D-1298
Contract Task Order 0038

Subject: Draft Final Quality Assurance Project Plan for RCRA Ground Water Monitoring at the Ammunition Burning Grounds, Old Rifle Range, and Demolition Range

Dear Mr. Johns:

Our submittal sent to you on March 15, 1999, included responses to U.S. EPA Notices of Deficiency dated December 12, 1998, and December 24, 1998, on the NSWC Crane Quality Assurance Project Plan. It has come to my attention that some pages of one of the standard operating procedures (SOPs) included as an attachment to the responses were inadvertently omitted. Enclosed for replacement is another copy of the SOP.

Please replace SOP LTL-8330 Revision 9, which is included as Attachment F-4 of the section that is tabbed "Attachments 11-18-98 Comments", with the enclosed copy of SOP LTL-8330 Revision 9.

Copies for U.S. EPA and IDEM have been sent to Tom Brent. It is our understanding that Tom Brent will distribute two copies of the enclosures to U.S. EPA and one copy to IDEM.

Please contact Daneen Resnick (412-921-8714, e-mail resnickd@ttnus.com) or Ralph Basinski (412-921-8308, e-mail basinskir@ttnus.com) regarding any questions or comments.

Sincerely,

Daneen C. Resnick
for
Ralph R. Basinski
Task Order Manager

RRB/vvp

Enclosure

c: Tom Brent, NSWC Crane (4 copies)
Ms. D. Wroblewski, w/o enclosure
Mr. S. Ruffing, w/o enclosure
Mr. M. Perry, w/enclosure (file copy)

Enclosure (1)

LAUCKS TESTING LABORATORIES INC.

Seattle, Washington

SOP #:LTL-8330

Determination of Nitroaromatics and Nitramines by SW-846 Method 8330

Revision history:

Number	Date
1	06/19/95
2	01/11/96
3	04/10/96
4	04/15/96
5	07/09/96
6	12/18/97
7	4/30/98
8	10/28/98
9	01/25/99

UNCONTROLLED

Revised by: Monica Carr, Organic Division Manager — Date: 1/25/99

Approved by: Harry Romberg, QA Officer — Date: 1-25-99

Approved by: Kathy Kreps, Laboratory Director — Date: 1-25-99

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1. Introduction and Scope

1.1 Method Description

1.1.1 This method is used for the trace analysis of ordnance compounds in water, soil, and sediment samples by high performance liquid chromatography. The concentrated water sample extracts are diluted 1:1 (v/v) with reagent grade water prior to analysis. The sample extracts are analyzed using a C18 (octadecyl) reverse phase column, and target analyte concentrations are measured at either 254 nm or 210 nm using a UV detector. All positive measurements observed on the C18 column are confirmed by a second analysis which uses a CN (cyano) column. The C18 column is considered the primary column and is used for quantitation of all target analytes.

1.1.2 This method is used to determine part per billion levels of the ordnance analytes listed below:

Compound	Acronym
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine	HMX
Hexahydro-1,3,5-trinitro-1,3,5-triazine	RDX
1,3,5-Trinitrobenzene	1,3,5-TNB
1,3-Dinitrobenzene	1,3-DNB
Methyl-2,4,6-trinitrophenylnitramine	Tetryl
Nitrobenzene	NB
2,4,6-Trinitrotoluene	2,4,6-TNT
4-Amino-2,6-dinitrotoluene	4-Am-DNT
2-Amino-4,6-dinitrotoluene	2-Am-DNT
2,4-Dinitrotoluene	2,4-DNT
2,6-Dinitrotoluene	2,6-DNT
2-Nitrotoluene	2-NT
3-Nitrotoluene	3-NT
4-Nitrotoluene	4-NT

6 Additional Compounds
(Separate Analysis at 254 nm)

Compound	Acronym
2,4-Diamino-6-nitrotoluene	2,4-DA-6-NT
2,6-Diamino-4-nitrotoluene	2,6-DA-4-NT
3,5-Dinitroaniline	3,5-DNA
1-Nitroso-3,5-dinitro-1,3,5-hexahydrotriazine	MNX
1,3,5-Trinitroso-1,3,5-hexahydrotriazine	TNX
2,2',6,6'-Tetranitro-4,4'-azoxytoluene	4,4'-TN-AZOXY

PETN/NG Compounds
(Separate Analysis at 210 nm)

Compound	Acronym
Nitroglycerin	NG
Pentaerythritoltetranitrate	PETN

1.1.3 Aqueous samples of higher concentration can be directly analyzed by diluting 1:1 (v/v) with methanol or acetonitrile, filtering, separating on a C18 reverse phase column, and determined at either 254 nm or 210 nm, and confirmed on a CN column.

1.1.4 This method is restricted to use by, or under the supervision of, analysts experienced in the use of high pressure liquid chromatography and in the interpretation of chromatograms. Each analyst performing this method must have demonstrated the ability to perform the described chromatographic analysis and/or data interpretation.

1.2 Method Deviations & Comments

1.2.1 The following items represent deviations from the published version of method SW 8330 which are followed as standard operating procedure in the performance of this method at Laucks.

1. Single injections of calibration standards are analyzed rather than triplicate injections in a random order. It has been determined at Laucks that single injections of these standards yield acceptable calibration and linearity data as evidenced by the calculated percent RSDs used to evaluate the initial calibration data.

2. Method SW 8330 specifies that all analyses are to be performed using a mobile phase which consists of a 50:50 mixture of methanol/water under

isocratic conditions. Laucks employs a gradient elution program (detailed in Section IV) in order to improve the separation of the target analytes on the CN column.

3. According to Method 8330, all working standards are to be prepared daily in a methanol/calcium chloride solution. The practice at Laucks for 8330 and the 6 additional compounds is to prepare working standards using acetonitrile and diluting 50/50 with water just prior to analysis. NG/PETN are prepared in a 50/50 mixture of acetonitrile and water. These working solutions have been demonstrated to be stable for at least 6 months. The stability of target analyte responses in the working solutions in use (especially that of tetryl) is used to determine whether new solutions should be prepared.

4. Tetryl decomposes rapidly in methanol/water solutions, as well as with heat. Laucks has experienced erratic recovery of tetryl from spiked sample extracts.

5. The confirmation column demonstrates full or partial coelution for the following target analyte pairs: 1,3-dinitrobenzene and 1,3,5-trinitrobenzene; 2-nitrotoluene, 3-nitrotoluene and 4-nitrotoluene; and 2,4-dinitrotoluene, 2,6-dinitrotoluene and 2,4,6-trinitrotoluene. Therefore, positive confirmation can not be made when two or more co-eluting peaks have been tentatively identified on the primary column.

6. At the time of this writing, analysis of the 6 additional compounds (not including PETN/NG) combined with the 14 ordnance compounds results in the co-elution of 2,6-DA-4-NT and HMX; and 3,5-DNA and tetryl on the C18 column using the 8330 method. However, tetryl and HMX do not co-elute with any attenuation compound on the CN column using the 8330 method. These two compounds (HMX and tetry) do not co-elute with any of the attenuation compounds on the primary colum nor confirmation column using the attenuation method.

7. Although the compounds NG and PETN are not listed in SW 846 Method 8330, Laucks has found that adequate recovery of these compounds can be achieved using this method by modifying the wavelength from 210 nm to 254 nm.

8. Picric and picramic acids exhibit substantial peak shift and may elute near or co-elute with HMX on the C18 column using the 8330 method. However, no attenuation compounds, nor NG/PETN coleute on the primary (PAH)

column using the picric/picramic method. The buffer is used in this method in order to stablize these compounds.

1.2.2 In order to attain lab-wide consistency among staff members for decision-making processes with regard to laboratory anomalies, several common items have been addressed in this SOP. Any occurrences which are not covered in this SOP should be discussed with the supervisor, prior to implementing a solution.

1.2.3 One example is the determination of potential carry-over in sample analyses. Any samples analyzed subsequent to a high level sample (which is defined by yielding one or more target analytes above the calibration range) should be thoroughly examined for potential carry-over of the same target analyte(s). Corrective action in the form of reanalysis for possible carry-over should be performed and documented in the narrative.

1.2.4 Another example is the review of all sample chromatograms for analytes which may not show up on the sample quantitation report due to data system error or retention time shift. All peaks should be examined and evaluated based on the retention times and sample concentration in order to prevent reporting false negatives.

1.3 Sample Collection, Sample Storage, Holding Times

1.3.1 Samples are collected in amber glass containers with Teflon-lined caps. All samples and sample extracts are stored at 4°C ±2°C. According to guidelines listed in SW-846 for the extraction of semivolatile compounds, water samples must be extracted within 7 days of collection, and soil samples must be extracted within 14 days of collection. All extracts must be analyzed within 40 days of sample preparation.

1.3.2 Although not a routine sampling practice, a soil holding time study performed by the U.S. Army Cold Regions Research and Engineering Laboratory recommends a maximum holding time of eight weeks when samples are frozen.

1.3.3 A similar study performed for water samples recommends a maximum holding time of 50 days for relatively sterile samples which are refrigerated. Surface waters, or samples likely to have significant microbial activity, may suffer significant losses of nitroaromatic compounds (particularly trinitrobenzene and trinitrotoluene) within 1-2 days, even under refrigeration. If microbial activity is suspected, water samples should be extracted as soon after collection as is practical.

1.4 Definition of Terms

1.4.1 This section defines terms and acronyms as they are used in this SOP. Other terms, such as MS/MSD or method blank, are not defined here since it is assumed that the user of this SOP already understands their more general meaning.

Method Blank Spike	A background free matrix (DIW for water, clean sand for soils/sediments) to which known amounts of target analytes and surrogates are added each time sample extracts are prepared. In the context of this SOP, a blank spike is the same as a QC check standard. See also QC check standard.
CCV	Continuing calibration verification. This is the same acronym used in the CLP program. This is a standard injected at some prescribed frequency during the analysis sequence to determine whether the instrument has remained in calibration.
CF	Calibration factor. The ratio of peak response to nanograms injected. This term is defined in the same way in both the CLP contract and SW-846.
DIW	Deionized water. Lab reagent water. Organic-free water. Since the systems used to provide DIW at Laucks all contain carbon polishing filters, they are capable of providing organic-free water for use in method blanks and method blank spikes.
IBLK	An instrument blank is solvent containing the method surrogates and is injected into the instrument to monitor for carryover between sample extract injections.
ICV	Initial calibration verification. It is a standard which is injected at the start of each QC period that is compared to the initial multi-point calibration to determine whether the instrument is still in calibration.
IDL	Instrument detection limit. The lowest concentration of a target analyte that will yield a signal:noise ratio of at least 3x. Used as a starting point for selecting MDL study spiking levels.

MDL	Method detection limit. The lowest concentration in a sample which will yield a positive result that is greater than zero at a known level of confidence. MDLs are empirically determined at Laucks.
QC Check Standard	Quality control check standard. Referred to in this SOP as a blank spike. A QC check standard is a requirement of SW-846 method 8000 and is used to determine whether the analytical system is in control if MS/MSD recoveries are out of control. See also blank spike.
QC Period	Quality control period. An analysis sequence initiated by the injection of one or more standards, followed by sample extracts, and terminated with a standard injection. A QC period can be open-ended chronologically, but calibration verification must be documented using the procedures in this SOP.
RSD or %RSD	Relative standard deviation or percent relative standard deviation. The ratio of the standard deviation of a set of values to the mean of the set of values. A measure of the similarity of the values one to another.
RT	Retention time. The time (in minutes) at which a target analyte elutes from the LC column.
RT Window	Retention time window. The ± value which is applied to the ICV to establish the time range used to make tentative compound identifications.
Sequence	A set of sample extracts and standard solutions injected into an instrument in a chronologically continuous group. See also QC period.

2. Equipment List and Standards

2.1 Chromatographic System

Primary Column:	C18 (octadecyl) reverse phase HPLC column, 15 cm x 4.6 mm, 5 µm particle size, (Rainin Microsorb or equivalent).

Secondary Column:	CN (cyano) reverse phase HPLC column, 25 cm x 4.6 mm, 5 µm particle size, (Supelcosil LC-CN or equivalent). C8 and CN reverse phase HPLC columns in series, 10cm x 3.9mm, 4µm particle size, (Waters Nova- Pak or equivalent). These are used in the for the analysis of the 6 additional compounds.
Mobile Phase:	Methanol (EM Science brand high purity solvent or equivalent). Reagent water (Modulab Polisher HPLC grade water or equivalent).
UV Detector:	254 nm (210 nm for NG and PETN)
HPLC System:	Rainin HPLC system - HPXL solvent delivery system capable of achieving 4000 psi. 50 µL sample loop. Knauer variable wavelength UV detector. Dynamax automatic sample injector. Digital integrator: EZChrom.
Waters System:	Waters 712 WISP Sample Processor or equivalent. 50 µL sample loop or equivalent. Waters 486 tunable absorbance detector or equivalent. Waters 600E Multisolvent delivery system or equivalent. Digital Integrator: EZChrom.
Hewlett Packard System:	HP 1313A Autosampler. HP G1311A QuatPump. HP G1322A On-line degasser. HP G1316A Thermostat column compartment. HP 1314A Variable wavelength detector. Digital Integrator: EZChrom.

2.1.1 Column Temperature Control:

2.1.2 Column temperature is controlled through the use of a column heater which is maintained at a temperature of 25°C.

2.2 Standards

2.2.1 Target Analyte Stock Solution

2.2.1.1 The stock solution used contains the 14 standard compounds in the mix at a concentration of 1000 µg/mL and is generally purchased from AccuStandard Inc. (25 Science Park, New Haven CT 06511). Equivalent solutions from this or other vendors are also acceptable.

2.2.1.2 The NG and PETN stock solutions are purchased individually from AccuStandard, Inc. (25 Science Park, New Haven, CT 06511). The NG solution comes in 1 mL ampules at 4000 µg/mL in ethanol. The PETN solution comes in 1 mL ampules at 1000 µg/mL in methanol. Equivalent solutions from this or other vendors are also acceptable.

2.2.1.3 The 3,5-Dinitroaniline and 1,3,5-Trinitroso-1,3,5-hexahydrotriazine standards are made from standard analytical reference materials obtained from the U.S. Army Environmental Center (Aberdeen Proving Ground, MD 21010). The 2,4-Diamino-6-nitrotoluene, 2,6-Diamino-4-nitrotoluene and 2,2',6,6'-Tetranitro-4,4'-azoxytoluene standards are made from standard analytical reference materials obtained from AccuStandard, Inc. (25 Science Park, New Haven, CT 06511). The 1-Nitroso-3,5-dinitro-1,3,5-hexahydrotriazine standards are made from standard analytical reference materials obtained from Stanford Research.

2.2.1.4 Calibration standard, surrogate stock and IBLK surrogate solutions are detailed in Appendix I.

2.2.2 Surrogate Stock Solution

2.2.2.1 The surrogate stock solution is received in a methanolic solution at a concentration of 1000 µg/mL. The surrogate can also be prepared by weighing 100 mg of 1,2-dinitrobenzene into a 100 mL volumetric flask and diluting to volume with methanol to yield a concentration of 1000 µg/mL.

2.2.3 IBLK Working Solution

2.2.3.1 The working instrument blank solution contains the surrogate compound only at a concentration of 2.0 µg/mL. Prepare this solution by adding 100 µL of the 1000 µg/mL surrogate stock solution to a 50 mL volumetric flask. Dilute to volume so that the final solvent concentration is methanol/water at a ratio of 1:1.

2.2.4 Working Calibration Standards

2.2.4.1 Prepare the working calibration standards for the 14 standard analytes in the following manner. All solutions are prepared in acetonitrile and diluted 1:1 with water just prior to analysis. These 14 analytes are combined into one working solution, since they do not co-elute on the primary column.

Standard	Source Solution	Amount Added (mL)	Final Volume (mL)	Final Concentration (µg /mL)
Standard #6	A	0.20	10	20.0
	B	0.20		
Standard #5	A	0.1	10	10.0
	B	0.1		
Standard #4	C	1.0	10	2.0
Standard #3	D	1.0	10	1.0
Standard #2	E	1.0	10	0.2
Standard #1	F	1.0	10	0.1

Source Solution	Concentration (µg /mL)
A = 14 Component Mix	1000
B = Surrogate Stock Solution	1000
C = Standard #6	20
D = Standard #5	10
E = Standard #4	2.0
F = Standard #3	1.0

2.2.4.2 Prepare the working calibration standards for the 6 additional analytes in the following manner. All solutions are prepared in acetonitrile and diluted 1:1 with water just prior to analysis.

Standard	Source Solution	Amount Added (mL)	Final Volume (mL)	Final Concentrations (µg /mL)
Standard #6	A	2.0	10	20.0
	B	0.2		
	C	0.2		
	D	0.2		
	E	0.2		
	F	2.0		
	G	0.2		
Standard #5	H	5.0	10	10.0
Standard #4	H	1.0	10	2.0
Standard #3	I	1.0	10	1.0
Standard #2	J	1.0	10	0.2
Standard #1	K	1.0	10	0.1

Source Solution	Concentration (µg/mL)
A = MNX	100
B = TNX	1000
C = 3,5-DNA	1000
D = 2,6-DA-4-NT	1000
E = 2,4-DA-6-NT	1000
F = 4,4'-TN-AZOXY	100
G = 1,2-DNB	1000
H = Standard 6	20.0
I = Standard 5	10.0
J = Standard 4	2.0
K = Standard 3	1.0

2.2.4.3 Prepare the working calibration standards for PETN/NG in the following manner. All solutions are prepared in a 1:1 acetonitrile/water mixture.

Standard	Source Solution	Amount Added (mL)	Final Volume (mL)	Final Concentrations NG:PETN:1,2-DNB (µg /mL)
Standard #5	A	0.05	10	20.0
	B	0.10	10	10.0
	C	0.10	10	10.0
Standard #4	D	5.0	10	10.0, 5.0, 5.0
Standard #3	E	5.0	10	5.0, 2.5, 2.5
Standard #2	F	2.0	10	1.0, 0.5, 0.5
Standard #1	G	5.0	10.0	0.5, 0.25, 0.25

Source Solution	Concentration (µg/mL)
A = Nitroglycerin	4000
B = PETN Solution	1000
C = Surrogate Stock Solution	1000
D = Standard 5	As Above
E = Standard 4	As Above
F = Standard 3	As Above
G = Standard 2	As Above

2.2.4.4 Note: Store all standard and surrogate solutions in amber glass vials with screw caps and Teflon-lined septa. Minimize headspace in these vials and store them in a refrigerator kept at 4°C ±2°C. Allow standards to come to room temperature prior to use.

2.2.4.5 Stock solutions may be used for up to one year, and working solutions are good for at least six months, or the expiration date of the parent standard, whichever is sooner.

2.2.4.6 All standards are assigned a unique identifier to enable cross-referencing of each individual standard back to the supplier's lot number. In addition, all standards are labeled with the standard concentration, the solvent, date prepared, expiration date, analyst's initials, and the standard reference number. Refer to Laucks' SOP on the traceability, documentation, and preparation standards.

3. Safety precautions

3.1 Routine Safety Precautions

3.1.1 All standards and sample extracts should be handled as if they are hazardous substances.

3.1.2 All compounds analyzed by this method are used either in the manufacture of explosives or are the degradation products of these compounds. When making stock solutions for calibration, treat each explosive compound with caution.

3.1.3 Refer to the instrument manufacturer's manual for routine instrument precautions.

3.1.4 Routine precautions include an awareness of the moving parts on the instrument you're using. These parts are often charged with power from an electrical component or with high pressure gas and have the potential to do harm if not used properly.

3.1.5 Electrical shock - All instruments present the possibility of electrical shock. The operator should take all precautions including ensuring that all instruments are operated with fully grounded power outlets, turning off the instrument and disconnecting the instrument from the electrical power supply before working on any electrical components, etc.

3.1.6 Almost all of the analytes under consideration are known or suspected carcinogens. Analysts should wash their hands after using any standard, solvent or sample extract. Additionally, a respirator should be worn or a fume hood utilized for extremely hazardous compounds or very dirty extracts.

3.2 Waste disposal

3.2.1 Out of date standards and sample extracts are disposed of in the designated organic waste container located in the organic preparation area.

3.2.2 HPLC liquid waste is disposed of by pouring into the designated organic waste container located in the solvent storage area.

4. Operation procedures

4.1 Analytical Conditions

4.1.1 Current C18 Column Conditions for the 14 Compounds:

HPLC3		HPLC4	
Pump A Solvent:	Methanol	Solvent:	Methanol/Water
Pump B Solvent:	Water	Flow:	0-24 min = 0.5 mL/min
Flow:	1.0 mL/minute:		24-35 min = 0.8 mL/min
Gradient:	Linear		35-40 min = 0.5 mL/min
Run Time:	60 minutes	Run Time:	40 minutes
Acquisition Time:	60 minutes	Acq. Time:	35 minutes
Injection Volume:	50 µL inj.	Time:	50 µL

Gradient Profile (HPLC3)		Run Time Profile
Initial %A:	40	
Initial %B:	60	
Hold Time:	5 minutes	0 - 5 minutes
Gradient 1 %A:	60	
Gradient 1 %B:	40	
Ramp Time 1:	15 minutes	
Hold Time 1 :	5 minutes	5 - 20 minutes
Gradient 2 %A:	95	
Gradient 2 %B:	05	
Ramp Time 2:	10 minutes	20 - 30 minutes
Hold Time 2:	5 minutes	30 - 35 minutes
Gradient 3 %A:	40	
Gradient 3 %B:	60	
Ramp Time 3:	5 minutes	35 - 40 minutes
Hold Time 3:	20 minutes	40 - 60 minutes (Column Equilibration)

4.1.2 Current CN Column Conditions for the 14 Compounds:

HPLC2 File #6:

Pump A Solvent:	Methanol	
Pump B Solvent:	Water	
Flow:	1.0 mL/minute	
Gradient:	Linear	
Run Time:	50 minutes	
Acquisition Time:	25 minutes	
Initial %A	25	
Initial %B	75	
Gradient 1 %A:	45	
Gradient 1 %B:	55	
Ramp Time 1:	2 minutes	0 - 2 minutes
Hold Time 1 :	3 minutes	2 - 5 minutes
Gradient 2 %A:	60	
Gradient 2 %B:	40	
Ramp Time 2:	5 minutes	5 - 10 minutes
Hold Time 2 :	8 minutes	10 - 18 minutes
Gradient 3 %A:	25	
Gradient 3 %B:	75	
Ramp Time 3:	7 minutes	18 - 25 minutes
Hold Time 3 :	25 minutes	25 - 50 minutes

4.1.3 Current C18 Column Conditions for the 6 Additional Compounds:

Gradient Profile (HPLC3)		**Run Time Profile**
HPLC3 File:	EXTRAS	
Pump A Solvent:	Water	
Pump B Solvent:	Methanol	
Flow:	1.0 mL/minute	
Gradient:	Linear	
Run Time:	53 minutes	
Acquisition Time:	30 minutes	
Initial %A	90	
Initial %B	10	
Gradient 1 %A:	70	
Gradient 1 %B:	30	
Ramp Time 1:	5 minutes	0 - 5 minutes
Hold Time 1 :	0 minutes	
Gradient 2 %A:	50	
Gradient 2 %B:	50	
Ramp Time 2:	3 minutes	5 - 8 minutes
Hold Time 2 :	7 minutes	8 - 15 minutes
Gradient 3 %A:	26	
Gradient 3 %B:	74	
Ramp Time 3:	2 minutes	15 - 17 minutes
Hold Time 3:	13 minutes	17 - 30 minutes
Gradient 4 %A:	90	
Gradient 4 %B:	10	
Ramp Time 4:	2 minutes	30 - 32 minutes
Hold Time 4:	21 minutes	32 - 53 minutes (Column Equilibration)

4.1.4 Current C8-CN in Series for the 6 Additional Compound Confirmation:

Time	Flow	Methanol	Water
	1.0	15	85
2.4	1.0	15	85
7.0	1.0	40	60
10.0	1.0	50	50
17.0	1.0	50	50
20.0	1.0	80	20
21.0	0	15	85

4.1.5 Current Column Conditions for NG/PETN:

C18		CN	
Pump A Solvent:	Methanol	Pump A Solvent:	Methanol
Pump B Solvent:	Water	Pump B Solvent	Water
Flow:	0.6 mL/minute:	Flow:	1.2 mL/minute
Gradient:	Linear (1:1)	Gradient:	Linear (1:1)
Run Time:	30 minutes	Run Time:	20 minutes
Acquisition Time:	30 minutes	Acq. Time:	20 minutes
Injection Volume:	50 µL inj.	Time:	50 µL

4.2 Method Detection Limit Study

4.2.1 Prior to the analysis of any samples, it is necessary to establish method detection limits. This procedure is fully described in the Laucks SOP on determination of detection limits. Briefly, it involves the analysis of 7 replicate samples spiked at a concentration near the anticipated method detection limit. A Student's T-test is then applied to these measured values to calculate the MDL. MDL studies are performed on both columns.

4.3 Method Validation

4.3.1 Prior to the analysis of any samples, it is necessary to validate the method. A method validation study is performed in a similar manner to an MDL study with the exception that a minimum of 4 replicates are required and the concentration levels are typically higher.

4.4 Retention Time Windows

4.4.1 It is necessary to establish retention time windows for the method by analyzing standards for all target analytes over at least a 24-hour period. A 24-hour time period may be deemed more appropriate for this analysis because the instrument is not always operated for longer periods when analyzing sample extracts. At least 5 standards must be injected during the 24-hour time period, at about equal time intervals. These standards should be interspersed with real sample extracts in order to mimic actual instrument operating conditions. Tabulate the retention times for all standard compounds and compute the sample (n-1) standard deviations of all the retention times.

4.4.2 The retention time window half-width is set at 3 times the above calculated standard deviation. This operation must be repeated whenever major equipment changes are made or whenever the chromatographic method is modified.

4.4.3 In some cases, the retention time window may be modified in order to take into account any pattern shifting. This shift is acknowledged by observation of the surrogate peak behavior and the surrounding CCV standards. If retention time shift has occurred and the possibility of misidentifying peaks exists, then the sample is reanalyzed bracketed by in-control CCV standards.

4.4.4 See the Laucks SOP on determination of retention time windows for more specifics on their determination and use.

4.5 Initial Multi-Point Calibration

4.5.1 Analyze standard solutions (including the surrogate solution) using at least 5 different concentration levels. The lowest concentration defines the reporting limit. The highest concentration should define the upper usable working range of the detector. Inject the standard solutions from the lowest concentration to the highest. Criteria for evaluating these standards are detailed in Section VI.

4.6 Initial Calibration Verification

4.6.1 At the beginning of an analysis sequence, analyze the mid-point calibration standard (STD 3). The computed calibration factor (CF) or concentration measurement must meet the criteria detailed in Section VI.

4.6.2 Since the retention time windows which were established by the retention time study are relative and not absolute, the windows are anchored by the ICV. This allows the retention times from the ICV.to become the mid-point of the retention time windows..

4.7 Instrument Blank

4.7.1 After the analysis of Standard 5, an instrument blank (IBLK) is analyzed. This is to verify that there is no carryover between sample injections. Evaluation criteria are detailed in Section VI.

4.7.2 Any sample that is suspected of containing high concentrations of target analytes should be followed by an IBLK. This IBLK analysis is used only to make a judgment as to the possibility of carryover into the sample analysis immediately following the IBLK. Evaluation criteria are detailed in Section VI.

4.8 Continuing Calibration Verification

4.8.1 A mid-range calibration standard (STD 3) is analyzed after every ten sample injections. In addition, this standard must be the last injection made in the analysis sequence. Evaluation criteria are detailed in Section VI.

4.9 Sample Analysis

4.9.1 Analysis sequence

See Appendix II for a detailed analysis injection sequence.

4.9.2 Compound Identification

4.9.2.1 Compounds are tentatively identified if a peak elutes in the retention time window characteristic of that compound on the primary column. To confirm the presence of that compound in the sample extract, the peak must also elute in its retention time window on a second column. There are several analyte co-elutions which occur on the secondary column. In instances where target analytes that have been tentatively identified on the C18 column co-elute on the CN column, positive confirmation can not be made. All analyte quantitations come from the C18 column. The CN column is typically not used for quantitation purposes. The responses of the eluting compounds are summed and reported as one peak. When the presence of these compounds are indicated on the C18 column, CN column concentration values can not be accurately determined due to the coeluting peaks and the sample results are flagged appropriately. Retention time windows are established as previously described and are updated each QC period. Compounds can only be identified if the ICV and CCV criteria detailed in Section VI are strictly adhered to. Due to constraints of the software, the RT of the co-eluting compounds is determined by assigning the RT to the highest point within a give window. For the co-eluting compounds that may have a slight but not complete separation, such as: 2-nitrotoluene, 3-nitrotoluene and 4-nitrotoluene, this determination of RT can result in RT

windows which exceed those identified by the standard (due to the changes in peak heights with concentration values). In order to avoid potential exceedance of RT windows, and in order to accurately identify these co-eluting target analytes, the retention time windows may be administratively set for the co-eluting compounds.

4.9.2.2 The experienced analyst's judgment weighs heavily in evaluating chromatograms for compound identification. For instance, the retention times of surrogate compounds may be outside their expected windows due to sample matrix effects. The analyst may decide to re-adjust the target analyte's retention time windows on an ad hoc basis based on such an observed shift. This can occur only on a sample-specific basis and is used when the analyst examining the data suspects that a retention time shift has occurred. If this is done, it must be fully documented in the case narrative notes. If the concentration of any target analyte exceeds the calibration range, the sample extract must be diluted and reanalyzed.

4.9.3 Compound Quantification

4.9.3.1 Target compound concentrations are calculated using the following equations:

4.9.3.2 Aqueous samples

The external standard equation, as expressed in SW-846 is

$$Concentration(\mu g / L) = \frac{A_x \times A \times V_t \times D}{A_s \times V_i \times V_s}$$

where:

A_x = Response for the analyte in the extract, in area or height units.
A = Amount of standard injected (in nanograms).
A_s = Response for the external standard, same units as A_x.
V_i = Volume of extract injected, µL.
D = Dilution factor of extract. The final result of an algebraic multiplication of the ratio of all dilution final volumes to initial volumes. For example, if an extract is diluted 10 µL to 1000 µL and subsequently diluted an additional 10 µL to 1000 µL, the expression is: (1000/10) * (1000/10) = 100 * 100 = 10,000. If no dilution was made, D = 1.
V_t = Volume of total extract, µL.
V_s = Initial sample size, mL.

4.9.3.3 In routine use at Laucks, the equation reduces as follows.

4.9.3.4 First, CF is used directly in the equation. Since Cf = A_s/A, this substitution is made. Next, since Laucks routinely measures all final extract volumes in mL, a conversion factor for

µL to mL must be made in the numerator of the expression - i.e., $V_t = 1000 * mL$. Finally, the sample preparation process is represented as the algebraic ratio of initial sample size to final extract volume.

4.9.3.5 The equation then becomes

$$Concentration(\mu g / L) = \frac{1000 \times Ax \times D}{CF \times Vi \times (Vs / Vt)}$$

4.9.3.6 This expression is completely equivalent to the SW-846 equation, yielding the same final result. To report concentrations in alternate units, apply an appropriate factor:

mg/L = µg/L * 0.001

4.9.3.7 Non-aqueous samples

4.9.3.8 The results calculation for non-aqueous samples is very similar to that for aqueous samples. The only difference is the inclusion of a total solids term to calculate the dry weight equivalent of the initial sample size.

$$Concentration(\mu g / kg) = \frac{1000 \times A_x \times D}{CF \times V_i \times (W / V_t) \times T_s}$$

where:

W_s = Sample size extracted in grams.
T_s = Total Solids in decimal format (i.e. 0.76 not 76).

5. Reports

5.1 Data Packet Organization

See Appendix III for a check list detailing data packet organization.

5.2 Quality Control Reports

5.2.1 All results for quality control tests are entered into the lab data base. Printouts of all data entered must be included in the data packet. The routine minimum is a method blank report, a method blank spike report, and an MS/MSD report.

5.3 Data Qualifying Flags

5.3.1 Sample report results are qualified with data qualifying flags. These flags have the following definitions:

U:	The analyte of interest was not detected, to the limit of detection indicated.
B:	The analyte of interest was detected in the method blank associated with the sample, as well as in the sample itself. The B flag is applied without regard to the relative concentrations detected in the blank and sample.
J:	The analyte of interest was detected below the practical quantitation limit. This value should be regarded as an estimate.
D:	The value reported is derived from the analysis of a diluted sample or sample extract.
P:	When a dual column technique is employed, this flag indicates that calculated results from the two columns differ by more than 25 percent.
E:	The value reported is based on a sample or sample extract in which the target analyte concentration exceeded the calibration range. The value reported should be considered an estimate.
X:	The sample has been analyzed at several dilutions. The value reported has been determined to be the most appropriate quantitative value.
Z:	The value reported is from the C18 column only. Due to coelution with another target analyte on the CN column, the compound could not be quantitatively confirmed.

- Due to software constraints, the percent difference will also be calculated when quantitative confirmation is not possible on the confirmation column due to co-elution. When this happens, the concentration value of the confirmation column used to determine the %D is calculated as the sum of the co-eluting compounds and reported as one peak.

6. Quality Control

See the Requirements and Corrective Actions table in Appendix IV for additional information.

6.1 Initial Calibration

6.1.1 Criteria

6.1.1.1 The initial calibration is evaluated by calculating the %RSD of the calibration factors from the five linearity standards.

6.1.1.2 CFs are calculated using the equation:

$$CF = \frac{\text{response}}{\text{ng injected}}$$

6.1.1.3 This %RSD method assumes a linear response with the calibration curve passing through the origin.

6.1.1.4 The calculated CFs are tabulated and the %RSDs calculated. All compounds must have a %RSD of 20% to meet the method criteria.

6.1.2 Corrective action

6.1.2.1 If the %RSD criteria of 20% are not met, the out of control standard should be reanalyzed. If the curve is still out of control, determine the cause of failure and correct. Recalibrate the instrument and reanalyze any samples associated with the out of control curve.

6.2 Initial Calibration Verification

6.2.1 Criteria

6.2.1.1 Using the average CF calculation technique, compute the CFs for each compound. The calibration factors for the ICV standard are compared to the mean CFs for the initial multi-point calibration. The percent difference for these calibration factors is calculated as follows:

$$\%\,Difference = \frac{CF_a - CF_i}{CF_a} \times 100$$

where:
CF_a = .Average CF from the initial multi-point calibration
CF_i = Calibration Factor of the calibration verification standard.

6.2.1.2 The %D cannot exceed 15% for any target analytes or surrogates.

6.2.1.3 The mid-point for the RT window of each compound is updated using the ICV RTs.

6.2.2 Corrective action

6.2.2.1 If the ICV criteria are not met, no sample extracts can be analyzed. Determine the cause of the ICV failure and correct. Reanalyze the ICV and if it is still out of control, a new calibration curve must be analyzed.

6.3 Continuing Calibration Verification

6.3.1 Criteria

6.3.1.1 A CCV standard is analyzed singly for every 10 injections, and after the last sample of the sequence. The CF for each compound is calculated and the percent difference is calculated as shown above.

6.3.1.2 The %D results cannot exceed 15% for any target analytes or surrogates.

6.3.1.3 The retention times for all target analytes must fall within the RT windows established by the ICV.

6.3.2 Corrective action

6.3.2.1 Determine the cause of failure and correct. Reanalyze the calibration curve. All samples bracketed by an out of control CCV must be reanalyzed unless the CCV demonstrates an

increase in response and no analytes are detected above the reporting limit in the associated samples.

6.4 Instrument Blank

6.4.1 Criteria

6.4.1.1 There must be no target analyte levels above the reporting limit in the initial IBLK.

6.4.2 Corrective action

6.4.2.1 If the initial IBLK contains measurable levels of target analytes, the system is out of control. The source of contamination must be identified and corrected.

6.4.2.2 IBLKs are used to monitor for possible carryover in high concentration extracts. Those IBLKs optionally placed into the sequence following suspected high concentration extracts are used to flag the possibility of analyte carryover into the following sample extract. The extract immediately following the out of control IBLK may need to be reanalyzed if there is a detectable amount of the analyte found in the IBLK.

6.5 Method Blank

6.5.1 Criteria

6.5.1.1 Method blanks are used to verify contamination free reagents and apparatus. They are prepared with every set of samples extracted at the same time or at least one blank for every 20 samples, whichever is more frequent. Analytes of interest should not be detected at levels greater than the current reporting limit in the method blank sample. If any analytes are detected above the reporting limit, corrective action must be taken.

6.5.2 Corrective action

6.5.2.1 Reanalyze the blank and check calculations. If it is still out of control, re-extract the entire batch of samples unless the analyte(s) present in the method blank are not present in the associated samples. In any event it is the laboratory's responsibility to ensure that method interferences caused by contaminants in solvents, reagents, glassware, and other sample processing hardware leading to discrete artifacts and/or elevated baselines in the chromatograms be minimized. In the extreme case of chronic contamination, blanks may have to be analyzed from each stage of the sample processing to determine the contamination source so it can be eliminated. In all cases where blank contamination exceeds the control limit a narrative comment must be made which documents the corrective actions taken.

6.6 Blank Spike or QC Check Sample (LCS)

6.6.1 Criteria

6.6.1.1 A blank spike follows the same protocol as the matrix spike analysis except that the spiking solution is added to a method blank solution instead of an actual sample. In addition, this spiking solution is supplied from a source other than the calibration standards. The use of a second source for the spiking standards is employed in order to verify the calibration standards. A method blank with added analytes is a blank spike. Blank spike percent recovery control limits are detailed in the laboratory QC database. These control limits are updated periodically.

6.6.2 Corrective action

6.6.2.1 The blank spike is used to determine whether a method is in control during sample preparation and analysis. Sample re-extraction and reanalysis would be triggered by any analytes falling outside of control limits in the blank spike sample unless all sample surrogate recoveries and MS/MSD spike recoveries are in control.

6.7 Matrix Spike

6.7.1 Criteria

6.7.1.1 A sample is chosen at random (unless designated by project-specific requirements) from the samples to be analyzed, and an aliquot of spiking solution is added to this sample prior to extraction. It is required that a matrix spike analysis be performed with each extraction batch. However, the minimum frequency for MS analysis is 1 each per 20 samples per matrix. This frequency may be changed on a project specific basis. This matrix spike sample is used to evaluate the matrix effect of the sample upon recovery of the analytes. This spiking solution is supplied from a source other than the calibration standards. The use of a second source for the spiking standards is employed in order to verify the calibration standards. The recovery of spike analytes is calculated as follows:

$$\%Recovery = \frac{SSR - SR}{SA} \times 100$$

where:
SSR = Concentration in spiked sample.
SR = Native concentration in unspiked sample.
SA = Concentration of spike added.

6.7.1.2 The current recovery criteria are detailed in the Laucks quality control database. These control limits are updated periodically.

6.7.2 Corrective action

6.7.2.1 Samples with spike recoveries outside control limits will be reviewed for possible corrective action. Corrective action may involve recalculation, re-extraction and/or reanalysis. This process should also look at the recovery of surrogate compounds in the MS sample and at the recovery of matrix spiking compounds from the extraction batch blank spike analysis. In all cases a narrative explanation of the condition is required to detail the corrective actions taken.

6.8 Matrix Spike Duplicate

6.8.1 Criteria

6.8.1.1 The compound recovery criteria are identical to those for the matrix spike sample. In addition, the matrix spike duplicate is used to measure method precision. This is done by computing the relative percent difference (RPD) between the matrix spike and matrix spike duplicate recovery values. This calculation is as follows:

$$RPD = \frac{S1 - S2}{(S1 + S2)/2} \times 100$$

where:
S1 = Measured concentration for MS sample.
S2 = Measured concentration for MSD sample.

6.8.1.2 The current RPD control limits are detailed in the Laucks quality control database. These control limits are updated periodically.

6.8.2 Corrective action

If a trend in out of control RPD values is observed, the methods used must be examined to determine the source of variance. Once this source is identified, the method must be changed so that samples can be analyzed with a predictable reproducibility.

6.9 Surrogate Recovery

6.9.1 Criteria

6.9.1.1 Surrogates are chemically similar compounds added to every sample, method blank, and QC sample prior to sample processing. They are used to monitor for potential sample processing errors and matrix effects. Surrogate compound recoveries are calculated as follows:

$$\%Recovery = \frac{S_m}{S_a} \times 100$$

where:
S_m = Concentration of surrogate measured in sample.
S_a = Concentration of surrogate added.

6.9.1.2 Detailed surrogate recovery control limits are detailed in the Laucks quality control database. These control limits are updated periodically.

6.9.2 Corrective Action

6.9.2.1 Reanalyze the sample. Low surrogate recoveries are greater potential indicators of poor method performance than high surrogate recoveries since non-GC/MS methods cannot separate co-eluting interferents. In instances where high surrogate recoveries are attributable to matrix effects, no corrective action is taken. However, if elevated surrogate recoveries are attributable to preparation error, re-extraction and reanalysis is performed.

6.9.2.2 Check calculations for possible error. Re-extract the sample if surrogate recovery is less than the lower control limit. If a poor injection is suspected, reanalyze the sample.

6.9.2.3 Low surrogate recoveries in the method blank may require that all the samples in the associated batch be re-extracted and reanalyzed. In any case, it is imperative to identify the problem associated with low recovery so that it can be corrected. It is a requirement that all out of control surrogate recoveries and the corrective action taken be discussed in the narrative.

7. References

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Method 8000B, "Gas Chromatography," Revision 1, July 1992, U.S.E.P.A.

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40 CFR Part 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Final Rule and Interim Final Rule and Proposed Rule," October 26, 1984, U.S.E.P.A.

Special Report 93-11, "Experimental Assessment of Analytical Holding Times for Nitroaromatic and Nitroamine Explosives in Soil," Clarence L. Grant et al, June 1993, U.S. Army Cold Regions Research and Engineering Laboratory.

Special Report 93-24, "Evaluation of Pre-extraction Analytical Holding Times for Nitroaromatic and Nitroamine Explosives in Water," Clarence L. Grant et al, August 1993, U.S. Army Cold Regions Research and Engineering Laboratory.

APPENDIX I

Calibration Standard Solution Concentrations, µg/mL

Compound	STD1	STD2	STD3	STD4	STD5
HMX	0.1	0.2	1.0	2.0	10.0
RDX	0.1	0.2	1.0	2.0	10.0
1,3,5-TNB	0.1	0.2	1.0	2.0	10.0
1,3-DNB	0.1	0.2	1.0	2.0	10.0
Tetryl	0.1	0.2	1.0	2.0	10.0
NB	0.1	0.2	1.0	2.0	10.0
2,4,6-TNT	0.1	0.2	1.0	2.0	10.0
4-Am-DNT	0.1	0.2	1.0	2.0	10.0
2-Am-DNT	0.1	0.2	1.0	2.0	10.0
2,4-DNT	0.1	0.2	1.0	2.0	10.0
2,6-DNT	0.1	0.2	1.0	2.0	10.0
2-NT	0.1	0.2	1.0	2.0	10.0
3-NT	0.1	0.2	1.0	2.0	10.0
4-NT	0.1	0.2	1.0	2.0	10.0
Surrogate					
1,2-Dinitrobenzene	0.1	0.2	1.0	2.0	10.0

Calibration Standard Solution Concentrations, µg/mL

Compound	STD1	STD2	STD3	STD4	STD5
TNX	0.1	0.2	1.0	2.0	10.0
2,6-DA-4-NT	0.1	0.2	1.0	2.0	10.0
2,4-DA-4-NT	0.1	0.2	1.0	2.0	10.0
MNX	0.1	0.2	1.0	2.0	10.0
3,5-DNA	0.1	0.2	1.0	2.0	10.0
4,4'-TN-AZOXY	0.1	0.2	1.0	2.0	10.0
Surrogate					
1,2-Dinitrobenzene	0.1	0.2	1.0	2.0	10.0

Calibration Standard Solution Concentrations, µg/mL

Compound	STD1	STD2	STD3	STD4	STD5
NG	0.5	1.0	5.0	10.0	20.0
PETN	0.25	0.5	2.5	5.0	10.0
Surrogate					
1,2-Dinitrobenzene	0.25	0.5	2.5	5.0	10.0

Surrogate Stock Solution

Compound	Concentration
1,2-Dinitrobenzene	1000 µg/mL

IBLK Solution

Compound	Concentration
1,2-Dinitrobenzene	2.0 µg/mL

APPENDIX II

Analysis Sequence

Injection	Sample
1	Standard #1
2	Standard #2
3	Standard #3 (ICV Standard)
4	Standard #4
5	Standard #5
6	IBLK
7	ICV
	injections (8-16)
17	IBLK
18	CCV Standard
19	injections (19-27)
28	BLK
29	CCV Standard
	injections (total of 10 injections between CCVs)
last	IBLK
last	CCV Standard

APPENDIX III

Data Packet Order List

I. QC SUMMARY

Surrogate Recovery Summary Report
Blank Spike Report
MS/MSD Report
Method Blank Summary

II. SAMPLE DATA:

Organic Analysis Data Sheet
Sample Confirmation Worksheet
Chromatogram, primary column
Quantitation Report, primary column
Chromatogram, secondary column
Quantitation Report, secondary column

III. STANDARD DATA:

Linearity Report
ICAL Data
ICAL Response, ICAL std concentrations
ICV reports
CCV reports
Other Standards Used to Support Sample Data and Instrument Blanks

V. Raw QC Data:

Method Blank
Chromatograms
Quantitation Report

Blank Spike
Chromatograms,
Quantitation Report

Matrix Spike
Chromatograms
Quantitation Report

Matrix Spike Duplicate
Chromatograms
Quantitation Report

V. Bench Sheets

SDG Report
Extraction Bench Sheets
Injection Sequence (logbook copy)
Miscellaneous Work Sheets. (%TS, calcs, HTVRs)
Standards Logs

VI. Reject Data:

Data not used to support reported sample results.
All data acquired but rejected on account of QC out of control.
Non-routine standards used to support sample data should be placed at the last of the Standard Data section.

APPENDIX IV

Method 8330 Requirements and Corrective Actions					
QA Element	**Method Criterion**	**Laucks Criterion**	**Frequency**	**Corrective Action**	**Documentation**
Initial Calibration	Minimum of 5 levels.	Minimum of 5 levels. Must demonstrate an RSD of <20%.	As necessary due to major instrument maintenance or difficulties meeting the CCV requirements.	Reanalyze out of control standard. If still out, determine cause of curve failure and correct. Re-analyze curve and any samples analyzed against curve.	Target forms and raw data.
Initial Calibration Verification	+/- 15% of the initial calibration response factor.	+/- 15% of the initial calibration response factor.	Every 24 hr. or at the beginning of an analytical sequence, which ever is more frequent, or as necessary.	Determine cause of ICV failure and correct. Reanalyze ICV and if out of control a new calibration curve must be analyzed.	Target form and raw data.
Continuing Calibration Verification	+/- 15% of the initial calibration response factor. Analyze singly every 10 injections and after the final sample.	+/- 15% of the initial calibration response factor.	Every 10 injections and at the end of an analytical sequence.	Determine cause of failure and correct. Reanalyze calibration curve. All samples bracketed by an out of control CCV must be reanalyzed unless the CCV demonstrates an increase in response and no analytes are detected above the reporting limit.	Target form and raw data.
Instrument Blank	Analyze after analysis of a sample with analyte levels which exceed the upper calibration level.	Analyze after samples when high levels of matrix are suspected.	As necessary.	Reanalyze any samples with suspected carryover.	Raw data
Method Blank	All analytes must be < MDL	All analytes must be less than the Reporting Limit	One method blank per 20 samples or each extraction batch, whichever is more frequent.	Reanalyze blank. If still out of control, re-extract the entire batch of samples unless the detected analyte(s) are not present in the associated samples.	Method Blank Summary and raw data. Narrative comment when necessary.

Method 8330 Requirements and Corrective Actions					
QA Element	**Method Criterion**	**Laucks Criterion**	**Frequency**	**Corrective Action**	**Documentation**
Surrogate Recovery	Minimum of 1 surrogate. Recoveries must fall within the labs established windows.	See latest version of Laucks Testing Labs control limits catalogue. All surrogate recoveries must fall within the generated limits. If laboratory limits are not established due to the implementation of a new surrogate , the default limits of 70% - 130% are used until enough data points are collected to generate new limits.	All samples, method blanks, and QC samples.	Re-extract if surrogate recovery is < the lower control limit. If a poor injection is suspected reanalyze the sample.	Target Surrogate Summary form or special test results. Narrative comment when necessary.
MS/MSD	One per 20 samples or extraction batch which ever is more frequent. Must be per matrix.	See latest version of Laucks control limits located in the QC database.	1 MS/MSD pair per 20 samples or every extraction batch which ever is more frequent.	Per SW 846, if analyte recoveries are out of control in the MS/MSD but are in control in the associated blank spike, no further action is required.	MS/MSD report and raw data. Narrative comment when necessary.
Blank Spike	N/A	See latest version of Laucks Testing Labs control limits in the QC database. All recoveries must fall within the generated limits.	1 per 20 samples or every extraction batch which ever is more frequent.	Re-extract if analyte recoveries are outside of control limits.	Blank Spike report and raw data. Narrative comment when necessary.

APPENDIX V

Elution Order of Target Analytes

Compound	Order of Elution on Primary (C18) Column	Order of Elution on Confirmation (CN) Column
HMX	1	10
RDX	2	8
1,3,5-TNB*	3	1
1,3-DNB	5	2
Tetryl	6	9
NB*	7	1
2,4,6-TNT*	8	4
4-Am-DNT	9	6
2-Am-DNT	10	7
2,4-DNT*	11	4
2,6-DNT*	12	4
2-NT*	13	3
3-NT*	14	3
4-NT*	15	3
Surrogate		
1,2-Dinitrobenzene	4	5

Elution Order of 6 Additional Analytes

Compound	Order of Elution on Primary (C18) Column	Order of Elution on Confirmation (C8+CN) Column
TNX	1	1
2,6-DA-4-NT	2	2
2,4-DA-6-NT	3	3
MNX	4	4
3,5-DNA	6	6
4,4'-TN-AZOXY	7	7
Surrogate		
1,2-Dinitrobenzene	5	5

Elution Order of PETN/NG

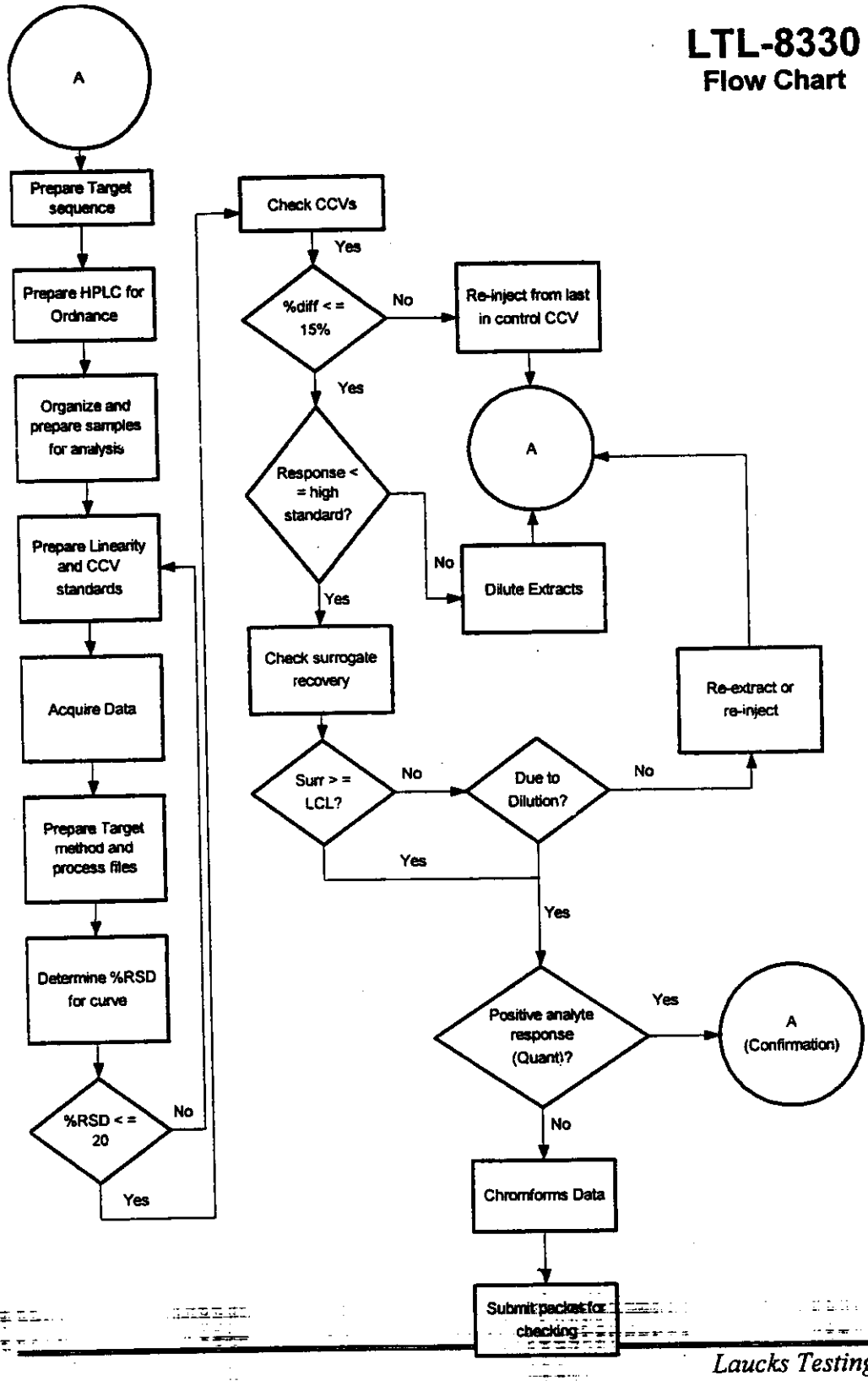
Compound	Order of Elution on Primary (C18) Column	Order of Elution on Confirmation (CN) Column
NG	2	2
PETN	3	3
Surrogate		
1,2-Dinitrobenzene	1	1

*Indicates the compound co-elutes on the confirmation (CN) column.

APPENDIX VI

Method 8330 Flow Chart

LTL-8330
Flow Chart



APPENDIX II

Analysis Sequence

Injection	Sample
1	Standard #1
2	Standard #2
3	Standard #3 (ICV Standard)
4	Standard #4
5	Standard #5
6	IBLK
7	ICV
	injections (8-16)
	17 IBLK
	18 CCV Standard
	19 injections (19-27)
	28 BLK
	29 CCV Standard
	injections (total of 10 injections between CCVs)
last	IBLK
last	CCV Standard