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

10-5-1998

IN REPLY REFER TO:
5090
Ser 095/8231
07 DEC 1998

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:

Crane Division, Naval Surface Warfare Center (NAVSURFWARCENDIV Crane) submits for review and approval three copies of the Revised Quality Assurance Project Plan (QAPP) for Full Scale (FS) Bioremediation Operations as enclosure (1). This revision has been made to include Quanterra Environmental Services Laboratory of California to perform analytical work in addition to Southwest of Oklahoma Laboratories. Only the pages affected by this revision are provided. Enclosure (2) is the required certification statement.

NAVSURFWARCENDIV Crane point of contact is Ms. Christine D. Freeman, Code 09511, telephone 812-854-4423.

Sincerely,

James M. Hemsicker

JAMES M. HEMSICKER
Director, Environmental Protection Department
By direction of
the Commander

Encl:
(1) Revised FS Bioremediation Operations QAPP (Quanterra addition)
(2) Certification Statement

Copy to:
ADMINISTRATIVE RECORD (2 copies)
COMNAVSEASYSCOM (SEA OOT) (w/o encls)
IDEM (S. Riddle)
MK Crane (D. Beall) (w/o encls)
SOUTHNAVFACENGCOM (Code 1864) (w/o encls)

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.

James M. Hunsicker
SIGNATURE

ENV. PROTECTION Dept. Mg.
TITLE

12/4/98
DATE

Quality Assurance Project Plan
For Full-Scale Operations at the Bioremediation Facility
NSWC Crane, Crane, Indiana
Revision 3 dated October 05, 1998

INSTRUCTIONS FOR REPLACEMENT PAGES

NOTE: THIS REVISION HAS BEEN MADE TO INCLUDE QUANTERRA ENVIRONMENTAL SERVICES LABORATORY TO PERFORM ANALYTICAL WORK IN ADDITION TO SOUTHWEST OF OKLAHOMA LABORATORIES. ONLY PAGES AFFECTED BY THIS REVISION ARE PROVIDED.

Cover Page: Remove Cover Page and Spine binder labeled Revision 2 dated March 12, 1998 and discard. Replace with new cover page and spine binder labeled Revision 3, October 05, 1998.

Title Page: Remove Title pages dated March 12, 1998 and discard. Replace with new title pages dated October 05, 1998 Revision 3.

Section 1: Remove pages 3 and 4 of 44 dated 03/04/98 and discard. Replace with new pages 3 and 4 of 44 dated 10/05/98.

Section 1: Remove Tables 1-1 through 1-4b on pages 5a through 9i and discard. Replace with new paged 5a through 9i all dated 10/05/98. Retain page 10 of 44 dated 3/4/98.

Section 1: Remove Table 1-5 on pages 11 and 12 of 44 dated 3/4/98 and discard. Replace with new pages 11 and 12 of 44 containing Table 1-5 dated 10/5/98.

Section 1: Remove pages 36 and 37 of 44 dated 3/4/98 and discard. Replace with new pages 36 and 37 of 44 dated 10/05/98.

Section 3: Remove Table 3.1 through Table 3.7 on pages 6 through 13 dated 3/4/98 and discard. Replace with new pages 6 though 13 of 14.

Section 6: Remove Table 6-1 on page 2 of 2 dated 2/09/98 and discard. Replace with new page 2 of 2 dated 10/05/98.

Section 7: Remove Table 7-1 on page 3 dated 3/04/98 and discard. Replace with new page 3 of 3 dated 10/05/98.

Appendix B: Include the attached Table of Contents for Appendix B after cover page for Appendix B. Append Appendix B by adding page B-14 to the end of the Appendix B after page B-13.

Appendix C: Remove the Table of Contents (1 page printed both sides), and replace with new Table of Contents (3 pages).

Appendix C: Append Appendix C by adding attached Section 3 (cover and pages C3-1 thru C3-16) and Section 4 (cover and pages C4-1 through C4-634) following the end of Section 2 (page C2-393).

Appendix D: Include the attached Table of Contents for Appendix D after cover page for Appendix D. Append Appendix D by adding attached pages D1-1 through D1-48 after page D-41.

Appendix F: Include the attached Table of Contents for Appendix F after cover page for Appendix F. Append Appendix F by adding attached pages F1-1 through F1-91 after page F-76.

QUALITY ASSURANCE PROJECT PLAN
For Full-Scale Operations
Soils Bioremediation Facility
NSWC Crane - Crane Indiana

CONTRACT
N62467-93-D
-1106

DO 0009
SOW 007

Prepared by

MORRISON
KNUDSEN
CORPORATION

Octobe 5, 1998

Rev. 3

QUALITY ASSURANCE PROJECT PLAN
FOR
FULL-SCALE OPERATIONS AT THE BIOREMEDIATION FACILITY

NSWC CRANE
CRANE, INDIANA

CONTRACT #N62467-93-D-1106
DELIVERY ORDER #0009
STATEMENT OF WORK #0007

REVISION ~~010~~203

~~February 09 March~~October ~~041~~205,1998

Prepared for:

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

Prepared by:

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

QUALITY ASSURANCE PROJECT PLAN
FOR
FULL-SCALE OPERATIONS AT THE BIOREMEDIATION FACILITY

NSWC CRANE
CRANE, INDIANA

CONTRACT #N62467-93-D-1106
DELIVERY ORDER #0009
STATEMENT OF WORK #0007

REVISION ~~010~~203

~~February 09 March~~October ~~0412~~05,1998

Prepared for:

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QUALITY ASSURANCE PROJECT PLAN
FOR
FULL-SCALE OPERATIONS AT THE BIOREMEDIATION FACILITY

NSWC CRANE
CRANE, INDIANA

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

REVISION ~~0102~~3

~~February 09 March~~ October ~~0412~~ 05, 1998

Prepared for:
SOUTHERN DIVISION
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NORTH CHARLESTON, SOUTH CAROLINA 29406

MK Program Manager	Date
MK Program Quality Control Manager	Date
US Navy Responsible Authority	Date
USEPA RCRA Project Coordinator	Date
USEPA Region 5 QAPP Coordinator	Date
Laboratory Representative	Date

The Department of Defense (DOD) has successfully utilized bioremediation, or composting, to treat explosives-contaminated soil since 1982. Bioremediation has proven to be successful at other sites having explosive-contaminated materials (Weston, 1993). Explosive contaminants in soils and sludges that have been successfully degraded by composting include: trinitrotoluene (TNT); hexahydro-1,3,5,-trinitro-1,3,5,-triazine (RDX); octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX); methyl-2,4,6-trinitropheylnitramine (Tetryl); and nitrocellulose (U.S. EPA, 1993a, 1993b). Based on this success, on-site bioremediation was selected as the preferred treatment alternative for the Interim Measures (IM) at ABG, Rockeye, Mine Fill A, and Mine Fill B. In addition to its effectiveness in treating explosives, on-site bioremediation involves limited transportation of contaminated soil and is less expensive that the other treatment alternatives evaluated.

1.1.2 Scope of Work and Overall Objectives

The scope of the of IM cleanup includes Full-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. IM cleanup actions will be completed in compliance with State of Indiana and U.S. EPA Region 5 guidance and requirements.

The first phase of the IM process consisted of a pilot-scale bioremediation operation which evaluated several mix recipes and provided lessons learned, a final recipe for full-scale operation, and cleanup goals for treated soil. Results and discussions on the pilot-scale operations are provided in the Pilot-Scale Completion Report (MK, 1998a). The second phase of the IM process is the full-scale operation for production treatment of explosives-contaminated soil. This QAPP discusses the second phase of operation, the full-scale operation.

The primary objective of full-scale operation at the Bioremediation Facility is to reduce, through composting, the explosive contaminant levels of affected soils to meet the National Contingency Plan (NCP) criteria of 90% to 99% reduction of toxicity and mobility (leachability), and to achieve human health (residential or industrial) and/or ecological risk-based remedial goals based on disposition of soil.

Human health (residential and industrial) and ecological risk-based remedial goals have been established using preliminary remedial goals and data quality levels by U.S. EPA Region 5 (U.S. EPA, 1998). Cumulative risk, other risk scenarios, and toxicity testing results are not considered in the U.S. EPA evaluation. These remedial goals are presented in Tables 1-1 through 1-3. The analytical methods and analytical reporting limits are also shown in these Tables. Method reporting limits for each of the target constituents are provided by Southwest Laboratory of Oklahoma and Quanterra

Environmental Services and have been evaluated by Morrison Knudsen Corporation (MK) for acceptability for data quality.

Tables 1-4a and 1-4b provides the cleanup goals for rinse water generated after washing the rocks at the excavation site. If the rinse water meets these cleanup levels or non-detect levels, the rocks are determined to be acceptable for use as backfill material; or the rocks may be triple rinsed, crushed, and sent to the Bioremediation Facility for composting.

Table 1-5 provides the allowable discharge limits without pretreatment, analytical methods, and analytical reporting limits for water generated in the storm-water retention ponds at the Biofacility.

Figures 1-2 and 1-3 of the Full-Scale Operations Plan (MK, 1998b) provide flowcharts describing the process flow path for decision-making of the excavation site soil and the treated soil respectively. Similarly, Figure 1-4 of the Full-Scale Operations Plan (MK, 1998b) provides a flowchart describing the process flow path for decision-making of decontamination of rocks.

Additional full-scale objectives include:

- Identifying areas for full-scale excavations, with respect to chemicals of concern at each of the SWMU sites,
- Determining if soil entering the Biofacility is hazardous with respect to metals ~~and~~ volatile organic compounds (VOCs) and semivolatiles. Soil containing hazardous levels of metals ~~or~~, VOCs or semivolatiles will be not be accepted into the Biofacility for treatment, and
- Properly disposing of waste water produced (i.e. storm-water collected, decontamination water) in compliance with NPDES permit and POTW requirements, as applicable.

Full-scale operations will include:

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

2. Excavation and treatment of approximately 60,000 cubic yards of soil from ABG; approximately 18,000 cubic yards of soil from Rockeye; approximately 11,000 cubic yards of soil from Mine Fill A; and approximately 22,000 cubic yards of soil from Mine Fill B.

TABLE 1 - 1
RESIDENTIAL USE SOIL REMEDIAL GOALS, SWMU-SPECIFIC CHEMICALS OF CONCERN, AND ACCEPTABLE REPORTING LIMITS
NAVAL SURFACE WARFARE CENTER - CRANE

Parameter	SWMU Parameter Applicable To				IM Residential Cleanup Goal (Human Health Only) (mg/kg)	Method of Analysis[1]	Analytical Reporting Limit (mg/kg)	
	MA	MB	ABG	RE			SWL	Quanterra
EXPLOSIVES								
Pentaerythritrol tetranitrate (PETN)	X	X	X	X	To be determined by U.S. EPA 1998	8330	0.25	0.25
2,4,6-trinitrotoluene (TNT)	X	X	X	X	15	8330	0.50	0.25
Cyclotrimethylene trinitramine (RDX) (Hexahydro 1,3,5 trinitro 1,3,5 triaziine)	X	X	X	X	4.0	8330	0.625	0.25
Cyclotetremethylene tetranitramine (HMX) (Octahydro 1,3,5,7 tetranitro 1,3,5,7 tetrazocine)	X	X	X	X	3,300	8330	1.60	0.25
Tetryl (Methyl 2,4,6 trinitro phenyl nitramine)	X	X	X	X	650	8330	0.38	0.25
Trinitrobenzene (TNB)	X	X	X	X	3.3	8330	0.575	0.25
1,3-Dinitrobenzene (DNB)	X	X	X	X	6.5	8330	0.49	0.25
~~1,2 DNB~~	~~X~~	~~X~~	~~X~~	~~X~~	~~26 *~~	~~8330~~		
~~1,4 DNB~~	~~X~~	~~X~~	~~X~~	~~X~~	~~26 *~~	~~8330~~		
Nitrobenzene (NB)	X	X	X	X	18	8330	0.26	0.25
~~Dinitrotoluene (DNT) (total)~~	~~X~~	~~X~~	~~X~~	~~X~~	~~0.65~~	~~8330~~		
4-Amino 2,6 Dinitrotoluene (4-Am DNT)	X	X	X	X	65 **	8330	0.45	0.25

TABLE 1 - 1
RESIDENTIAL USE SOIL REMEDIAL GOALS, SWMU-SPECIFIC CHEMICALS OF CONCERN, AND ACCEPTABLE REPORTING LIMITS NAVAL SURFACE WARFARE CENTER - CRANE

Parameter	SWMU Parameter Applicable To				IM Residential Cleanup Goal (Human Health Only) (mg/kg)	Method of Analysis[1]	Analytical Reporting Limit (mg/kg)	
	MA	MB	ABG	RE			SWL	Quanterra
2-Amino 4,6 Dinitrotoluene (2-Am DNT)	X	X	X	X	130 ***	8330	0.40	0.25
2,4 DNT	X	X	X	X	130	8330	0.24	0.25
2,6-DNT	X	X	X	X	65	8330	0.39	0.25
2-nitrotoluene (2 NT)	X	X	X	X	650	8330	0.72	0.25
3 NT	X	X	X	X	650	8330	0.50	0.25
4 NT	X	X	X	X	650	8330	0.48	0.25

NOTES: MA = Mine Fill A, MB = Mine Fill B, ABG = Ammunition Burning Ground, and RE = Rockeye.
X = Parameter is a Chemical of Concern at the SWMU.
1. Analytical methods listed above are SW-846 methods, unless otherwise indicated.
* = Region 9 Preliminary Remedial Goal (PRG) value.
** = Using 2,6 DNT as the surrogate for toxicity potential.
*** = Using 2,4 DNT as the surrogate for toxicity potential.

TABLE 1 - 1
RESIDENTIAL USE SOIL REMEDIAL GOALS, SWMU-SPECIFIC CHEMICALS OF CONCERN, AND ACCEPTABLE REPORTING LIMITS
NAVAL SURFACE WARFARE CENTER - CRANE

Parameter	SWMU Parameter Applicable To				IM Residential Cleanup Goal (Human Health Only) (mg/kg)	Method of Analysis[1]	Analytical Reporting Limit (mg/kg)	
	MA	MB	ABG	RE			SWL	Quanterra
METALS								
Aluminum	X	X	X	X	77,000	6010	15.0	10.0
Antimony			X	X	31	6010	6.0	6.0
Arsenic	T	T	X T	X T	0.32	7060	0.80	0.50
Barium	X T	X T	X T	X T	5,300	6010	1.0	1.0
Beryllium			X	X	0.14	6010	0.5	0.2
Cadmium	X T	X T	X T	X T	38	6010	0.5	0.5
Chromium (total)	X T	X T	X T	X T	210	6010	1.0	1.0
Cobalt			X	X	4,600	6010	2.5	1.0
Copper			X	X	2,800	6010	2.5	2.0
Iron				X	EN-BG	6010	10.0	10.0
Lead	X T	X T	X T	X T	400	6010	10.0	5.0
Magnesium				X	EN-BG	6010	50.0	50.0
Nickel			X	X	1,500	6010	4.0	4.0
Zinc			X	X	23,000	6010	2.0	2.0
Mercury	X T	X T	X T	X T	23	7471	0.033	0.10

TABLE 1 - 1
RESIDENTIAL USE SOIL REMEDIAL GOALS, SWMU-SPECIFIC CHEMICALS OF CONCERN, AND ACCEPTABLE REPORTING LIMITS NAVAL SURFACE WARFARE CENTER - CRANE

Parameter	SWMU Parameter Applicable To				IM Residential Cleanup Goal (Human Health Only) (mg/kg)	Method of Analysis[1]	Analytical Reporting Limit (mg/kg)	
	MA	MB	ABG	RE			SWL	Quanterra
Selenium	T	T	T	T	380	6010	25.0	20.0
Silver	T	T	T	T	380	6010	1.0	1.0
Manganese			X		3,200 +	6010	1.0	1.0
Thallium			X		6.3 ++	6010	25.0	200
Vanadium			X		540 +++	6010	2.0	1.0
Cyanide			X		1,300 ++++	9010	0.50	0.50

NOTES: MA = Mine Fill A, MB = Mine Fill B, ABG = Ammunition Burning Ground, and RE = Rockeye.
X = Parameter is a Chemical of Concern at the SWMU.
T = Parameter is a TCLP parameter for waste characterization.
1. Analytical methods listed above are SW-846 methods, unless otherwise indicated.
EN-BG = Essential nutrient; individual sample levels must be compared to unimpacted background levels at the site.
+ = Region 9 PRG value.
++ = Average of Region 9 PRGs for Thallium compounds.
+++ = Region 9 PRG for Elemental Vanadium.
++++ = Region 9 PRG value for "Free Cyanide"; this is the remedial goal that should be compared to the "Total Cyanide" measurement.

TABLE 1 - 1
RESIDENTIAL USE SOIL REMEDIAL GOALS, SWMU-SPECIFIC CHEMICALS OF CONCERN, AND ACCEPTABLE REPORTING LIMITS
NAVAL SURFACE WARFARE CENTER - CRANE

Parameter	SWMU Parameter Applicable To				IM Residential Cleanup Goal (Human Health Only) (mg/kg)	Method of Analysis[1]	Analytical Reporting Limit (mg/kg)	
	MA	MB	ABG	RE			SWL	Quanterra
VOLATILE ORGANICS								
Dichloromethane (methylene chloride)	X	X	X		7.8	8260	0.005	0.005
Acetone	X	X	X		2,000	8260	0.005	0.010
Methyl ethyl ketone (2 - butanone)	X T	X T	X T		7,100	8260	0.005	0.010
Methyl isobutyl ketone (4 - methyl -2-pentanone)	X	X			770	8260	0.005	0.010
Toluene	X	X			790	8260	0.005	0.005
Xylene (total)	X	X		X	320	8260	0.005	0.005
Trans 1,3 dichloropropene				X	0.25	8260	0.005	0.005
1,1,1 Trichloroethane			X	X	1,200	8260	0.005	0.005
1,1,2 Trichloroethane			X	X	0.65	8260	0.005	0.005

TABLE 1 - 1
RESIDENTIAL USE SOIL REMEDIAL GOALS, SWMU-SPECIFIC CHEMICALS OF CONCERN, AND ACCEPTABLE REPORTING LIMITS
NAVAL SURFACE WARFARE CENTER - CRANE

Parameter	SWMU Parameter Applicable To				IM Residential Cleanup Goal (Human Health Only) (mg/kg)	Method of Analysis[1]	Analytical Reporting Limit (mg/kg)	
	MA	MB	ABG	RE			SWL	Quanterra
1,1 Dichloroethylene			X		0.07 #	8260	0.005	0.005
1,2 Dichloroethylene			X		35 *	8260	0.010	0.005
Trichloroethylene (TCE)			X		5 #	8260	0.005	0.005
1,1,2,2 Tetrachloroethane			X T		0.6 #	8260	0.005	0.005
Vinyl Chloride			X T		0.03 #	8260	0.005	0.010
Chloroethane			X		1,100 **	8260	0.005	0.010
Chloroform	T		X T		0.03 #	8260	0.005	0.005
Tetrachloroethylene			X		11 #	8260	0.005	0.005
1,2 Dichloroethane	T				0.4 #	8260	0.005	0.005
1,1 Dichloroethane	T				500 **	8260	0.005	0.005

NOTES: MA = Mine Fill A, MB = Mine Fill B, ABG = Ammunition Burning Ground, and RE = Rockeye.
X = Parameter is a Chemical of Concern at the SWMU.
T = Parameter is a TCLP parameter for waste characterization.
1. Analytical methods listed above are SW-846 methods, unless otherwise indicated.
* = For mixture of cis- and trans- isomers; Region 9 PRG.
** = Region 9 PRG.
# = Soil screening level for inhalation of volatile chemical.

TABLE 1 - 1
RESIDENTIAL USE SOIL REMEDIAL GOALS, SWMU-SPECIFIC CHEMICALS OF CONCERN, AND ACCEPTABLE REPORTING LIMITS
NAVAL SURFACE WARFARE CENTER - CRANE

Parameter	SWMU Parameter Applicable To				IM Residential Cleanup Goal (Human Health Only) (mg/kg)	Method of Analysis[1]	Analytical Reporting Limit (mg/kg)	
	MA	MB	ABG	RE			SWL	Quanterra
SEMI-VOLATILE ORGANICS								
Phenanthrene				X	100 *	8270	0.33	0.33
Fluoranthene				X	2,600	8270	0.33	0.33
Pyrene				X	100	8270	0.33	0.33
Bis (2-ethylhexyl) phthalate			X		46 +	8270	0.33	0.33
di-n-butyl phthalate			X		6,500 *	8270	0.33	0.33
N-Nitrosodiphenylamine			X		130 +	8270	0.33	0.33
Benzo (b) fluoranthene			X		0.9 +	8270	0.33	0.33
4 - Nitrophenol			X		NA	8270	1.60	1.60
Benzo (a) anthracene			X		0.9 +	8270	0.33	0.33
Chrysene			X		7 *	8270	0.33	0.33

NOTES: MA = Mine Fill A, MB = Mine Fill B, ABG = Ammunition Burning Ground, and RE = Rockeye.
X = Parameter is a Chemical of Concern at the SWMU.
T = Parameter is a TCLP parameter for waste characterization.
1. Analytical methods listed above are SW-846 methods, unless otherwise indicated.
* = Region 9 PRG.
+ = Soil screening level.
* = Using Pyrene as the surrogate for volatile PAHs which are not suspected to be human carcinogens.

TABLE 1 - 1
RESIDENTIAL USE SOIL REMEDIAL GOALS, SWMU-SPECIFIC CHEMICALS OF CONCERN, AND ACCEPTABLE REPORTING LIMITS
NAVAL SURFACE WARFARE CENTER - CRANE

Parameter	SWMU Parameter Applicable To				IM Residential Cleanup Goal (Human Health Only) (mg/kg)	Method of Analysis[1]	Analytical Reporting Limit (mg/kg)	
	MA	MB	ABG	RE			SWL	Quanterra
POLYCHLORINATED BIPHENYLS (PCBs)								
Arochlor 1242		X			0.066 #	8081/8082	0.033	0.050
Arochlor 1254		X			0.066 #	8081/8082	0.067	0.050
Arochlor 1260		X			0.066 #	8081/8082	0.067	0.050

NOTES: MA = Mine Fill A, MB = Mine Fill B, ABG = Ammunition Burning Ground, and RE = Rockeye.
X = Parameter is a Chemical of Concern at the SWMU.
T = Parameter is a TCLP parameter for waste characterization.
1. Analytical methods listed above are SW-846 methods, unless otherwise indicated.
= This is probably a single assay for PCBs; you cannot analyze PCBs in an old release to soil by assuming that the PCB congener/chlorine content will be similar to the original commercial product.

TABLE 1 - 1
RESIDENTIAL USE SOIL REMEDIAL GOALS, SWMU-SPECIFIC CHEMICALS OF CONCERN, AND ACCEPTABLE REPORTING LIMITS
NAVAL SURFACE WARFARE CENTER - CRANE

Parameter	SWMU Parameter Applicable To				IM Residential Cleanup Goal (Human Health Only) (mg/kg)	Method of Analysis[1]	Analytical Reporting Limit (mg/kg)
	MA	MB	ABG	RE			SWL
DIOXINS							
2,3,7,8 TCDD equivalents			X *		0.001 +	8290	0.00001

NOTES: MA = Mine Fill A, MB = Mine Fill B, ABG = Ammunition Burning Ground, and RE = Rockeye.
X = Parameter is a Chemical of Concern at the SWMU.
T = Parameter is a TCLP parameter for waste characterization.
* - Dioxin Soil Sampling is limited to a proximity around solvent burning areas.
1. Analytical methods listed above are SW-846 methods, unless otherwise indicated.
+ = This is U.S. EPA remedial policy for residential areas (1 part per billion); However, U.S. EPA may elect to adopt a more stringent state standard if one is available.

TABLE 1 - 1
RESIDENTIAL USE SOIL REMEDIAL GOALS, SWMU-SPECIFIC CHEMICALS OF CONCERN, AND ACCEPTABLE REPORTING LIMITS NAVAL SURFACE WARFARE CENTER - CRANE

Preliminary Exposure Scenarios and Land Use Options:

These values only address direct inhalation and ingestion.
No cummulative risk is calculated.
No agricultural crop use is allowed.
No gardening or food chain use is allowed.
No animal grazing is allowed.
No site-specific risk assessment values are calculated.
Leaching and ground water impacts at the disposal site are not addressed.
Uncontrolled use of soil is not allowed without restrictions.

TABLE 1 - 2
INDUSTRIAL USE SOIL REMEDIAL GOALS, SWMU-SPECIFIC CHEMICALS OF CONCERN, AND ACCEPTABLE REPORTING LIMITS
NAVAL SURFACE WARFARE CENTER - CRANE

Parameter	SWMU Parameter Applicable To				IM Industrial Cleanup Goal (Human Health Only) (mg/kg)	Method of Analysis[1]	Analytical Reporting Limit (mg/kg)	
	MA	MB	ABG	RE			SWL	Quanterra
EXPLOSIVES								
Pentaerythritol tetranitrate (PETN)	X	X	X	X	To be determined by U.S. EPA 1998	8330	0.25	0.25
2,4,6-trinitrotoluene (TNT)	X	X	X	X	64	8330	0.50	0.25
Cyclotrimethylene trinitramine (RDX) (Hexahydro 1,3,5 trinitro 1,3,5 triazine)	X	X	X	X	17	8330	0.625	0.25
Cyclotetremethylene tetranitramine (HMX) (Octahydro 1,3,5,7 tetranitro 1,3,5,7 tetrazocine)	X	X	X	X	34,000	8330	1.60	0.25
Tetryl (Methyl 2,4,6 trinitro phenylnitroamine)	X	X	X	X	6,800	8330	0.38	0.25
Trinitrobenzene (TNB)	X	X	X	X	34	8330	0.575	0.25
1,3-Dinitrobenzene (DNB)	X	X	X	X	68	8330	0.49	0.25
~~1,2 DNB~~	~~X~~	~~X~~	~~X~~	~~X~~	~~270~~	~~8330~~		
~~1,4 DNB~~	~~X~~	~~X~~	~~X~~	~~X~~	~~270~~	~~8330~~		
Nitrobenzene (NB)*	X	X	X	X	94	8330	0.26	0.25
~~Dinitrotoluene (DNT) (total)~~	~~X~~	~~X~~	~~X~~	~~X~~	~~2.8~~	~~8330~~		

TABLE 1 - 2
INDUSTRIAL USE SOIL REMEDIAL GOALS, SWMU-SPECIFIC CHEMICALS OF CONCERN, AND ACCEPTABLE REPORTING LIMITS
NAVAL SURFACE WARFARE CENTER - CRANE

Parameter	SWMU Parameter Applicable To				IM Industrial Cleanup Goal (Human Health Only) (mg/kg)	Method of Analysis[1]	Analytical Reporting Limit (mg/kg)	
	MA	MB	ABG	RE			SWL	Quanterra
4-Amino 2,6 Dinitrotoluene (4-Am DNT)	X	X	X	X	680 **	8330	0.45	0.25
2-Amino 4,6 Dinitrotoluene (2-Am DNT)	X	X	X	X	1,400 ***	8330	0.40	0.25
2,4 DNT	X	X	X	X	1,400	8330	0.24	0.25
2,6-DNT	X	X	X	X	680	8330	0.39	0.25
2-nitrotoluene (2 NT)	X	X	X	X	6,800	8330	0.72	0.25
3 NT	X	X	X	X	6,800	8330	0.50	0.25
4 NT	X	X	X	X	6,800	8330	0.48	0.25

NOTES: MA = Mine Fill A, MB = Mine Fill B, ABG = Ammunition Burning Ground, and RE = Rockeye.
X = Parameter is a Chemical of Concern at the SWMU.
1. Analytical methods listed above are SW-846 methods, unless otherwise indicated.
* = Region 9 Preliminary Remedial Goal (PRG) value.
* * = Using 2,6 DNT as the surrogate for toxicity potential.
*** = Using 2,4 DNT as the surrogate for toxicity potential.

TABLE 1 - 2
INDUSTRIAL USE SOIL REMEDIAL GOALS, SWMU-SPECIFIC CHEMICALS OF CONCERN, AND ACCEPTABLE REPORTING LIMITS
NAVAL SURFACE WARFARE CENTER - CRANE

Parameter	SWMU Parameter Applicable To				IM Industrial Cleanup Goal (Human Health Only) (mg/kg)	Method of Analysis[1]	Analytical Reporting Limit (mg/kg)	
	MA	MB	ABG	RE			SWL	Quanterra
METALS								
Aluminum	X	X	X	X	100,000	6010	15.0	10.0
Antimony			X	X	680	6010	6.0	6.0
Arsenic	T	T	X T	X T	2.4	7060	0.80	0.50
Barium	X T	X T	X T	X T	100,000	6010	1.0	1.0
Beryllium			X	X	1.1	6010	0.5	0.2
Cadmium	X T	X T	X T	X T	850	6010	0.5	0.5
Chromium (total)	X T	X T	X T	X T	450	6010	1.0	1.0
Cobalt			X	X	97,000	6010	2.5	1.0
Copper			X	X	63,000	6010	2.5	2.0
Iron				X	EN-BG	6010	10.0	10.0
Lead	X T	X T	X T	X T	1,000	6010	10.0	5.0
Magnesium				X	EN-BG	6010	50.0	50.0
Nickel			X	X	34,000	6010	4.0	4.0
Zinc			X	X	100,000	6010	2.0	2.0
Mercury	X T	X T	X T	X T	510 **	7471	0.033	0.10

TABLE 1 - 2
INDUSTRIAL USE SOIL REMEDIAL GOALS, SWMU-SPECIFIC CHEMICALS OF CONCERN, AND ACCEPTABLE REPORTING LIMITS NAVAL SURFACE WARFARE CENTER - CRANE

Parameter	SWMU Parameter Applicable To				IM Industrial Cleanup Goal (Human Health Only) (mg/kg)	Method of Analysis[1]	Analytical Reporting Limit (mg/kg)	
	MA	MB	ABG	RE			SWL	Quanterra
Selenium	T	T	T	T	8,500	6010	25.0	20.0
Silver	T	T	T	T	8,500	6010	1.0	1.0
Manganese			X		43,000 *	6010	1.0	1.0
Thallium			X		140 *	6010	25.0	200
Vanadium			X		12,000 *	6010	2.0	1.0
Cyanide			X		14,000 *	9010	0.50	0.50

NOTES: MA = Mine Fill A, MB = Mine Fill B, ABG = Ammunition Burning Ground, and RE = Rockeye.
X = Parameter is a Chemical of Concern at the SWMU.
T = Parameter is a TCLP parameter for waste characterization.
1. Analytical methods listed above are SW-846 methods, unless otherwise indicated.
* = Region 9 PRG.

TABLE 1 - 2
INDUSTRIAL USE SOIL REMEDIAL GOALS, SWMU-SPECIFIC CHEMICALS OF CONCERN, AND ACCEPTABLE REPORTING LIMITS
NAVAL SURFACE WARFARE CENTER - CRANE

Parameter	SWMU Parameter Applicable To				IM Industrial Cleanup Goal (Human Health Only) (mg/kg)	Method of Analysis[1]	Analytical Reporting Limit (mg/kg)	
	MA	MB	ABG	RE			SWL	Quanterra
VOLATILE ORGANICS								
Dichloromethane (methylene chloride)	X	X	X		18	8260	0.005	0.005
Acetone	X	X	X		8,800	8260	0.005	0.010
Methyl ethyl ketone (2 - butanone)	XT	XT	XT		27,000	8260	0.005	0.010
Methyl isobutyl ketone (4 - methyl -2-pentanone)	X	X			2,800	8260	0.005	0.005
Toluene	X	X			880	8260	0.005	0.005
Xylene (total)	X	X		X	320	8260	0.005	0.005
Trans 1,3 dichloropropene				X	0.55 *	8260	0.005	0.005
1,1,1 Trichloroethane			X	X	3,000	8260	0.005	0.005
1,1,2 Trichloroethane			X	X	1.5	8260	0.005	0.005

TABLE 1 - 2
INDUSTRIAL USE SOIL REMEDIAL GOALS, SWMU-SPECIFIC CHEMICALS OF CONCERN, AND ACCEPTABLE REPORTING LIMITS
NAVAL SURFACE WARFARE CENTER - CRANE

Parameter	SWMU Parameter Applicable To				IM Industrial Cleanup Goal (Human Health Only) (mg/kg)	Method of Analysis[1]	Analytical Reporting Limit (mg/kg)	
	MA	MB	ABG	RE			SWL	Quanterra
1,1 Dichloroethylene			X		0.08 *	8260	0.005	0.005
1,2 Dichloroethylene			X		0.55 *	8260	0.010	0.005
Trichloroethylene (TCE)			X		7 *	8260	0.005	0.005
1,1,2,2 Tetrachloroethane			X T		1.1 *	8260	0.005	0.005
Vinyl Chloride			X T		0.035 *	8260	0.005	0.010
Chloroethane			X		1,600 *	8260	0.005	0.010
Chloroform	T		X T		0.53 *	8260	0.005	0.005
Tetrachloroethylene			X		17 *	8260	0.005	0.005
1,2 Dichloroethane	T				0.55 *	8260	0.005	0.005
1,1 Dichloroethane	T				1,700 *	8260	0.005	0.005

NOTES: MA = Mine Fill A, MB = Mine Fill B, ABG = Ammunition Burning Ground, and RE = Rockeye.
X = Parameter is a Chemical of Concern at the SWMU.
T = Parameter is a TCLP parameter for waste characterization.
1. Analytical methods listed above are SW-846 methods, unless otherwise indicated.
* = Region 9 PRG.

TABLE 1 - 2
INDUSTRIAL USE SOIL REMEDIAL GOALS, SWMU-SPECIFIC CHEMICALS OF CONCERN, AND ACCEPTABLE REPORTING LIMITS
NAVAL SURFACE WARFARE CENTER - CRANE

Parameter	SWMU Parameter Applicable To				IM Industrial Cleanup Goal (Human Health Only) (mg/kg)	Method of Analysis[1]	Analytical Reporting Limit (mg/kg)	
	MA	MB	ABG	RE			SWL	Quanterra
SEMI-VOLATILE ORGANICS								
Phenanthrene				X		8270	0.33	0.33
Fluoranthene				X	27,000	8270	0.33	0.33
Pyrene				X	100	8270	0.33	0.33
Bis (2-ethylhexyl) phthalate			X		140 *	8270	0.33	0.33
di-n-butyl phthalate			X		68,000 *	8270	0.33	0.33
N-Nitrosodiphenylamine			X		390 *	8270	0.33	0.33
Benzo (b) fluoranthene			X		2.6 *	8270	0.33	0.33
4 - Nitrophenol			X		NA	8270	1.60	1.60
Benzo (a) anthracene			X		2.6 *	8270	0.33	0.33
Chrysene			X		7 *	8270	0.33	0.33

NOTES: MA = Mine Fill A, MB = Mine Fill B, ABG = Ammunition Burning Ground, and RE = Rockeye.
X = Parameter is a Chemical of Concern at the SWMU.
T = Parameter is a TCLP parameter for waste characterization.
1. Analytical methods listed above are SW-846 methods, unless otherwise indicated.
* = Region 9 PRG.

TABLE 1 - 2
INDUSTRIAL USE SOIL REMEDIAL GOALS, SWMU-SPECIFIC CHEMICALS OF CONCERN, AND ACCEPTABLE REPORTING LIMITS NAVAL SURFACE WARFARE CENTER - CRANE

Parameter	SWMU Parameter Applicable To				IM Industrial Cleanup Goal (Human Health Only) (mg/kg)	Method of Analysis[1]	Analytical Reporting Limit (mg/kg)	
	MA	MB	ABG	RE			SWL	Quanterra
POLYCHLORINATED BIPHENYLS (PCBs)								
Arochlor 1242		X			0.34 #	8081/8082	0.033	0.050
Arochlor 1254		X			19 #	8081/8082	0.067	0.050
Arochlor 1260		X			0.34 #	8081/8082	0.067	0.050

NOTES: MA = Mine Fill A, MB = Mine Fill B, ABG = Ammunition Burning Ground, and RE = Rockeye.
X = Parameter is a Chemical of Concern at the SWMU.
T = Parameter is a TCLP parameter for waste characterization.
1. Analytical methods listed above are SW-846 methods, unless otherwise indicated.
= This is probably a single assay for PCBs; you cannot analyze PCBs in an old release to soil by assuming that the PCB conger/chlorine content will be similar to the original commercial product.

TABLE 1 - 2
INDUSTRIAL USE SOIL REMEDIAL GOALS, SWMU-SPECIFIC CHEMICALS OF CONCERN, AND ACCEPTABLE REPORTING LIMITS NAVAL SURFACE WARFARE CENTER - CRANE

Parameter	SWMU Parameter Applicable To				IM Industrial Cleanup Goal (Human Health Only) (mg/kg)	Method of Analysis[1]	Analytical Reporting Limit (mg/kg)
	MA	MB	ABG	RE			SWL
DIOXINS							
2,3,7,8 TCDD equivalents			X *		0.005 - 0.020 +	8290	0.000001

NOTES: MA = Mine Fill A, MB = Mine Fill B, ABG = Ammunition Burning Ground, and RE = Rockeye.
X = Parameter is a Chemical of Concern at the SWMU.
T = Parameter is a TCLP parameter for waste characterization.
* - Dioxin Soil Sampling is limited to a proximity around solvent burning areas.
1. Analytical methods listed above are SW-846 methods, unless otherwise indicated.
+ = This is U.S. EPA remedial policy for industrial areas (5-10 parts per billion); However, U.S. EPA may elect to adopt a more stringent State standard if one is available.

TABLE 1 - 2
INDUSTRIAL USE SOIL REMEDIAL GOALS, SWMU-SPECIFIC CHEMICALS OF CONCERN, AND ACCEPTABLE REPORTING LIMITS NAVAL SURFACE WARFARE CENTER - CRANE

Preliminary Exposure Scenarios and Land Use Options:

These values only address direct inhalation and ingestion.
Worker safety is not addressed.
No cummulative risk is calculated.
No agricultural crop use is allowed.
No residential use is allowed.
No gardening or food chain use is allowed.
No animal grazing is allowed.
No site-specific risk assessment values are calculated.
Leaching and ground water impacts at the disposal site are not addresed.
Uncontrolled use of soil is not allowed without restrictions.
Industrial construction exposure is not addressed.

TABLE 1- 3
ECOLOGICAL USE SOIL REMEDIAL GOALS, SWMU-SPECIFIC CHEMICALS OF CONCERN, AND ACCEPTABLE REPORTING LIMITS
NAVAL SURFACE WARFARE CENTER - CRANE

Parameter	SWMU Parameter Applicable To				IM Cleanup Goal (Ecological Only) (mg/kg)	Method of Analysis[1]	Analytical Reporting Limit (mg/kg)	
	MA	MB	ABG	RE			SWL	Quanterra
EXPLOSIVES								
Pentaerythritol tetranitrate (PETN)	X	X	X	X	To be determined by U.S. EPA in 1998	8330	0.25	0.25
2,4,6-trinitrotoluene (TNT)	X	X	X	X	No listing *	8330	0.50	0.25
Cyclotrimethylene trinitramine (RDX) (Hexahydro 1,3,5 trinitro 1,3,5 triazine)	X	X	X	X	No listing *	8330	0.625	0.25
Cyclotetremethylene tetranitramine (HMX) (Octahydro 1,3,5,7 tetranitro 1,3,5,7 tetrazocine)	X	X	X	X	No listing *	8330	1.60	0.25
Tetryl (Methyl 2,4,6 trinitro phenylnitroamine)	X	X	X	X	No listing *	8330	0.38	0.25
Trinitrobenzene (TNB)	X	X	X	X	0.00946 +	8330	0.575	0.25
1,3-Dinitrobenzene (DNB)	X	X	X	X	0.00748 +	8330	0.49	0.25
~~1,2 DNB~~	~~X~~	~~X~~	~~X~~	~~X~~	~~No listing *~~	~~8330~~	–	
~~1,4 DNB~~	~~X~~	~~X~~	~~X~~	~~X~~	~~No listing *~~	~~8330~~		
Nitrobenzene (NB)	X	X	X	X	0.0148 +	8330	0.26	0.25
~~Dinitrotoluene (DNT) (total)~~	~~X~~	~~X~~	~~X~~	~~X~~	~~0.000029 ++~~	~~8330~~		

TABLE 1- 3
ECOLOGICAL USE SOIL REMEDIAL GOALS, SWMU-SPECIFIC CHEMICALS OF CONCERN, AND ACCEPTABLE REPORTING LIMITS NAVAL SURFACE WARFARE CENTER - CRANE

Parameter	SWMU Parameter Applicable To				IM Cleanup Goal (Ecological Only) (mg/kg)	Method of Analysis[1]	Analytical Reporting Limit (mg/kg)	
	MA	MB	ABG	RE			SWL	Quanterra
4-Amino 2,6 Dinitrotoluene (4-Am DNT)	X	X	X	X	No listing *	8330	0.45	0.25
2-Amino 4,6 Dinitrotoluene (2-Am DNT)	X	X	X	X	No listing *	8330	0.40	0.25
2,4 DNT	X	X	X	X	0.033 +	8330	0.24	0.25
2,6-DNT	X	X	X	X	0.000029 ++	8330	0.39	0.25
2-nitrotoluene (2 NT)	X	X	X	X	No listing *	8330	0.72	0.25
3 NT	X	X	X	X	No listing *	8330	0.50	0.25
4 NT	X	X	X	X	No listing *	8330	0.48	0.25

NOTES: MA = Mine Fill A, MB = Mine Fill B, ABG = Ammunition Burning Ground, and RE = Rockeye.
X = Parameter is a Chemical of Concern at the SWMU.
1. Analytical methods listed above are SW-846 methods, unless otherwise indicated.
* = The Navy must do a literature search or see if similar surrogates are available to justify a cleanup goal for this parameter.
+ = U.S. EPA Region 5 toxicity information adjusted for shrews, back calculated for appropriate soil concentration.
++ = Based on shrews, extrapolated from dog data.

TABLE 1 - 3
ECOLOGICAL USE SOIL REMEDIAL GOALS, SWMU-SPECIFIC CHEMICALS OF CONCERN, AND ACCEPTABLE REPORTING LIMITS
NAVAL SURFACE WARFARE CENTER - CRANE

Parameter	SWMU Parameter Applicable To				IM Cleanup Goal (Ecological Only) (mg/kg)	Method of Analysis[1]	Analytical Reporting Limit (mg/kg)	
	MA	MB	ABG	RE			SWL	Quanterra
METALS								
Aluminum	X	X	X	X	No listing *	6010	15.0	10.0
Antimony			X	X	0.000771 +	6010	6.0	6.0
Arsenic	T	T	X T	X T	0.000796 +	7060	0.8	0.50
Barium	X T	X T	X T	X T	0.00562 +	6010	1.0	1.0
Beryllium			X	X	0.0665 +	6010	0.5	0.2
Cadmium	X T	X T	X T	X T	0.000519 +	6010	0.5	0.5
Chromium (total)	X T	X T	X T	X T	0.336 +	6010	1.0	1.0
Cobalt			X	X	0.0000052 ++	6010	2.5	1.0
Copper			X	X	0.00373 +	6010	2.5	2.0
Iron				X	No listing *	6010	10.0	10.0
Lead	X T	X T	X T	X T	0.00000862 ++	6010	10.0	5.0
Magnesium				X	No listing *	6010	50.0	50.0
Nickel			X	X	0.0505 +	6010	4.0	4.0
Zinc			X	X	0.305 +	6010	2.0	2.0
Mercury	X T	X T	X T	X T	0.00108 +	7471	0.033	0.10

TABLE 1 - 3
ECOLOGICAL USE SOIL REMEDIAL GOALS, SWMU-SPECIFIC CHEMICALS OF CONCERN, AND ACCEPTABLE REPORTING LIMITS
NAVAL SURFACE WARFARE CENTER - CRANE

Parameter	SWMU Parameter Applicable To				IM Cleanup Goal (Ecological Only) (mg/kg)	Method of Analysis[1]	Analytical Reporting Limit (mg/kg)	
	MA	MB	ABG	RE			SWL	Quanterra
Selenium	T	T	T	T	0.00015 +	6010	25.0	20.0
Silver	T	T	T	T	0.253 +	6010	1.0	1.0
Manganese			X		No listing *	6010	1.0	1.0
Thallium			X		**0.000308** +	6010	25.0	200
Vanadium			X		**0.00859** +	6010	2.0	1.0
Cyanide			X		**0.137** +++	9010	0.50	0.50

NOTES: MA = Mine Fill A, MB = Mine Fill B, ABG = Ammunition Burning Ground, and RE = Rockeye.
X = Parameter is a Chemical of Concern at the SWMU.
T = Parameter is a TCLP parameter for waste characterization.
1. Analytical methods listed above are SW-846 methods, unless otherwise indicated.
* = The Navy must do a literature search or see if similar surrogates are available to justify a cleanup goal for this parameter.
+ = U.S. EPA Region 5 toxicity information adjusted for shrews, back calculated for appropriate soil concentration.
++ = Based on shrews, extrapolated from sheep data.
+++ = U.S. EPA Region 5 toxicity information adjusted for voles, back calculated for appropriate soil concentration.

TABLE 1 - 3
ECOLOGICAL USE SOIL REMEDIAL GOALS, SWMU-SPECIFIC CHEMICALS OF CONCERN, AND ACCEPTABLE REPORTING LIMITS
NAVAL SURFACE WARFARE CENTER - CRANE

Parameter	SWMU Parameter Applicable To				IM Cleanup Goal (Ecological Only) (mg/kg)	Method of Analysis[1]	Analytical Reporting Limit (mg/kg)	
	MA	MB	ABG	RE			SWL	Quanterra
VOLATILE ORGANICS								
Dichloromethane (methylene chloride)	X	X	X		0.072 +	8260	0.005	0.005
Acetone	X	X	X		0.257 ++	8260	0.005	0.010
Methyl ethyl ketone (2 - butanone)	X T	X T	X T		9.22 ++	8260	0.005	0.010
Methyl isobutyl ketone (4 - methyl -2-pentanone)	X	X			58 +	8260	0.005	0.005
Toluene	X	X			0.2997 +	8260	0.005	0.005
Xylene (total)	X	X		X	1.25 +	8260	0.005	0.005
Trans 1,3 dichloropropene				X	0.00449 +	8260	0.005	0.005
1,1,1 Trichloroethane			X	X	0.2225 +	8260	0.005	0.005
1,1,2 Trichloroethane			X	X	0.3229 +	8260	0.005	0.005

TABLE 1 - 3
ECOLOGICAL USE SOIL REMEDIAL GOALS, SWMU-SPECIFIC CHEMICALS OF CONCERN, AND ACCEPTABLE REPORTING LIMITS NAVAL SURFACE WARFARE CENTER - CRANE

Parameter	SWMU Parameter Applicable To				IM Cleanup Goal (Ecological Only) (mg/kg)	Method of Analysis[1]	Analytical Reporting Limit (mg/kg)	
	MA	MB	ABG	RE			SWL	Quanterra
1,1 Dichloroethylene			X		0.00167 +	8260	0.005	0.005
1,2 Dichloroethylene			X		0.10245 +	8260	0.010	0.005
Trichloroethylene (TCE)			X		0.10604 +	8260	0.005	0.005
1,1,2,2 Tetrachloroethane			X T		0.00114 +	8260	0.005	0.005
Vinyl Chloride			X T		0.00729 +	8260	0.005	0.010
Chloroethane			X		No listing *	8260	0.005	0.010
Chloroform	T		X T		0.15574 +	8260	0.005	0.005
Tetrachloroethylene			X		0.45674 +	8260	0.005	0.005
1,2 Dichloroethane	T				0.23156 +	8260	0.005	0.005
1,1 Dichloroethane	T				0.00167 +	8260	0.005	0.005

NOTES: MA = Mine Fill A, MB = Mine Fill B, ABG = Ammunition Burning Ground, and RE = Rockeye.
X = Parameter is a Chemical of Concern at the SWMU.
T = Parameter is a TCLP parameter for waste characterization.
1. Analytical methods listed above are SW-846 methods, unless otherwise indicated.
* = The Navy must do a literature search or see if similar surrogates are available to justify a cleanup goal for this parameter.
+ = U.S. EPA Region 5 toxicity information adjusted for shrews, back calculated for appropriate soil concentration.
++ = U.S. EPA Region 5 toxicity information adjusted for voles, back calculated for appropriate soil concentration.

TABLE 1 - 3
ECOLOGICAL USE SOIL REMEDIAL GOALS, SWMU-SPECIFIC CHEMICALS OF CONCERN, AND ACCEPTABLE REPORTING LIMITS
NAVAL SURFACE WARFARE CENTER - CRANE

Parameter	SWMU Parameter Applicable To				IM Cleanup Goal (Ecological Only) (mg/kg)	Method of Analysis[1]	Analytical Reporting Limit (mg/kg)	
	MA	MB	ABG	RE			SWL	Quanterra
SEMI-VOLATILE ORGANICS								
Phenanthrene				X	0.13986 +	8270	0.33	0.33
Fluoranthene				X	4.32 +	8270	0.33	0.33
Pyrene				X	2.94 +	8270	0.33	0.33
Bis (2-ethylhexyl) phthalate			X		0.00152 +	8270	0.33	0.33
di-n-butyl phthalate			X		No listing *	8270	0.33	0.33
N-Nitrosodiphenylamine			X		0.0065 +	8270	0.33	0.33
Benzo (b) fluoranthene			X		0.1732 +	8270	0.33	0.33
4 - Nitrophenol			X		0.05779 +	8270	1.60	1.60
Benzo (a) anthracene			X		0.01088 +	8270	0.33	0.33
Chrysene			X		0.01088 #	8270	0.33	0.33

NOTES: MA = Mine Fill A, MB = Mine Fill B, ABG = Ammunition Burning Ground, and RE = Rockeye.
X = Parameter is a Chemical of Concern at the SWMU.
T = Parameter is a TCLP parameter for waste characterization.
1. Analytical methods listed above are SW-846 methods, unless otherwise indicated.
* = The Navy must do a literature search or see if similar surrogates are available to justify a cleanup goal for this parameter.
+ = U.S. EPA Region 5 toxicity information adjusted for shrews, backcalculated for appropriate soil concentration.
# = No data available; some reports use benzo (a) anthracene as surrogate data.

TABLE 1 - 3
ECOLOGICAL USE SOIL REMEDIAL GOALS, SWMU-SPECIFIC CHEMICALS OF CONCERN, AND ACCEPTABLE REPORTING LIMITS
NAVAL SURFACE WARFARE CENTER - CRANE

Parameter	SWMU Parameter Applicable To				IM Cleanup Goal (Ecological Only) (mg/kg)	Method of Analysis[1]	Analytical Reporting Limit (mg/kg)	
	MA	MB	ABG	RE			SWL	Quanterra
POLYCHLORINATED BIPHENYLS (PCBs)								
Arochlor 1242		X			0.00158 +	8081/8082	0.033	0.050
Arochlor 1254		X			0.00158 +	8081/8082	0.067	0.050
Arochlor 1260		X			0.00158 +	8081/8082	0.067	0.050

NOTES: MA = Mine Fill A, MB = Mine Fill B, ABG = Ammunition Burning Ground,and RE = Rockeye.
X = Parameter is a Chemical of Concern at the SWMU.
T = Parameter is a TCLP parameter for waste characterization.
1. Analytical methods listed above are SW-846 methods, unless otherwise indicated.
+ = U.S. EPA Region 5 toxicity information adjusted for shrews, backcalculated for appropriate soil concentration

TABLE 1 - 3
ECOLOGICAL USE SOIL REMEDIAL GOALS, SWMU-SPECIFIC CHEMICALS OF CONCERN, AND ACCEPTABLE REPORTING LIMITS
NAVAL SURFACE WARFARE CENTER - CRANE

Parameter	SWMU Parameter Applicable To				IM Cleanup Goal (Ecological Only) (mg/kg)	Method of Analysis[1]	Analytical Reporting Limit (mg/kg)
	MA	MB	ABG	RE			SWL
DIOXINS							
2,3,7,8 TCDD equivalents			X *		0.00000000079 +	8290	0.000001

NOTES: MA = Mine Fill A, MB = Mine Fill B, ABG = Ammunition Burning Ground,and RE = Rockeye.
X = Parameter is a Chemical of Concern at the SWMU.
T = Parameter is a TCLP parameter for waste characterization.
* = Dioxin Soil Sampling is limited to a proximity around solvent burning areas.
1. Analytical methods listed above are SW-846 methods, unless otherwise indicated.
+ = U.S. EPA Region 5 toxicity information adjusted for shrews, back calculated for appropriate soil concentration.

TABLE 1 -3
ECOLOGICAL USE SOIL REMEDIAL GOALS, SWMU-SPECIFIC CHEMICALS OF CONCERN, AND ACCEPTABLE REPORTING LIMITS
NAVAL SURFACE WARFARE CENTER - CRANE

Preliminary Exposure and Land Use Options:

These values only address direct ingestion in animals.
No cummulative risk is calculated.
No site-specific risk assessment values are calculated.
Leaching and ground water impacts at the disposal site are not addressed.
Uncontrolled use of soil is not allowed without restrictions.
Soil may not be placed in or along surface water bodies.
Soil may not be placed in erosion areas leading to surface water bodies.
Vegetation exposure is not addressed.

TABLE 1 - 4a
HUMAN DRINKING WATER USE REMEDIAL GOALS, SWMU-SPECIFIC CHEMICALS OF CONCERN, AND ACCEPTABLE REPORTING LIMITS
NAVAL SURFACE WARFARE CENTER - CRANE

Parameter	SWMU Parameter Applicable To				IM Drinking Water Cleanup Goal (Human Health Only) (ug/l)	Method of Analysis[1]	Analytical Reporting Limit (ug/l)	
	MA	MB	ABG	RE			SWL	Quanterra
EXPLOSIVES								
Pentaerythritrol tetranitrate (PETN)	X	X	X	X	To be determined by U.S. EPA 1998	8330	3.65	0.5
2,4,6-trinitrotoluene (TNT)	X	X	X	X	2.2	8330	0.12	0.10
Cyclotrimethylene trinitramine (RDX) (Hexahydro 1,3,5 trinitro 1,3,5 triazine)	X	X	X	X	0.61	8330	0.84	0.80
Cyclotetremethylene tetranitramine (HMX) (Octahydro 1,3,5,7 tetranitro 1,3,5,7 tetrazocine)	X	X	X	X	1,800	8330	1.0	1.0
Tetryl (Methyl 2,4,6 trinitro phenyl nitroamine)	X	X	X	X	370	8330	0.60	1.0
Trinitrobenzene (TNB)	X	X	X	X	1.8	8330	0.26	0.30
1,3-Dinitrobenzene (DNB)	X	X	X	X	3.7	8330	0.11	0.10
~~1,2-DNB~~	~~X~~	~~X~~	~~X~~	~~X~~	~~15 *~~	~~8330~~		
~~1,4-DNB~~	~~X~~	~~X~~	~~X~~	~~X~~	~~15 *~~	~~8330~~		
Nitrobenzene (NB)	X	X	X	X	3.4	8330	0.30	1.0
~~Dinitrotoluene (DNT) (total)~~	~~X~~	~~X~~	~~X~~	~~X~~	~~0.09~~	~~8330~~		

TABLE 1 - 4a
HUMAN DRINKING WATER USE REMEDIAL GOALS, SWMU-SPECIFIC CHEMICALS OF CONCERN, AND ACCEPTABLE REPORTING LIMITS
NAVAL SURFACE WARFARE CENTER - CRANE

Parameter	SWMU Parameter Applicable To				IM Drinking Water Cleanup Goal (Human Health Only) (ug/l)	Method of Analysis[1]	Analytical Reporting Limit (ug/l)	
	MA	MB	ABG	RE			SWL	Quanterra
4-Amino 2,6 Dinitrotoluene (4-Am DNT)	X	X	X	X	37 **	8330	0.06	0.10
2-Amino 4,6 Dinitrotoluene (2-Am DNT)	X	X	X	X	60.8 ***	8330	0.06	0.10
2,4 DNT	X	X	X	X	60.8	8330	0.04	0.10
2,6-DNT	X	X	X	X	37	8330	0.1	0.3
2-nitrotoluene (2 NT)	X	X	X	X	370	8330	0.78	1.0
3 NT	X	X	X	X	370	8330	0.65	1.0
4 NT	X	X	X	X	370	8330	0.65	1.0

NOTES: MA = Mine Fill A, MB = Mine Fill B, ABG = Ammunition Burning Ground, and RE = Rockeye.
X = Parameter is a chemical of Conceern at the SWMU.
1. Analytical methods listed above are SW-846 methods, unless otherwise indicated.
* = Region 9 Preliminary Remedial Goal (PRG) value.
** = Using 2,6 DNT as the surrogate for toxicity potential.
*** = Using 2,4 DNT as the surrogate for toxicity potential.

TABLE 1 - 4a
HUMAN DRINKING WATER USE REMEDIAL GOALS, SWMU-SPECIFIC CHEMICALS OF CONCERN, AND ACCEPTABLE REPORTING LIMITS
NAVAL SURFACE WARFARE CENTER - CRANE

Parameter	SWMU Parameter Applicable To				IM Drinking Water Cleanup Goal (Human Health Only) (ug/l)	Method of Analysis[1]	Analytical Reporting Limit (ug/l)	
	MA	MB	ABG	RE			SWL	Quanterra
METALS								
Aluminum	X	X	X	X	37,000	6010/6020	14	50
Antimony			X	X	6	7041/6020	4.0	2.0
Arsenic	T	T	X T	X T	0.038	7060/6020	4.0	2.0
Barium	X T	X T	X T	X T	2,000	6010/6020	1.0	1.0
Beryllium			X	X	0.016	6010/6020	1.0	1.0
Cadmium	X T	X T	X T	X T	1.1	7131/6020	1.0	1.0
Chromium (total)	X T	X T	X T	X T	100	6010/6020	1.9	1.0
Cobalt			X	X	2,200	6010/6020	2.8	1.0
Copper			X	X	1,400	6010/6020	8.3	1.0
Iron				X	300 #	6010/6020	18	50.0
Lead	X T	X T	X T	X T	4.0	7421/6020	3.0	1.0
Magnesium				X	EN-BG	6010/6020	25	50.0
Nickel			X	X	100	6010/6020	6.5	1.0
Zinc			X	X	11,000	6010/6020	11	2.0
Mercury	X T	X T	X T	X T	2	7470	0.12	0.2

TABLE 1 - 4a
HUMAN DRINKING WATER USE REMEDIAL GOALS, SWMU-SPECIFIC CHEMICALS OF CONCERN, AND ACCEPTABLE REPORTING LIMITS NAVAL SURFACE WARFARE CENTER - CRANE

Parameter	SWMU Parameter Applicable To				IM Drinking Water Cleanup Goal (Human Health Only) (ug/l)	Method of Analysis[1]	Analytical Reporting Limit (ug/l)	
	MA	MB	ABG	RE			SWL	Quanterra
Selenium	T	T	T	T	180	6010/6020	28	2.0
Silver	T	T	T	T	180	6010/6020	1.4	1.0
Manganese			X		50 #	6010/6020	1.2	1.0
Thallium			X		2 ##	7841/6020	2.0	1.0
Vanadium			X		260 ###	6010/6020	2.2	5.0
Cyanide			X		200 ##	9010	10	10

NOTES: MA = Mine Fill A, MB = Mine Fill B, ABG = Ammunition Burning Ground, and RE = Rockeye.
X = Parameter is a chemical of Conceern at the SWMU.
T = Parameter is a TCLP parameter for waste characterization.
1. Analytical methods listed above are SW-846 methods, unless otherwise indicated.
= Secondary MCL (Federal guideline only; check with State to determine if State has an enforable level).
= MCL.
= Region 9 PRG value.
EN-BG = Essential nutrient; individual sample levels must be compared to unimpacted background levels at the site.

TABLE 1 - 4a
HUMAN DRINKING WATER USE REMEDIAL GOALS, SWMU-SPECIFIC CHEMICALS OF CONCERN, AND ACCEPTABLE REPORTING LIMITS
NAVAL SURFACE WARFARE CENTER - CRANE

Parameter	SWMU Parameter Applicable To				IM Drinking Water Cleanup Goal (Human Health Only) (ug/l)	Method of Analysis[1]	Analytical Reporting Limit (ug/l)	
	MA	MB	ABG	RE			SWL	Quanterra
VOLATILE ORGANICS								
Dichloromethane (methylene chloride)	X	X	X		4.3	8260	5	1
Acetone	X	X	X		610	8260	5	5
Methyl ethyl ketone (2 - butanone)	XT	XT	XT		1,900	8260	5	5
Methyl isobutyl ketone (4 - methyl -2-pentanone)	X	X			160	8260	5	5
Toluene	X	X			720	8260	5	1
Xylene (total)	X	X		X	1,400	8260	5	1
Trans 1,3 dichloropropene				X	0.081	8260	5	1
1,1,1 Trichloroethane			X	X	200	8260	5	1
1,1,2 Trichloroethane			X	X	0.2	8260	5	1

TABLE 1 - 4a
HUMAN DRINKING WATER USE REMEDIAL GOALS, SWMU-SPECIFIC CHEMICALS OF CONCERN, AND ACCEPTABLE REPORTING LIMITS
NAVAL SURFACE WARFARE CENTER - CRANE

Parameter	SWMU Parameter Applicable To				IM Drinking Water Cleanup Goal (Human Health Only) (ug/l)	Method of Analysis[1]	Analytical Reporting Limit (ug/l)	
	MA	MB	ABG	RE			SWL	Quanterra
1,1 Dichloroethylene			X		7 *	8260	5	1
1,2 Dichloroethylene			X		70 **	8260	10	1
Trichloroethylene (TCE)			X		5 *	8260	5	1
1,1,2,2 Tetrachloroethane			X T		0.06 ***	8260	5	1
Vinyl Chloride			X X T		2 *	8260	5	1
Chloroethane			X		710 ***	8260	5	1
Chloroform	T		X T		80 *	8260	5	1
Tetrachloroethylene			X		5 *	8260	5	1
1,2 Dichloroethane	T				5 *	8260	5	1
1,1 Dichloroethane	T				810 ***	8260	5	1

NOTES: MA = Mine Fill A, MB = Mine Fill B, ABG = Ammunition Burning Ground, and RE = Rockeye.
X = Parameter is a chemical of Conceern at the SWMU.
T = Parameter is a TCLP parameter for waste characterization.
1. Analytical methods listed above are SW-846 methods, unless otherwise indicated.
* = MCL
** = MCL for cis- isomer.
*** = Region 9 PRG.

TABLE 1 - 4a
HUMAN DRINKING WATER USE REMEDIAL GOALS, SWMU-SPECIFIC CHEMICALS OF CONCERN, AND ACCEPTABLE REPORTING LIMITS NAVAL SURFACE WARFARE CENTER - CRANE

Parameter	SWMU Parameter Applicable To				IM Drinking Water Cleanup Goal (Human Health Only) (ug/l)	Method of Analysis[1]	Analytical Reporting Limit (ug/l)	
	MA	MB	ABG	RE			SWL	Quanterra
SEMI-VOLATILE ORGANICS								
Phenanthrene				X	1.0	8270	10	10
Fluoranthene				X	1,500	8270	10	10
Pyrene				X	180	8270	10	10
Bis (2-ethylhexyl) phthalate			X		6 *	8270	10	10
di-n-butyl phthalate			X		3,700 ***	8270	10	10
N-Nitrosodiphenylamine			X		14 ***	8270	10	10
Benzo (b) fluoranthene			X		0.2 **	8270	10	10
4 - Nitrophenol			X		NA	8270	50	50
Benzo (a) anthracene			X		0.2 **	8270	10	10
Chrysene			X		0.2 **	8270	10	10

NOTES: MA = Mine Fill A, MB = Mine Fill B, ABG = Ammunition Burning Ground, and RE = Rockeye.
X = Parameter is a chemical of Conceern at the SWMU.
T = Parameter is a TCLP parameter for waste characterization.
1. Analytical methods listed above are SW-846 methods, unless otherwise indicated.
* = MCL
** = Use the MCL for benzo[a]pyrene as the surrogate value.
*** = Region 9 PRG.

TABLE 1 - 4a
HUMAN DRINKING WATER USE REMEDIAL GOALS, SWMU-SPECIFIC CHEMICALS OF CONCERN, AND ACCEPTABLE REPORTING LIMITS
NAVAL SURFACE WARFARE CENTER - CRANE

Parameter	SWMU Parameter Applicable To				IM Drinking Water Cleanup Goal (Human Health Only) (ug/l)	Method of Analysis[1]	Analytical Reporting Limit (ug/l)	
	MA	MB	ABG	RE			SWL	Quanterra
POLYCHLORINATED BIPHENYLS (PCBs)								
Arochlor 1242		X			0.0087 #	8081	1.0	1.0
Arochlor 1254		X			0.0087 #	8081	2.0	1.0
Arochlor 1260		X			0.0087 #	8081	2.0	1.0

NOTES: MA = Mine Fill A, MB = Mine Fill B, ABG = Ammunition Burning Ground, and RE = Rockeye.
X = Parameter is a chemical of Conceern at the SWMU.
T = Parameter is a TCLP parameter for waste characterization.
1. Analytical methods listed above are SW-846 methods, unless otherwise indicated.
= This is probably only a single analytical assay; you cannot analyze water soluable PCBs by assuming that the PCB pattern will be similar to the original commercial product. The MCL for PCB is 0.5 ug/l.

TABLE 1 - 4a
HUMAN DRINKING WATER USE REMEDIAL GOALS, SWMU-SPECIFIC CHEMICALS OF CONCERN, AND ACCEPTABLE REPORTING LIMITS NAVAL SURFACE WARFARE CENTER - CRANE

Preliminary Exposure Scenarios and Land Use Options:

These values only address direct ingestion.
No cummulative risk is calculated.
Water values for rock washing using final rinse sample, does not reflect if certain rocks still have soil attached to them.
Rocks may not be placed in stream beds or along shorelines.
Rocks may not be placed in areas where leaching or erosion are not addressed.
No site-specific risk assessment values are calculated.
Leaching and ground water impacts at the disposal site are not addressed.
Rock wash values not meeting these values must have rocks buried and mapped, instead of direct exposure on the ground.
Uncontrolled use of rocks is not allowed without restrictions.

TABLE 1 - 4b
ECOLOGICAL SURFACE WATER USE REMEDIAL GOALS, SWMU-SPECIFIC CHEMICALS OF CONCERN, AND ACCEPTABLE REPORTING LIMITS
NAVAL SURFACE WARFARE CENTER - CRANE

Parameter	SWMU Parameter Applicable To				IM Cleanup Goal (Ecological Only) (ug/l)	Method of Analysis[1]	Analytical Reporting Limit (ug/l)	
	MA	MB	ABG	RE			SWL	Quanterra
EXPLOSIVES								
Pentaerythritrol tetranitrate (PETN)	X	X	X	X	To be determined by U.S. EPA 1998	8330	3.65	0.5
2,4,6-trinitrotoluene (TNT)	X	X	X	X	No listing *	8330	0.12	0.10
Cyclotrimethylene trinitramine (RDX) (Hexahydro 1,3,5 trinitro 1,3,5 triazine)	X	X	X	X	No listing *	8330	0.84	0.80
Cyclotetremethylene tetranitramine (HMX) (Octahydro 1,3,5,7 tetranitro 1,3,5,7 tetrazocine)	X	X	X	X	No listing *	8330	1.0	1.0
Tetryl (Methyl 2,4,6 trinitro phenyl nitramine)	X	X	X	X	No listing *	8330	0.60	1.0
Trinitrobenzene (TNB)	X	X	X	X	No listing *	8330	0.26	0.30
1,3-Dinitrobenzene (DNB)	X	X	X	X	2.36 +	8330	0.11	0.10
~~1,2 DNB~~	~~X~~	~~X~~	~~X~~	~~X~~	~~No listing *~~	~~8330~~		
~~1,4 DNB~~	~~X~~	~~X~~	~~X~~	~~X~~	~~No listing *~~	~~8330~~		
Nitrobenzene (NB)	X	X	X	X	740 ++	8330	0.30	1.0
~~Dinitrotoluene (DNT) (total)~~	~~X~~	~~X~~	~~X~~	~~X~~	~~No listing *~~	~~8330~~		

TABLE 1 - 4b
ECOLOGICAL SURFACE WATER USE REMEDIAL GOALS, SWMU-SPECIFIC CHEMICALS OF CONCERN, AND ACCEPTABLE REPORTING LIMITS
NAVAL SURFACE WARFARE CENTER - CRANE

Parameter	SWMU Parameter Applicable To				IM Cleanup Goal (Ecological Only) (ug/l)	Method of Analysis[1]	Analytical Reporting Limit (ug/l)	
	MA	MB	ABG	RE			SWL	Quanterra
4-Amino 2,6 Dinitrotoluene (4-Am DNT)	X	X	X	X	No listing *	8330	0.06	0.10
2-Amino 4,6 Dinitrotoluene (2-Am DNT)	X	X	X	X	No listing *	8330	0.06	0.10
2,4 DNT	X	X	X	X	230 +	8330	0.04	0.10
2,6-DNT	X	X	X	X	42 +	8330	0.1	0.3
2-nitrotoluene (2 NT)	X	X	X	X	No listing *	8330	0.78	1.0
3 NT	X	X	X	X	No listing *	8330	0.65	1.0
4 NT	X	X	X	X	No listing *	8330	0.65	1.0

NOTES: MA = Mine Fill A, MB = Mine Fill B, ABG = Ammunition Burning Ground, and RE = Rockeye.
X = Parameter is a Chemical of Concern at the SWMU.
1. Analytical methods listed above are SW-846 methods, unless otherwise noted.
* = No listing available; the Navy must do a literature search or see if similar surrogates are available for justification for the goal for this parameter.
+ = Interim Water Quality Criteria.
++ = Lowest Chronic Criteria.

TABLE 1 - 4b
ECOLOGICAL SURFACE WATER USE REMEDIAL GOALS, SWMU-SPECIFIC CHEMICALS OF CONCERN, AND ACCEPTABLE REPORTING LIMITS
NAVAL SURFACE WARFARE CENTER - CRANE

Parameter	SWMU Parameter Applicable To				IM Cleanup Goal (Ecological Only) (ug/l)	Method of Analysis[1]	Analytical Reporting Limit (ug/l)	
	MA	MB	ABG	RE			SWL	Quanterra
METALS								
Aluminum	X	X	X	X	No listing *	6010/6020	14	50
Antimony			X	X	30 ++	6010/6020	12	2.0
Arsenic	T	T	X T	X T	53	7060/6020	4.0	2.0
Barium	X T	X T	X T	X T	No listing *	6010/6020	1.0	1.0
Beryllium			X	X	5.3	6010/6020	1.0	1.0
Cadmium	X T	X T	X T	X T	0.0216 ++	6010/6020	1.3	1.0
Chromium (total)	X T	X T	X T	X T	11.7	6010/6020	1.9	1.0
Cobalt			X	X	5.0	6010/6020	2.8	1.0
Copper			X	X	5.99	6010/6020	8.3	1.0
Iron				X	No listing *	6010/6020	18	50.0
Lead	X T	X T	X T	X T	1.30	6010/6020	12	1.0
Magnesium				X	No listing *	6010/6020	25	50.0
Nickel			X	X	36.79	6010/6020	6.5	1.0
Zinc			X	X	27.57	6010/6020	11	2.0
Mercury	X T	X T	X T	X T	0.0069	7470	0.12	0.2

TABLE 1 - 4b
ECOLOGICAL SURFACE WATER USE REMEDIAL GOALS, SWMU-SPECIFIC CHEMICALS OF CONCERN, AND ACCEPTABLE REPORTING LIMITS
NAVAL SURFACE WARFARE CENTER - CRANE

Parameter	SWMU Parameter Applicable To				IM Cleanup Goal (Ecological Only) (ug/l)	Method of Analysis[1]	Analytical Reporting Limit (ug/l)	
	MA	MB	ABG	RE			SWL	Quanterra
Selenium	T	T	T	T	5.0	7740/6020	4.0	2.0
Silver	T	T	T	T	0.06	6010/6020	1.4	1.0
Manganese			X			6010/6020	1.2	1.0
Thallium			X		**0.28**	6010/6020	48	1.0
Vanadium			X		**No Listing** *	6010/6020	2.2	5.0
Cyanide			X		**4.96**	9010	10	10

NOTES: MA = Mine Fill A, MB = Mine Fill B, ABG = Ammunition Burning Ground, and RE = Rockeye.
X = Parameter is a Chemical of Concern at the SWMU.
T = Parameter is a TCLP parameter for waste characterization.
1. Analytical methods listed above are SW-846 methods, unless otherwise noted.
* = No listing available; the Navy must do a literature search or see if similar surrogates are available for justification for the goal for this parameter.
+ = Interim Water Quality Criteria.
++ = Lowest Chronic Criteria.

TABLE 1 - 4b
ECOLOGICAL SURFACE WATER USE REMEDIAL GOALS, SWMU-SPECIFIC CHEMICALS OF CONCERN, AND ACCEPTABLE REPORTING LIMITS
NAVAL SURFACE WARFARE CENTER - CRANE

Parameter	SWMU Parameter Applicable To				IM Cleanup Goal (Ecological Only) (ug/l)	Method of Analysis[1]	Analytical Reporting Limit (ug/l)	
	MA	MB	ABG	RE			SWL	Quanterra
VOLATILE ORGANICS								
Dichloromethane (methylene chloride)	X	X	X		45	8260	5	1
Acetone	X	X	X		122,000	8260	5	5
Methyl ethyl ketone (2 - butanone)	XT	XT	XT		26,000	8260	5	5
Methyl isobutyl ketone (4 - methyl -2-pentanone)	X	X			3,680	8260	5	5
Toluene	X	X			253	8260	5	1
Xylene (total)	X	X		X	117.0	8260	5	1
Trans 1,3 dichloropropene				X	7.9 ++	8260	5	1
1,1,1 Trichloroethane			X	X	88	8260	5	1
1,1,2 Trichloroethane			X	X	650	8260	5	1

TABLE 1 - 4b
ECOLOGICAL SURFACE WATER USE REMEDIAL GOALS, SWMU-SPECIFIC CHEMICALS OF CONCERN, AND ACCEPTABLE REPORTING LIMITS NAVAL SURFACE WARFARE CENTER - CRANE

Parameter	SWMU Parameter Applicable To				IM Cleanup Goal (Ecological Only) (ug/l)	Method of Analysis[1]	Analytical Reporting Limit (ug/l)	
	MA	MB	ABG	RE			SWL	Quanterra
1,1 Dichloroethylene			X		**78**	8260	5	1
1,2 Dichloroethylene			X		**310**	8260	10	1
Trichloroethylene (TCE)			X		**75**	8260	5	1
1,1,2,2 Tetrachloroethane			X T		**13**	8260	5	1
Vinyl Chloride			X T		**9.2**	8260	5	1
Chloroethane			X		**230,000**	8260	5	1
Chloroform	T		X T		**79**	8260	5	1
Tetrachloroethylene			X		**8.9**	8260	5	1
1,2 Dichloroethane	T				**190**	8260	5	1
1,1 Dichloroethane	T				**No Listing** *	8260	5	1

NOTES: MA = Mine Fill A, MB = Mine Fill B, ABG = Ammunition Burning Ground, and RE = Rockeye.
X = Parameter is a Chemical of Concern at the SWMU.
T = Parameter is a TCLP parameter for waste characterization.
1. Analytical methods listed above are SW-846 methods, unless otherwise noted.
* = No listing available; the Navy must do a literature search or see if similar surrogates are available for justification for the goal for this parameter.
+ = Interim Water Quality Criteria.
++ = Lowest Chronic Criteria.

TABLE 1 - 4b
ECOLOGICAL SURFACE WATER USE REMEDIAL GOALS, SWMU-SPECIFIC CHEMICALS OF CONCERN, AND ACCEPTABLE REPORTING LIMITS
NAVAL SURFACE WARFARE CENTER - CRANE

Parameter	SWMU Parameter Applicable To				IM Cleanup Goal (Ecological Only) (ug/l)	Method of Analysis[1]	Analytical Reporting Limit (ug/l)	
	MA	MB	ABG	RE			SWL	Quanterra
SEMI-VOLATILE ORGANICS								
Phenanthrene				X	2.1	8270	10	10
Fluoranthene				X	8.9	8270	10	10
Pyrene				X	0.3 +	8270	10	10
Bis (2-ethylhexyl) phthalate			X		**8.4**	8270	10	10
di-n-butyl phthalate			X		**3.0**	8270	10	10
N-Nitrosodiphenylamine			X		**13**	8270	10	10
Benzo (b) fluoranthene			X		**12.6** +++	8270	10	10
4 - Nitrophenol			X		**35**	8270	50	50
Benzo (a) anthracene			X		**1.17** +++	8270	10	10
Chrysene			X		**1.37** +++	8270	10	10

NOTES: MA = Mine Fill A, MB = Mine Fill B, ABG = Ammunition Burning Ground, and RE = Rockeye.
X = Parameter is a Chemical of Concern at the SWMU.
T = Parameter is a TCLP parameter for waste characterization.
1. Analytical methods listed above are SW-846 methods, unless otherwise noted.
* = No listing available; the Navy must do a literature search or see if similar surrogates are available for justification for the goal for this parameter.
+ = Interim Water Quality Criteria.
++ = Lowest Chronic Criteria.
+++ = **Based on mink, extrapolated from rodent data.**

TABLE 1 - 4b
ECOLOGICAL SURFACE WATER USE REMEDIAL GOALS, SWMU-SPECIFIC CHEMICALS OF CONCERN, AND ACCEPTABLE REPORTING LIMITS NAVAL SURFACE WARFARE CENTER - CRANE

Parameter	SWMU Parameter Applicable To				IM Cleanup Goal (Ecological Only) (ug/l)	Method of Analysis[1]	Analytical Reporting Limit (ug/l)	
	MA	MB	ABG	RE			SWL	Quanterra
POLYCHLORINATED BIPHENYLS (PCBs)								
Arochlor 1242		X			No listing *#	8081	1.0	1.0
Arochlor 1254		X			No listing *#	8081	1.0	1.0
Arochlor 1260		X			No listing *#	8081	1.0	1.0

NOTES: MA = Mine Fill A, MB = Mine Fill B, ABG = Ammunition Burning Ground, and RE = Rockeye.
X = Parameter is a Chemical of Concern at the SWMU.
T = Parameter is a TCLP parameter for waste characterization.
1. Analytical methods listed above are SW-846 methods, unless otherwise noted.
* = No listing available; the Navy must do a literature search or see if similar surrogates are available for justification for the goal for this parameter.
+ = Interim Water Quality Criteria.
++ = Lowest Chronic Criteria.
# = PCB (CAS # 1336-36-3) value 0.000029 ug/l.

TABLE 1 - 4b
ECOLOGICAL SURFACE WATER USE REMEDIAL GOALS, SWMU-SPECIFIC CHEMICALS OF CONCERN, AND ACCEPTABLE REPORTING LIMITS
NAVAL SURFACE WARFARE CENTER - CRANE

Preliminary Exposure and Land Use Options:

These values only address direct ingestion in animals.
No cummulative risk is calculated.
No site-specific risk assessment values are calculated.
Leaching and ground water impacts at the disposal site are not addressed.
Uncontrolled use of washed rocks is not allowed without restrictions.
Washed rocks may not be placed in or along surface water bodies.
Washed rocks may not be placed in erosion areas leading to surface water bodies.
Vegetation exposure is not addressed.

TABLE 1-5
DISCHARGE LIMITS FOR POND WATER WITHOUT PRETREATMENT

Parameter	Method of Analysis[1]	Discharge To WWTP Acceptance[2] Level (lbs/day)	Discharge To Surface Acceptance Level (NPDES monthly limit - mg/L)	Acceptable Reporting Limit (~~ug/L~~mg/L)	
				SWL	Quanterra
Explosives					
TNT	8330	HMX+RDX+TNT = 0.17 lbs/day Max. HMX+RDX+TNT = 2.0 mg/l Max.	-	~~6.4~~0.0064 (High Level Method) ~~0.12~~ 0.00012(Low Level Method)	0.0001
RDX	8330	as above	-	~~14.0~~ 0.0014(High Level Method) ~~0.84~~ 0.00084(Low Level Method)	0.0008
HMX	8330	as above	-	~~13.0~~ 0.0013(High Level Method) ~~1.0~~ 0.001(Low Level Method)	0.001
Nitrates	300.0	-	90 Max.	~~1,000~~1	0.05
Other parameters					
BOD	EPA 405.1	4.6	10 Ave/15 Max	~~5,000~~5	5
COD	EPA 410.4	-	10 Ave/15 Max	~~10,000~~10	10
TSS	EPA 160.2	4.6	10 Ave/15 Max	~~10,000~~10	5
Ammonia-N (May 1 to Nov. 30)	EPA 350.2	0.9	3 Ave/ 6 Max	~~1,000~~1	0.10
Ammonia-N (Dec. 1 to Apr. 30)	EPA 350.2	1.8	3 Ave/ 6 Max	~~1,000~~1	0.10

TABLE 1-5 (Continued)
DISCHARGE LIMITS FOR POND WATER WITHOUT PRETREATMENT

Parameter	Method of Analysis[1]	Discharge To WWTP Acceptance[2] Level (lbs/day)	Discharge To Surface Acceptance Level (NPDES monthly limit - mg/L)	Acceptable Reporting Limit (~~ug/L~~mg/L)	
				SWL	Quanterra
Oil & Grease	EPA 413.1		26 Ave/ 52 Max	~~5000~~.5	5
pH	Field Measure-ment	-	5.0 to 11.0 units	n/a	n/a
Cadmium	6010/6020	0.0009	-	~~4.00~~.004	0.001
Chromium	6010/6020	0.18	-	~~5.00~~.005	0.001
Copper	6010/6020	0.006	-	~~10.00~~.01	0.001
Lead	6010/6020	0.004	-	~~40.00~~.04	0.001
Nickel	6010/6020	0.05	-	~~10.00~~.01	0.001
Zinc	6010/6020	0.09	-	~~5510.00~~.01	0.002
Silver	6010/6020	0.004	-	~~5.00~~.005	0.001
Mercury	7470	0.00003	-	~~0.150~~.00015	0.0002
Total Cyanide	9010	0.003	-	~~10,000~~10	0.010

Notes:
1. *Analytical methods listed above are SW-846 methods, unless otherwise indicated.*
2. Based on NSWC Crane Memorandum dated September 26, 1997 "Current Waste Water Dumping Guidelines" NSWC Crane Code 0957 to Code 09511 (NSWC Crane, 1997a)

n/a: not applicable

benzo(b)fluoranthene.

Dioxins: 2,3,7,8-TCDD Equivalents

The constituents of concern for Rockeye (SWMU-10/15) are listed below. They are derived from the RFI Phase III Soils Report for Rockeye [ACOE, 1992].

Explosives: PETN; TNT; RDX; HMX; Tetryl; TNB; 1,3-DNB; NB; 4-Am-DNT; 2-Am-DNT; 2,4-DNT; 2,6-DNT; 2-NT; 3-NT; and 4-NT.

Note: 2,4 DNT and NB will also be evaluated for waste characterization of toxicity characteristic.

Metals: Aluminum; antimony; arsenic; barium; beryllium; cadmium; chromium (total); cobalt; copper; iron; lead; magnesium; nickel; mercury; and zinc.

Note: The following list of metals will be evaluated for waste characterization of Toxicity Characteristic (TC): Arsenic, Barium, Cadmium, Chromium, Lead, Mercury, Selenium, and Silver.

VOCs: trans-1,3,dichloropropene; xylene (total); 1,1,1-trichloroethane; 1,1,2-trichloroethane.

Note: The following list of VOCs will be evaluated for waste characterization of Toxicity Characteristic (TC): Methyl Ethyl Ketone (MEK), Chloroform, 1,2 Dichloroethane, and 1,1 Dichloroethane.

SVOCs: Phenanthrene; fluoranthene; and pyrene.

1.4.2.2 Remedial Goals

Human health (residential and industrial) and ecological risk-based remedial goals have been established using preliminary remedial goals and data quality levels by U.S. EPA Region 5 (U.S. EPA, 1998). Cumulative risk, other risk scenarios, and toxicity testing results are not considered in the U.S. EPA evaluation. These remedial goals are presented in Tables 1-1 through 1-3. The analytical methods and analytical reporting limits are also shown in these Tables. Method reporting limits for each of the target constituents are provided by Southwest Laboratory of Oklahoma and Quanterra Environmental Services and have been evaluated by Morrison Knudsen Corporation (MK) for acceptability for data quality.

Tables 1-4a and 1-4b provides the cleanup goals for rinse water generated after washing the rocks at the excavation site. If the rinse water meets these cleanup levels or non-detect levels the rocks are determined to be acceptable for use as backfill

material; or the rocks may be triple rinsed, crushed, and sent to the Bioremediation Facility for composting.

Table 1-5 provides the allowable discharge limits without pretreatment, analytical methods, and analytical reporting limits for water generated in the storm-water retention ponds at the Bioremediation Facility.

1.4.2.3 Performance Goals

The 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 (U.S. EPA, 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.

Figures 1-2 and 1-3 of the Full-Scale Operations Plan (MK, 1998b) provide flowcharts describing the process flow path for decision-making of the excavation site soil and the treated soil respectively. Similarly, Figure 1-4 of the Full-Scale Operations Plan (MK, 1998b) provides a flowchart describing the process flow path for decision-making of decontamination of rocks.

Toxicity tests of the treated soil will be performed, as specified in Appendix G of this QAPP, at a later date to determine if treated soil meeting the remedial goals listed in Tables 1-1 (residential levels); 1-2 (industrial levels); or 1-3 (ecological levels) can be used as soil amendment for revegetation at the excavation sites or other NSWC Crane EPD-approved areas.

1.4.3 Process Monitoring Tasks

During full-scale operation process parameters will be measured in the field to evaluate the effectiveness of the compost process. The individual field measurement data collection tasks are summarized in Table 1-9 and described below.

TABLE 3-1
QA OBJECTIVES FOR
PRECISION, ACCURACY AND COMPLETENESS
VOLATILE ORGANICS (Method 8260)

Data Quality Parameter	Matrix	Method of Determination	Frequency	QC Limits	
				SWL	Quanterra
Precision	Water	Laboratory MS/MSD	One per 20 samples (one per analytical batch)	RPD = ~~0-300~~-40 %	RPD = 0 - 35%
	Water	Laboratory Control Sample/Duplicate	One per 20 rinse events	RPD = ~~0-300~~-40 %	RPD = 0 - 35%
	Soil, compost	Laboratory MS/MSD	One per 20 samples (one per analytical batch)	RPD = ~~0-400~~-45 %	RPD = 0 - 35%
	Soil, compost	Laboratory Control Sample/Duplicate	One per 20 samples (one per analytical batch)	RPD = ~~0-400~~-45 %	RPD = 0 - 35%
Accuracy	Water	Surrogate	All samples and standards	~~See Appendix C, Page C2-21~~Recovery = 86-118 %	Recovery = 75 - 121 %
	Water	Laboratory Control Sample/Duplicate	One per 20 samples (one per analytical batch)	~~See Appendix C, Page C2-17~~Recovery = 23-185 %	Recovery = 70 - 130 %
	Water	Laboratory MS/MSD	One per 20 samples (one per analytical batch)	~~See Appendix C, Page C2-17~~Recovery = 23-185 %	Recovery = 70 - 130 %
	Soil, Compost	Surrogate	All samples and standards	~~See Appendix C, Page C2-21~~Recovery = 74-120 %	Recovery = 75 - 120 %
	Soil, Compost	Laboratory Control Sample/Duplicate	One per 20 samples (one per analytical batch)	~~See Appendix C, Page C2-17~~Recovery = 50-151 %	Recovery = 68 - 130 %
	Soil, Compost	Laboratory MS/MSD	One per 20 samples (one per analytical batch)	~~See Appendix C, Page C2-17~~Recovery = 50-151 %	Recovery = 68 - 130 %
Completeness	Water, Soil, Compost	Valid Samples	All Samples	90 ~~percent~~%	90 %

Notes: MS/MSD = matrix spike/ matrix spike duplicate; RPD = relative percent difference.
Water refers to the analysis of storm water, retention pond water, rock wash water, and various blanks.

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

Data Quality Parameter	Matrix	Method of Determination	Frequency	QC Limits	
				SWL	Quanterra
Precision	Water	Laboratory MS/MSD	One per 20 samples (one per analytical batch)	~~See Appendix C, Page C2-41~~RPD = 0-72 %	RPD = 0 - 35%
	Water	Laboratory Control Sample/Duplicate	One per 20 samples (one per analytical batch)	RPD = ~~0 - 400~~ 72 %	RPD = 0 - 35%
	Soil, Compost	Laboratory MS/MSD	One per 20 samples (one per analytical batch)	~~See Appendix C, Page C2-41~~RPD = 0-40 %	RPD = 0 - 35%
	Soil-, Compost	Laboratory Control Sample/Duplicate	One per 20 samples (one per analytical batch)	RPD = 0-40 %	RPD = 0 - 35%
Accuracy	Water	Surrogate	All samples and standards	~~60-140% using 3,4-DNT~~Recovery = 16-160 %	Recovery = 40 - 135%
	Water	Laboratory Control Sample/Duplicate	One per 20 samples (one per analytical batch)	~~See Appendix C, Page C2-41~~Recovery = 16-160 %	Recovery = 65 - 135%
	Water	Laboratory MS/MSD	One per 20 samples (one per analytical batch)	~~See Appendix C, Page C2-41~~Recovery = 16-160 %	Recovery = 65 - 135%
	Soil, Compost	Surrogate	All samples and standards	~~60-140% using 3,4-DNT~~Recovery = 19-137 %	Recovery = 40 - 135%
	Soil, Compost	Laboratory Control Sample/Duplicate	One per 20 samples (one per analytical batch)	~~See Appendix C, Page C2-41~~Recovery = 19-137 %	Recovery = 65 - 135%
	Soil, Compost	Laboratory MS/MSD	One per 20 samples (one per analytical batch)	~~See Appendix C, Page C2-41~~ Recovery = 19-137 %	Recovery = 65 - 135%
Completeness	Water, Soil, Compost	Valid Samples	All Samples	90 ~~percent~~%	90%

Notes: MS/MSD = matrix spike/ matrix spike duplicate; RPD = relative percent difference.
Water refers to the analysis of storm water, retention pond water, rock wash water, and various blanks.

TABLE 3-3
QA OBJECTIVES FOR
PRECISION, ACCURACY AND COMPLETENESS
METALS (Method 6010)

Data Quality Parameter	Matrix	Method of Determination	Frequency	QC Limits	
				SWL	Quanterra
Precision	Water, Soil, Compost	Laboratory Duplicate	One per 20 samples (one per analytical batch)	RPD = ~~0-200~~-15 % water 0-25 % soil	RPD - 20% water 35% soil
Accuracy	Water, Soil, Compost	Laboratory MS/MSD or Laboratory MS and Sample Duplicate	One per 20 samples (one per analytical batch)	Recovery = ~~80-120~~77-117 %	Recovery = 71 - 128%
	Water	Laboratory Control Sample/Duplicate	One per 20 samples (one per analytical batch)	Recovery = ~~80-120~~77-110 %	Recovery = 71 - 128%
	Soil, Compost	Laboratory Control Sample/Duplicate	One per 20 samples (one per analytical batch)	Recovery = ~~80-120~~78-117 %	Recovery = 80 - 120%
Completeness	Water, Soil, Compost	Valid Samples	All Samples	90 ~~percent~~%	90 %

Notes:
MS/MSD = matrix spike/ matrix spike duplicate; RPD = relative percent difference.
Water refers to the analysis of storm water, retention pond water, rock wash water, and various blanks.

TABLE 3-4
QA OBJECTIVES FOR
PRECISION, ACCURACY AND COMPLETENESS
SEMIVOLATILE ORGANICS (Method 8270)

Data Quality Parameter	Matrix	Method of Determination	Frequency	QC Limits	
				SWL	Quanterra
Precision	Water	Laboratory MS/MSD	One per 20 samples (one per analytical batch)	~~See Appendix G, Page G2-222~~RPD = 0-65 %	RPD = 0 - 30%
	Water	Laboratory Control Sample/Duplicate	One per 20 samples (one per analytical batch)	~~See Appendix G, Page G2-222~~RPD = 0-65 %	RPD = 0 - 30%
	Soil, Compost	Laboratory MS/MSD	One per 20 samples (one per analytical batch)	~~See Appendix G, Page G2-222~~RPD = 0-19 %	RPD = 0 - 40%
	Soil , Compost	Laboratory Control Sample/Duplicate	One per 20 samples (one per analytical batch)	~~See Appendix G, Page G2-222~~RPD = 0-19 %	RPD = 0 - 40%
Accuracy	Water	Surrogate	All samples and standards	~~See Appendix G, Page G2-222~~Recovery = 10-141 %	Recovery = 10 - 141%
	Water	Laboratory Control Sample/Duplicate	One per 20 samples (one per analytical batch)	~~See Appendix G, Page G2-222~~Recovery = 10-135 %	Recovery = 10 - 150%
	Water	Laboratory MS/MSD	One per 20 samples (one per analytical batch)	~~See Appendix G, Page G2-222~~Recovery = 10-135 %	Recovery = 10 - 150%
	Soil, Compost	Surrogate	All samples and standards	~~See Appendix G, Page G2-222~~Recovery = 18-137 %	Recovery = 18 - 137%
	Soil, Compost	Laboratory Control Sample/Duplicate	One per 20 samples (one per analytical batch)	~~See Appendix G, Page G2-222~~Recovery = 34-115 %	Recovery = 10 - 150%
	Soil, Compost	Laboratory MS/MSD	One per 20 samples (one per analytical batch)	~~See Appendix G, Page G2-222~~Recovery = 34-115 %	Recovery = 10 - 150%
Completeness	Water, Soil, Compost	Valid Samples	All Samples	90 ~~percent~~%	90%

Notes: MS/MSD = matrix spike/ matrix spike duplicate; RPD = relative percent difference.
Water refers to the analysis of storm water, retention pond water, rock wash water, and various blanks.

TABLE 3-5
QA OBJECTIVES FOR
PRECISION, ACCURACY AND COMPLETENESS
PCBs (Method 8081, 8082)

Data Quality Parameter	Matrix	Method of Determination	Frequency	QC Limits	
				SWL	Quanterra
Precision	Water	Laboratory MS/MSD	One per 20 samples (one per analytical batch)	~~See Appendix G, Page G2-258~~RPD = 0-85 %	RPD = 0 - 35%
	Water	Laboratory Control Sample/Duplicate	One per 20 samples (one per analytical batch)	~~See Appendix G, Page G2-258~~RPD = 0-85 %	RPD = 0 - 35%
	Soil, Compost	Laboratory MS/MSD	One per 20 samples (one per analytical batch)	~~See Appendix G, Page G2-258~~RPD = 0-32 %	RPD = 0 - 35%
	Soil, Compost	Laboratory Control Sample/Duplicate	One per 20 samples (one per analytical batch)	~~See Appendix G, Page G2-258~~RPD = 0-30 %	RPD = 0 - 35%
Accuracy	Water	Surrogate	All samples and standards	~~See Appendix G, Page G2-258~~Recovery = 34-126 %	Recovery = 30 - 150%
	Water	Laboratory Control Sample/Duplicate	One per 20 samples (one per analytical batch)	~~See Appendix G, Page G2-258~~Recovery = 25-126 %	Recovery = 70 - 130%
	Water	Laboratory MS/MSD	One per 20 samples (one per analytical batch)	~~See Appendix G, Page G2-258~~Recovery = 25-126 %	Recovery = 70 - 130%
	Soil, Compost	Surrogate	All samples and standards	~~See Appendix G, Page G2-258~~Recovery = 47-125 %	Recovery = 30 - 150%
	Soil, Compost	Laboratory Control Sample/Duplicate	One per 20 samples (one per analytical batch)	~~See Appendix G, Page G2-258~~Recovery = 15-139 %	Recovery = 70 - 130%
	Soil, Compost	Laboratory MS/MSD	One per 20 samples (one per analytical batch)	~~See Appendix G, Page G2-258~~Recovery = 15-139 %	Recovery = 70 - 130%

TABLE 3-5 (continued)
QA OBJECTIVES FOR
PRECISION, ACCURACY AND COMPLETENESS
PCBs (Method 8081, 8082)

Data Quality Parameter	Matrix	Method of Determination	Frequency	QC Limits	
				SWL	Quanterra
Completeness	Water, Soil, Compost	Valid Samples	All Samples	90 ~~percent~~%	90 %

Notes: MS/MSD = matrix spike/ matrix spike duplicate; RPD = relative percent difference.
Water refers to the analysis of storm water, retention pond water, rock wash water, and various blanks.

TABLE 3-6
QA OBJECTIVES FOR
PRECISION, ACCURACY AND COMPLETENESS
DIOXINS (Method 8290)

Data Quality Parameter	Matrix	Method of Determination	Frequency	QC Limits SWL
Precision	Water	Laboratory Control Sample/Duplicate	One per 20 samples (one per analytical batch)	~~See Appendix G, Page G2-169~~RPD = 0-80 %
	Soil-, Compost	Laboratory Control Sample/Duplicate	One per 20 samples (one per analytical batch)	~~See Appendix G, Page G2-169~~RPD = 0-80 %
Accuracy	Water	Laboratory Control Sample/Duplicate	One per 20 samples (one per analytical batch)	~~See Appendix G, Page G2-169~~Recovery = 24-140 %
	Soil, Compost	Laboratory Control Sample/Duplicate	One per 20 samples (one per analytical batch)	~~See Appendix G, Page G2-169~~Recovery = 40-135 %
Completeness	Water, Soil	Valid Samples	All Samples	90 ~~percent~~%

Notes: Water refers to the analysis of storm water, retention pond water, rock wash water, and various blanks.

TABLE 3-7
QA OBJECTIVES FOR
PRECISION, ACCURACY AND COMPLETENESS
MISCELLANEOUS PARAMETERS

Parameter	Laboratory	SOP Number[1]	Section (in SOP)	Page Number Appendix C-
TSS	SWL	SWL-IN-600	Section 12	C2-118
	Quanterra	CORP-WC-0002SAC	Section 17	C4-425
COD	SWL	SWL-IN-403	Section 10	C2-125
	Quanterra	LM-CAL-1019	Section 9	C4-430
BOD	SWL	SWL-IN-415	Section 8	C2-131
	Curtis & Tompkins	Curtis-Wet\BOD.sop	Section 4	C4-432
Oil and Grease	SWL	SWL-06-105	Section 9	C2-334
	Quanterra	LN-CRL-1026	Section 10	C4-597
Nitrate	SWL	SWL-IN-030B	Section 10	C2-325
	Quanterra	SAC-WC-0036	Section 9	C4-580
Ammonia-N	SWL	SWL-IN-020	Section 9	C2-319
	Quanterra	SAC-WC-0105	Section 9	C4-566

~~NOTE~~Note: ~~1~~SOPs are laboratory confidential.— SOPs are ~~laboratory confidential. SOPs are~~ provided by Southwest Laboratories (Broken Arrow, Oklahoma) and Quanterra Environmental Services (Sacramento, California).

TABLE 6-1
CALIBRATION CRITERIA - LABORATORY METHODS

Parameter	Laboratory	SOP Number	Section Number (in SOP)	Page Number Appendix C-
VOCs	SWL	SWL-OV-303	Section 8.0	C2-8
	Quanterra	CORP-MS-0002	Section 10	C4-22
Explosives	SWL	SWL-OL-200	Section 7.4	C2-38
	Quanterra	SAC-LC-0001	Section 11	C4-84
Metals	SWL	SWL-IN-205	Section 7.4	C2-87
	Quanterra	CORP-MT-0001 SAC-MT-0001 CORP-MT-0003	Section 10 Section 10 Section 10	C4-151 C4-196 C4-258
Mercury	SWL	SWL-IN-207	Section 8.5	C2-93
	Quanterra	CORP-MT-0007	Section 10	C4-301
Total Cyanide	SWL	SWL-IN-	Section IV	C2-347
	Quanterra	LM-CAL-1034	Section 8	C4-613
SVOCs	SWL	SWL-OS-500	Section 10.7	C2-216
	Quanterra	CORP-MS-0001	Section 10	C4-456
PCBs	SWL	SWL-OP-412	Section 8.4	C2-252
	Quanterra	CORP-GC-0001	Section 10	C4-504
Dioxins	SWL	SWL-OD-205	Section 8.8	C2-160
Oil and grease	SWL	SWL-OG-105	Section 7.2	C2-332
	Quanterra	LM-CRL-1026	NA	NA
Nitrates	SWL	SWL-IN-030B	Section 8.0	C2-324
	Quanterra	SAC-WC-0036	Section 10	C4-581
Ammonia-N	Quanterra	SAC-WC-0105	Section 10	C4-568
TSS, COD, BOD	(none required)			

TABLE 7-1
SUMMARY OF SOPs

Parameters[1]	SOP Number[2] SWL	SOP Number[2] Quanterra	Analytical Method
SOIL			
Explosives	SWL-OL-200	SAC-LC-0001	8330
VOCs	SWL-OV-303	CORP-MS-0002	8260
Metals	SWL-IN-205	CORP-MT-0001 SAC-MT-0001 CORP-MT-0003	6010 6020 7000 series
Mercury	SWL-IN-207	CORP-MT-0003-SAC	7471
SVOCs	SWL-OS-500	CORP-MS-0001	8270
PCBs	SWL-OP-412	CORP-GC-0001	~~8084~~8082
Dioxins	SWL-OD-205	NA	~~2890~~8290
TCLP-Metals	SWL-IN-700	NA	1311/6010
TCLP-VOCs	SWL-OV-309	NA	1311/8260
WATER			
Explosives	SWL-OL-200	SAC-LC-0001	8330
VOCs	SWL-OV-303	CORP-MS-0002	8260
Metals	SWL-IN-205	CORP-MT-0001 SAC-MT-0001 CORP-MT-0003	6010 6020 7000 series
Total Cyanide	SWL-IN-307	LM-CAL-1034	9010
TSS	SWL-IN-600	CORP-WC-0002-SAC	EPA 160.2
COD	SWL-IN-603	LM-CAL-1019	EPA 410.2
5-day BOD	SWL-IN-415	CURTIS-WET\BOD.SOP	EPA 405.1
Oil and Grease	SWL-OG-105	LM-CRL-1026	EPA 413.1
Nitrates	SWL-IN-030B	SAC-WC-0036	EPA 300.0
Ammonia-N	SWL-IN-020	SAC-WC-0105	EPA 350.2

Notes:
1. For a complete listing of individual compounds refer to Section 1.4.2.1 and Tables 1-1, 1-2, 1-3, 1-4a, and 1-4b.
2. SOPs are laboratory confidential. SOPs are provided by Southwest Laboratories (Broken Arrow, Oklahoma) and Quanterra Environmental Services (Sacramento, California)

APPENDIX B
STANDARD METHOD DETECTION LIMITS FOR ANALYTICAL METHODS

TABLE OF CONTENTS

	PAGE
Method Detection Limits for Southwest Laboratories	B-1
Method Detection Limits for Quanterra Environmental Services	B-14

LOCATION OF REPORTING LIMITS FOR ANALYTICAL METHODS FOR QUANTERRA ENVIRONMENTAL SERVICES

Parameter	SOP Number	LIQUID REPORTING LIMITS	SOLID REPORTING LIMITS
VOCs	CORP-MS-0002	Table A1, page C4-57	Table A1, page C4-57
Explosives	SAC-LC-0001	Table 1, page C4-136b	Table 1, page C4-136b
Metals by ICP	CORP-MT-0001	Table 1, page C4-165	Table 1, page C4-165
Metals by ICP/MS	SAC-MT-0001	Appendix A, page C4-206	Appendix A, page C4-206
Metals by GFAA	CORP-MT-0003 SAC	Table 1, page C4-318	Table 1, page C4-318
Mercury	CORP-MT-0007	Table 1, page C4-273	Table 1, page C4-273
Total Cyanide	LM-CAL-1034	Section1, page C4-608	Section1, page C4-608
SVOCs	CORP-MS-0001	Table 1, page C4-472	Table 1, page C4-472
PCBs	CORP-GC-0001	Table C-1, page C4-552	Table C-1, page C4-552
Oil and grease	LM-CRL-1026	Section1, page C4-592	Section1, page C4-592
Nitrates	SAC-WC-0036	Section 1, page C4-574	NA
Ammonia	SAC-WC-0105	Section 1, page C4-562	NA
TSS	CORP-WC-0002-SAC	Section 13, page C4-419	NA
COD	LM-CAL-1019	Section 1, page C4-426	NA
BOD	CURTIS-WET \BOD.SOP	Section1, page C4-432	NA

APPENDIX C
LABORATORY STANDARD OPERATING PROCEDURES

TABLE OF CONTENTS

SECTION		PAGE
1	PREVENTIVE MAINTENANCE FOR SWL	C1-1
2	STANDARD OPERATING PROCEDURES FOR ANALYSIS FOR SWL	C2-1
	VOLATILE ORGANICS BY METHOD 8260	C2-1
	NITROAROMATICS AND NITROAMINES BY 8330	C2-29
	METALS BY METHOD 6010	C2-47
	(PAGES C2-83 TO C2-86 ARE NOT INCLUDED)	
	MERCURY BY METHOD 7471/7470	C2-87
	TCLP NON-VOLATILE EXTRACTION BY METHOD 1311	C2-100
	TOTAL SUSPENDED SOLIDS BY METHOD 160.21	C2-110
	CHEMICAL OXYGEN DEMAND BY METHOD 410.2	C2-121
	BIOCHEMICAL OXYGEN DEMAND BY METHOD 405.1	C2-127
	(PAGES C2-132 TO C2-135 ARE NOT INCLUDED)	
	DIOXINS BY METHOD 8290	C2-136
	SEMIVOLATILE ORGANICS BY METHOD 8270	C2-197
	PCBs BY METHOD 8081	C2-243
	TCLP VOLATILE EXTRACTION BY METHOD 1311	C2-264
	TOTAL KJELDAHL NITROGEN BY METHOD 351.3	C2-281
	TOTAL ORGANIC CARBON BY METHOD 9060 (soil)	C2-289
	TOTAL ORGANIC CARBON BY METHOD 415.1 (water)	C2-296

TABLE OF CONTENTS (Continued)

SECTION		PAGE
	AMMONIA-N BY METHOD 350.2	C2-307
	NITRATE-N BY METHOD 300.0	C2-320
	OIL AND GREASE BY METHOD 413.1	C2-327
	TOTAL CYANIDE	C2-347
	HOMOGENIZATION OF SOIL, SEDIMENT AND SLUDGE SAMPLES	C2-356
	CORRECTIVE ACTION PROCEDURE	C2-360
	PETN IN SOIL AND WATER BY HPLC	C2-380
3	PREVENTIVE MAINTENANCE	C3-1
4	STANDARD OPERATING PROCEDURES FOR ANALYSIS FOR QUANTERRA	C4-1
	VOLATILE ORGANICS BY METHOD 8260	C4-1
	NITROAROMATICS AND NITROAMINES BY 8330	C4-67
	METALS BY METHOD 6010	C4-137
	METALS BY METHOD 6020	C4-186
	MERCURY BY METHOD 7471/7470	C4-246
	METALS BY METHOD 7000	C4-287
	TCLP EXTRACTION BY METHOD 1311	C4-340
	TOTAL SUSPENDED SOLIDS BY METHOD 160.2	C4-394
	CHEMICAL OXYGEN DEMAND BY METHOD 410.2	C4-426
	BIOCHEMICAL OXYGEN DEMAND BY METHOD 405.1	C4-432
	SEMIVOLATILE ORGANICS BY METHOD 8270	C4-443
	PCBs BY METHOD 8082	C4-497

TABLE OF CONTENTS (Continued)

SECTION	PAGE
AMMONIA-N BY METHOD 350.2	C4-561
NITRATE-N BY METHOD 300.0	C4-573
OIL AND GREASE BY METHOD 413.1	C4-591
TOTAL CYANIDE DISTILLATION	C4-599
TOTAL CYANIDE AUTOMATED ANALYSIS	C4-607
CORRECTIVE ACTION PROCEDURE	C4-618

SECTION 3
PREVENTIVE MAINTENANCE FOR QUANTERRA ENVIRONMENTAL SERVICES

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

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

8.11 Preventive Maintenance and Service

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

8.11.1 Analytical Instrumentation and Equipment

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

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

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

The record of maintenance shall include at a minimum:

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

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

8.11.2 Facilities

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

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

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

8.11.3 Frequency of Maintenance

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

8.12 Other Requirements

8.12.1 Water

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

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

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

8.12.2 Compressed Air and Gases

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

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

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

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

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

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

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

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

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

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

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

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

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

TABLE 8.11-7
Instrument Maintenance Schedule
ICP[1]

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

TABLE 8.11-14
Instrument Maintenance Schedule
Sonicator [1]

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

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

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

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

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

TABLE 8.11-17
Instrument Maintenance Schedule
Ovens[1]

Daily	As Needed
Temperatures checked and logged.	Electronics serviced.

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

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

TABLE 8.11-19
Instrument Maintenance Schedule
Turbidimeter[1]

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

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

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

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

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

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

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

TABLE 8.11-23
Instrument Maintenance Schedule
Spectrophotometer[1]

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

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

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

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

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

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

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

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

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

Footnotes to Preventive Maintenance Tables

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

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

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

SECTION 4
STANDARD OPERATING PROCEDURES FOR QUANTERRA ENVIRONMENTAL SERVICES

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QUANTERRA STANDARD OPERATING PROCEDURE

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

(SUPERSEDES: REVIS ON 1)

Prepared by: Richard Burrows

Reviewed by: _____
Technology Standardization Committee

Approved by: _____
Director, Quality Assurance

Approved by: _____
Director, Environmental Health and Safety

Approved by: _____
Management

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TABLE OF CONTENTS

1. SCOPE AND APPLICATION 4

2. SUMMARY OF METHOD 4

3. DEFINITIONS 5

4. INTERFERENCES 6

5. SAFETY 7

6. EQUIPMENT AND SUPPLIES 8

7. REAGENTS AND STANDARDS 10

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE 11

9. QUALITY CONTROL 19

10. CALIBRATION AND STANDARDIZATION 22

11. PROCEDURE 26

12. DATA ANALYSIS AND CALCULATIONS 31

13. METHOD PERFORMANCE 38

14. POLLUTION PREVENTION 39

15. WASTE MANAGEMENT 39

16. REFERENCES 39

17. MISCELLANEOUS 39

APPENDIX A

APPLICATION OF METHOD 524.2 AND METHOD 8260A TO THE DRINKING WATER ANALYTE LIST 55

APPENDIX B

ANALYSIS OF VOLATILE ORGANICS BY METHOD 624 63

LIST OF TABLES

Table 1 Quanterra Primary Standard and Reporting Limits

Table 2 Quanterra Primary Standard Calibration Levels

Table 3 Quanterra Appendix IX Standard and Reporting Limits

Table 4 Quanterra Appendix IX Standard Calibration Levels

Table 5 Reportable Analytes for Quanterra Standard Tests, Primary Standard

Table 6 Reportable Analytes for Quanterra Standard Tests, Appendix IX Standard

Table 7 Internal Standards

Table 8 Surrogate Standards

Table 9 Matrix Spike / LCS Standards

Table 10 BFB Tune Criteria

Table 11 SPCC Compounds and Minimum Response Factors

Table 12 CCC Compounds

Table 13 Characteristic Ions

Table A-1 Quanterra Drinking Water List Standard and Reporting Limits

Table A-2 Internal Standards, Drinking Water List

Table A-3 Surrogate Standards, Drinking Water List

Table A-4 Calibration Levels, Drinking Water List

Table B-1 Method 624 Analytes and Reporting Limits

Table B-2 Method 624 QC Acceptance Criteria

1. SCOPE AND APPLICATION

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

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

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

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

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

2. SUMMARY OF METHOD

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

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

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

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

3. DEFINITIONS

3.1. Batch

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

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

3.2. Method Blank

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

3.3. Laboratory Control Sample (LCS)

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

3.4. Surrogates

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

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

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

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

3.6. Calibration Check Compound (CCC)

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

3.7. System Performance Check Compounds (SPCC)

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

4. INTERFERENCES

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

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

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

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

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

5. SAFETY

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

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

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

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

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

5.4.2. Chemicals known to be flammable are: Methanol.

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

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

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

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

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

6. EQUIPMENT AND SUPPLIES

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

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

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

6.4. Glassware:

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

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

6.5. Spatula: Stainless steel.

6.6. Disposable pipets: Pasteur.

6.7. pH paper: Wide range.

6.8. Gases:

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

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

6.8.3. Compressed air: Used for instrument pneumatics.

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

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

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

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

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

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

6.10. Gas Chromatograph/Mass Spectrometer System:

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

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

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

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

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

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

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

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

7. REAGENTS AND STANDARDS

7.1. Reagents

7.1.1. Methanol: Purge and Trap Grade, High Purity

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

7.2. Standards

7.2.1. Calibration Standard

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

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

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

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

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

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

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

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

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

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

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

8.6.2. Seal the bottle and attach a label.

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

8.6.4. Ship with appropriate sampling instructions.

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

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

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

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

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

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

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

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

8.7. Low level procedure

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

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

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

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

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

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

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

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

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

8.8. Unpreserved soils

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

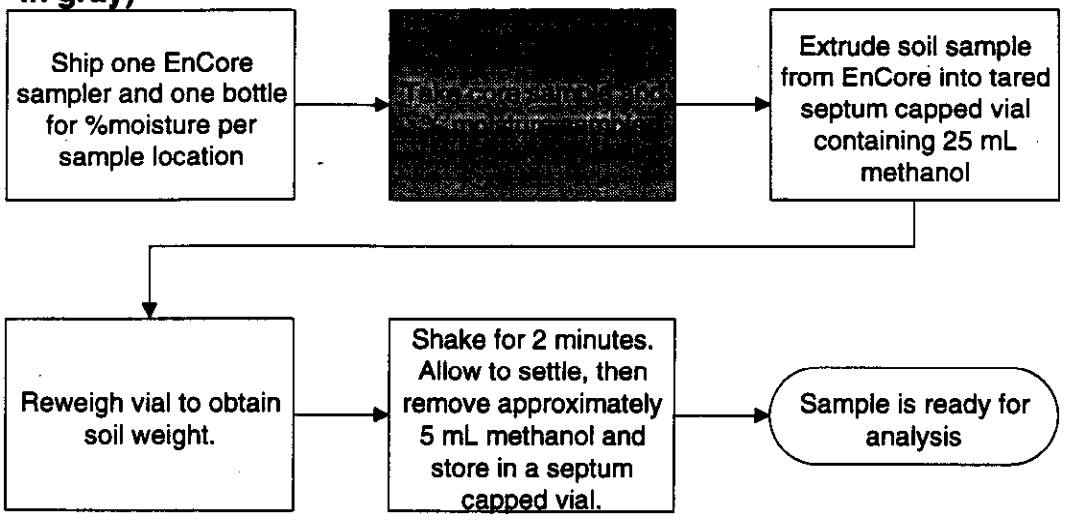
8.9. Aqueous samples are stored in glass containers with Teflon lined septa at 4°C +/- 2°C, with minimum headspace.

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

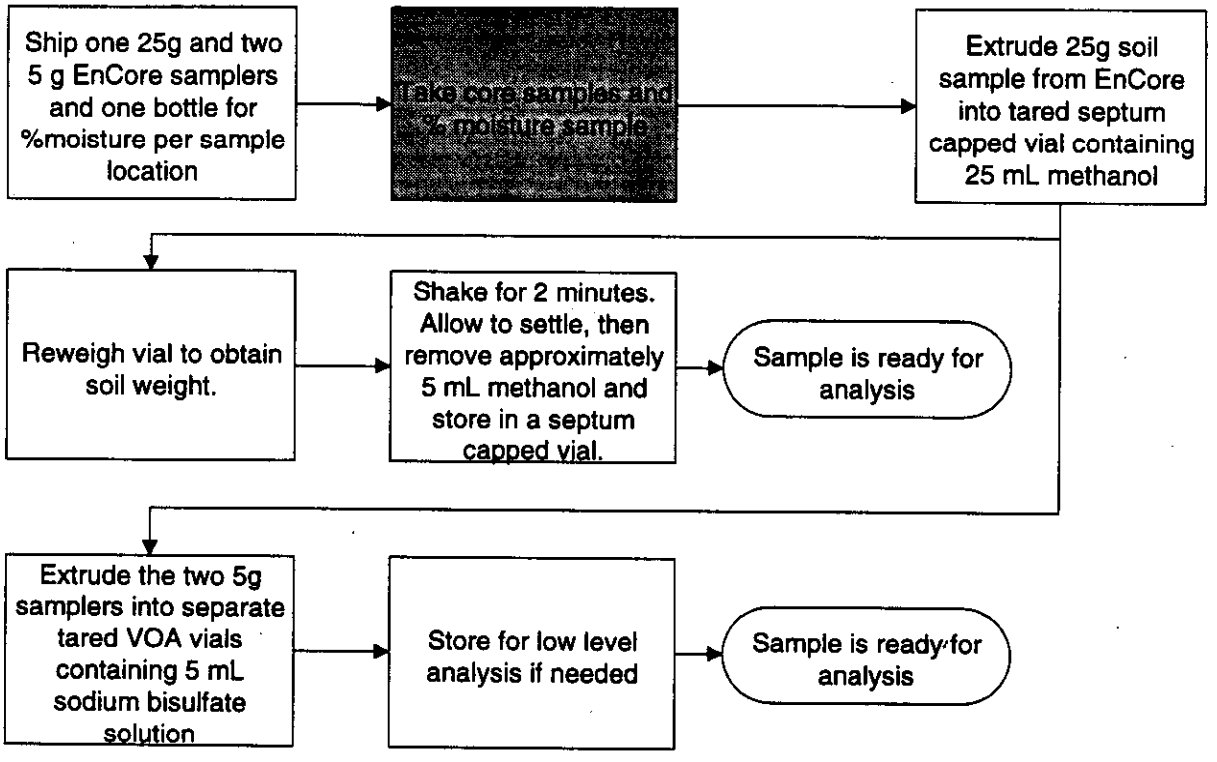
8.11. The maximum holding time is 14 days from sampling until the sample is analyzed. (Samples that are found to be unpreserved still have a 14 day holding time. However they should be analyzed as soon as possible. The lack of preservation should be addressed in the case narrative). Maximum holding time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

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

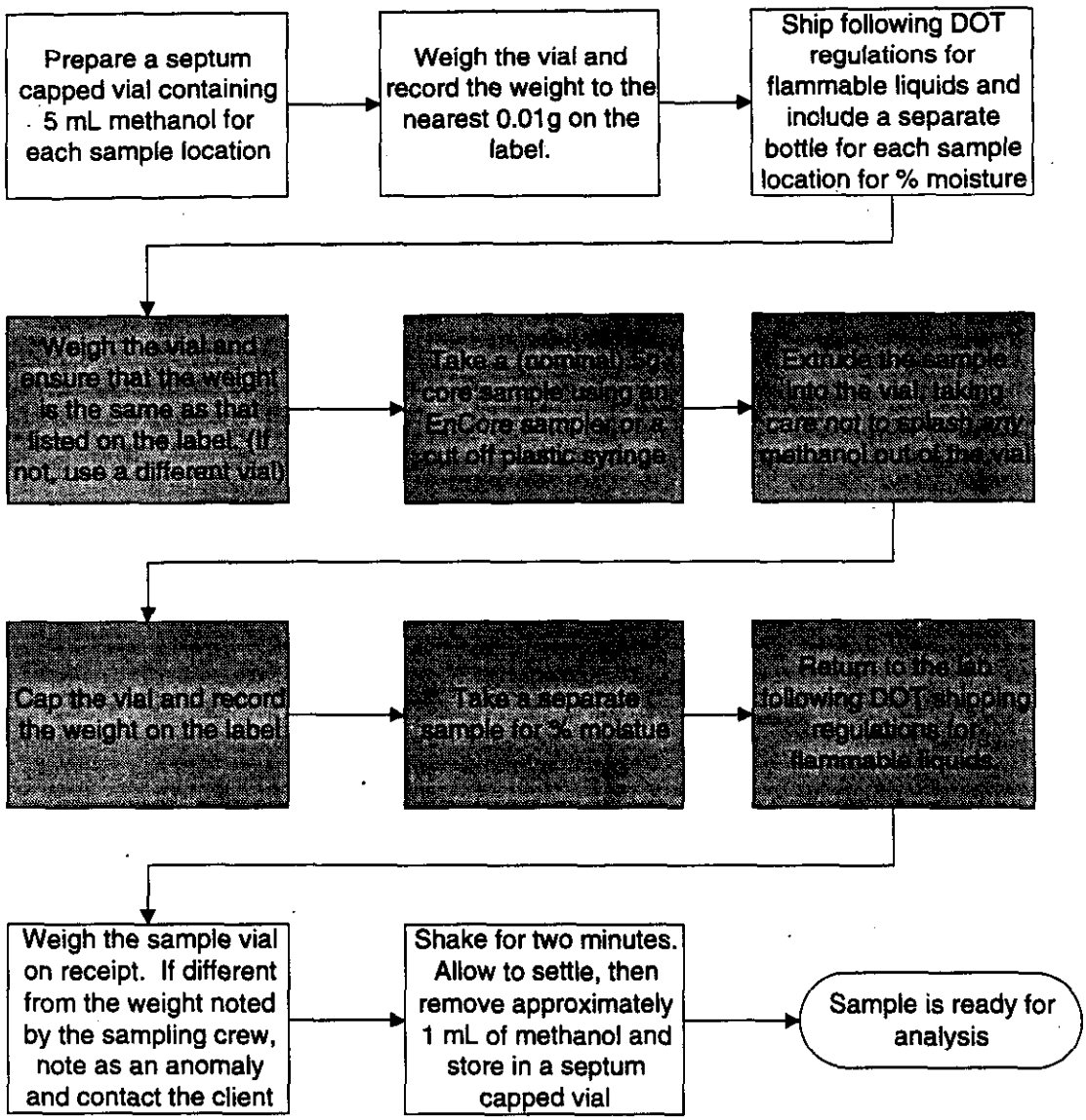
EnCore procedure when low level is not required (field steps in gray)



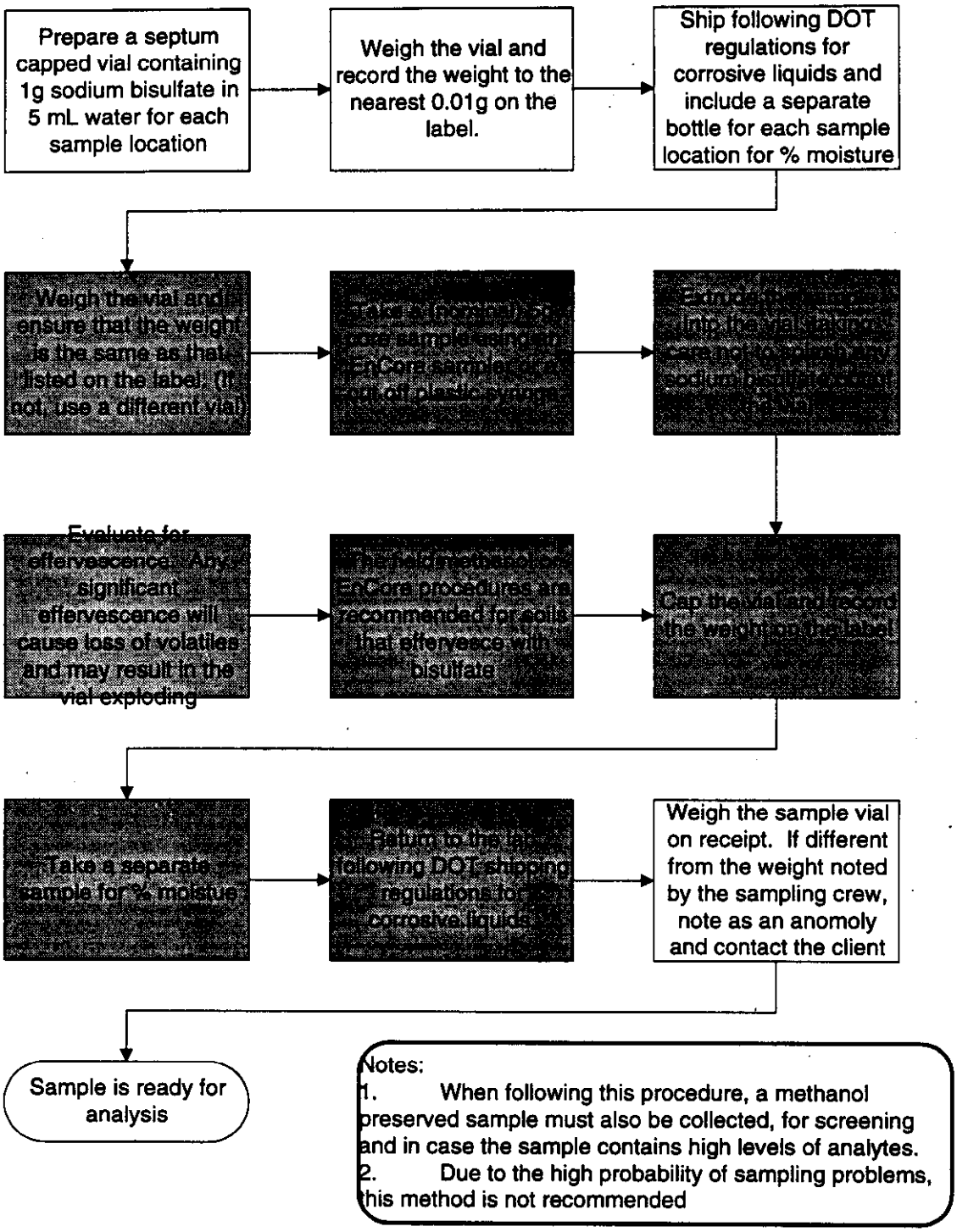
EnCore procedure when low level is required



Field methanol extraction procedure (field steps in gray)



Field bisulfate preservation procedure (field steps in gray)



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

9. QUALITY CONTROL

9.1. Initial Demonstration of Capability

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

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

9.2. Control Limits

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

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

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

9.3. Surrogates

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

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

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

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

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

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

9.4. Method Blanks

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

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

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

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

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

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

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

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

9.5. Laboratory Control Samples (LCS)

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

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

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

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

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

9.6. Matrix Spikes

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

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

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

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

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

9.7. Nonconformance and Corrective Action

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

9.8. Quality Assurance Summaries

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

9.9. Quanterra QC Program

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

10. CALIBRATION AND STANDARDIZATION

10.1. Summary

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

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

10.2. Recommended Instrument Conditions

10.2.1. General

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

10.2.2. Gas chromatograph suggested temperature program

10.2.2.1. BFB Analysis

Isothermal:	170°C

10.2.2.2. Sample Analysis

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

10.3. Instrument Tuning

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

10.4. Initial Calibration

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

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

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

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

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

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

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

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

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

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

10.4.7. Weighting of data points

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

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

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

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

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

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

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

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

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

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

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

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

11. PROCEDURE

11.1. Procedural Variations

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

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

11.2. Preliminary Evaluation

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

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

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

11.3. Sample Analysis Procedure

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

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

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

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

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

11.4. Water Samples

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

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

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

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

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

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

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

11.5. Methanol Extract Soils

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

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

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

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

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

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

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

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

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

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

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

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

11.8. Low-Level Solids Analysis using discrete autosamplers

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

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

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

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

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

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

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

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

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

11.9. Initial review and corrective actions

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

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

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

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

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

11.10. Dilutions

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

11.10.1. *Guidance for Dilutions Due to Matrix*

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

11.10.2. Reporting Dilutions

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

12. DATA ANALYSIS AND CALCULATIONS

12.1. Qualitative identification

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

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

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

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

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

12.2. Tentatively Identified Compounds (TICs)

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

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

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

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

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

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

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

12.3. Calculations.

12.3.1. Response factor (RF):

Equation 1

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

Where:

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

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

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

C_x = Concentration of the compound being measured, ng

12.3.2. Standard deviation (SD):

Equation 2

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

X_i = Value of X at i through N

N = Number of points

X = Average value of X_i

12.3.3. Percent relative standard deviation (%RSD):

Equation 3

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

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

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

Equation 4

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

Where

$C_{expected}$ = Known concentration in standard

C_{found} = Measured concentration using selected quantitation method

12.3.5. Target compound and surrogate concentrations:

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

12.3.5.1. Calculation of concentration using Average Response Factors

Equation 5

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

12.3.5.2. Calculation of concentration using Linear fit

Equation 6

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

12.3.5.3. Calculation of concentration using Quadratic fit

Equation 7

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

x is defined in equations 8, 9 and 10

A is a constant defined by the intercept

B is the slope of the curve

C is the curvature

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

Equation 8

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

Where:

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

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

I_s = Amount of internal standard added in ng

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

V_o = Volume of water purged, mL

12.3.5.5. Calculation of x for Medium level soils:

Equation 9

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

Where:

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

V_t = Volume of total extract, mL (Typically 25 mL)

V_a = Volume of extract added for purging, µL

W_s = Weight of sample extracted, g

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

12.3.5.6. Calculation of x for Low level soils:

Equation 10

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

Where:

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

D is as for medium level soils

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

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

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

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

RF = 1

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

12.3.6. MS/MSD Recovery

Equation 11

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

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

12.3.7. Relative % Difference calculation for the MS/MSD

Equation 12

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

Where:

RPD = Relative percent difference

MSR = Matrix spike result

MSDR = Matrix spike duplicate result

13. METHOD PERFORMANCE

13.1. Method Detection Limit

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

13.2. Initial Demonstration

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

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

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

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

13.3. Training Qualification

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

14. POLLUTION PREVENTION

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

15. WASTE MANAGEMENT

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

16. REFERENCES

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

17. MISCELLANEOUS

17.1. Modifications from the reference method

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

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

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

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

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

17.2. Modifications from previous revision

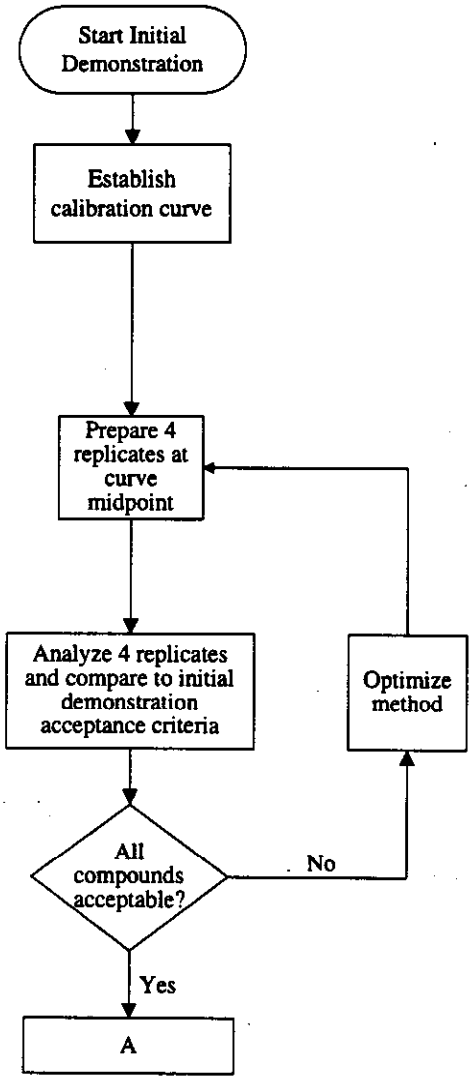
This SOP has been substantially revised to reflect the changes included in Update III to SW-846. Directions for method 524.2 and method 624 have also been added.

17.3. Facility specific SOPs

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

17.4. Flow diagrams

17.4.1. Initial Demonstration and MDL



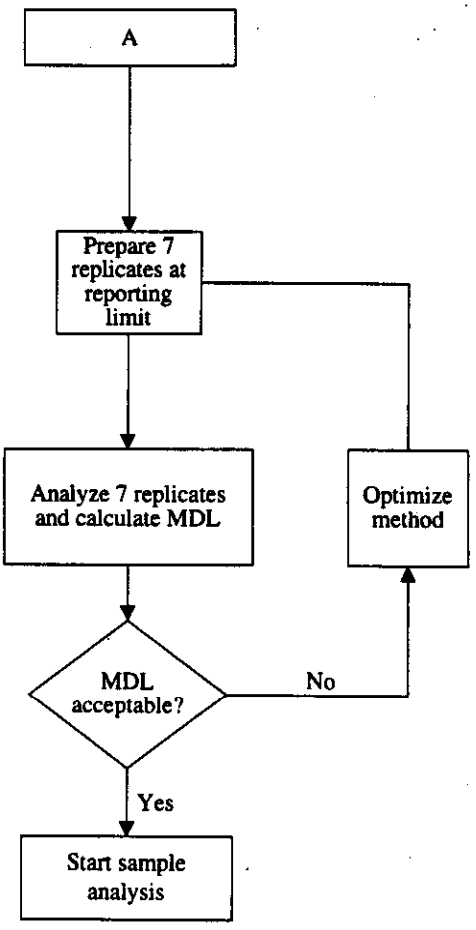


Table 1

Quanterra Primary Standard and Reporting Limits

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

Table 1

Quanterra Primary Standard and Reporting Limits

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

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

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

Table 2

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

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

Table 2

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

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

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

Table 3

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

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

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

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

Table 4

Quanterra Appendix IX Standard Calibration Levels, µg/L

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

Table 5

Reportable Analytes for Quanterra Standard Tests, Primary Standard

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

Table 5

Reportable Analytes for Quanterra Standard Tests, Primary Standard

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

Table 6

Reportable Analytes for Quanterra Standard Tests, Appendix IX standard

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

Table 7

Internal Standards

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

Notes:

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

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

Table 8

Surrogate Standards

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

Notes:

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

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

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

Table 9

Matrix Spike / LCS Compounds

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

Notes:

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

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

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

Table 10

BFB Key Ion Abundance Criteria

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

Table 11

SPCC Compounds and Minimum Response Factors

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

Table 12

CCC compounds

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

Table 13

Characteristic ions

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

Table 13

Characteristic ions

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

Table 13

Characteristic ions

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

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

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

18. SUMMARY

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

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

19. MODIFICATIONS REQUIRED FOR DRINKING WATER ANALYSIS BY METHOD 524.2

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

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

19.3. Sample concentrations are calculated using initial calibration curve.

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

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

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

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

19.8. Initial calibration curve requirements:

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

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

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

19.9. Continuing calibration verification (CCV) requirements:

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

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

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

19.10. Method clarifications, modifications and additions

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

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

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

Table A-1

Quanterra 8260 Drinking Water List Standard and Reporting Limits

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

Table A-1

Quanterra 8260 Drinking Water List Standard and Reporting Limits

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

Table A-1

Quanterra 8260 Drinking Water List Standard and Reporting Limits

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

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

Table A-2
Internal Standards, Method 8260A Drinking water list

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

Notes:

1) Fluorobenzene only is used for method 524.2

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

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

Table A-3
Surrogate Standards, Drinking water list

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

[1] 8260B surrogate

[2] 524.2 surrogate

Notes:

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

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

Table A-4

Quanterra Drinking water list Standard: Calibration Levels

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

Table A-4

Quanterra Drinking water list Standard: Calibration Levels

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

Table A-4

Quanterra Drinking water list Standard: Calibration Levels

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

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

20. REQUIREMENTS FOR EPA 624

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

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

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

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

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

20.6. Initial demonstration of Proficiency

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

20.7. Initial calibration curve requirements:

20.7.1. Target compounds must have RSD ≤ 35%.

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

20.8. Continuing calibration verification requirements:

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

20.9. Matrix Spike and LCS requirements

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

20.10. Method clarifications, modifications and additions

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

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

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

Table B-1.

Method 624 Analytes and Reporting Limits

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

Table B-2.

Method 624 QC Acceptance Criteria

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

Quanterra
Environmental Services

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Page 1 of 63

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

SUPERSEDES: Revision 4.0; August 3, 1995

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1. SCOPE AND APPLICATION

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

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

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

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

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

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

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

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

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

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

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

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

Compound	CAS No
Nitroguanidine	556-88-7

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

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

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

2. SUMMARY OF METHOD

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

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

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

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

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

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

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

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

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

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

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

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

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

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

3. DEFINITIONS

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

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

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

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

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

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

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

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

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

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

4. INTERFERENCES

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

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

5. SAFETY

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

6. EQUIPMENT AND SUPPLIES

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

6.2 Centrifuge.

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

6.4 Liquid chromatograph

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

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

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

6.6 Mortar and pestle, ceramic.

6.7 Recirculator cooler.

6.8 Spatula, stainless steel, or equivalent.

6.9 Stirring bars, magnetic.

6.10 Stirring plate, magnetic.

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

6.12 Temperature controlled ultrasonic water bath.

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

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

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

7. REAGENTS AND SOLVENTS

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

7.1 Acetic acid, Glacial, Reagent grade.

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

7.2 Acetone, pesticide quality.

7.3 Acetonitrile, HPLC grade or equivalent.

7.4 Ammonium acetate, HPLC grade or equivalent.

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

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

7.6 Hexane, pesticide quality.

7.7 Hydrochloric acid, concentrated, reagent grade.

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

7.8 Methanol, HPLC grade or equivalent.

7.9 Methylene chloride, pesticide quality.

7.10 n-Propanol, HPLC grade or equivalent.

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

7.12 Sodium chloride, reagent grade.

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

7.13 Tetrahydrofuran (THF), HPLC grade or equivalent.

7.14 Water, distilled.

7.15 Water, HPLC grade or equivalent.

8. **STANDARDS**

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

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

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

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

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

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

8.3 Surrogate for HPLC/UV.

8.3.1 Several chemicals listed below can be used as surrogate.

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

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

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

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

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

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

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

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

8.5 Analytical Curve for 8330 HPLC/UV.

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

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

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

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

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

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

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

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

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

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

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

8.8 Internal Standards for LC/TSP/MS;

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

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

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

8.9 Analytical Curve for LC/TSP/MS.

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

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

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

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

8.10 Reference Standards

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

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

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

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

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

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

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

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

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

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

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

10. QUALITY CONTROL

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

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

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

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

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

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

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

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

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

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

11. CALIBRATION AND STANDARDIZATION

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

12. PROCEDURE

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

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

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

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

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

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

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

For control, use distilled water or HPLC grade water.

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

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

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

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

12.1.4 Add the spiking solution if applicable.

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

12.3.7 Samples ready for extraction by the Zymark Autotrace SPE.

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

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

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

Step 1: Process 6 samples using the following procedure:

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

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

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

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

Step 6: Load 80 mL of sample onto column

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

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Step 8: Dry Column with gas for 0.5 minutes

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

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

Step 11: END

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

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

NEVER USE ACETONE ON THE ZYMARK AUTOTRACE SPE.

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

12.5.7 Samples ready for extraction by the Zymark Autotrace SPE.

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

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

Step 1: Process 6 samples using the following procedure:

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

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

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

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

Step 6: Load 800 mL of sample onto column

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

Step 8: Dry Column with gas for 0.5 minutes

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

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

Step 11: END

FLOW RATES

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

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

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

NEVER USE ACETONE ON THE ZYMARK AUTOTRACE SPE.

12.5.10 Zymark Autotrace process time for six samples is approximately 124 minutes.

12.5.11 Before starting for the day, solvent lines should be purged by loading the solvent purging program. Check the solvents and replace the solvents if necessary.

12.5.12 The sample lines should have been in a plastic bag, indicating the lines have been cleaned with methanol followed by distilled water from the Cleaning Sample Lines program.

12.5.13 Set the SPE columns in place and depress the plunger. Once in place the green light should be on.

12.5.14 Set the receiving vessels (test tubes) in place. Be sure the first sample is on your left and the sixth sample on your right.

12.5.15 Check the solvents - replace if neccessary, and set the samples in its place.

12.5.16 Check the waste containers (Aqueous and Organic) and replace if necessary. Do NOT allow waste containers to overflow.

12.5.17 Load the program '8330-A' in the disk drive of the Zymark Autotrace, and press Load.

12.5.18 When the disk has been loaded, the Zymark Autotrace is ready to start. Follow instructions displayed on the screen "Press CONT to start" if ready to start.

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

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

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

12.5.22 Adjust the final volume to 5.0 mL with acetonitrile, and add 5 uL of concentrated acetic acid to stablize TETRYL. Mix the contents well. The extract concentration is 800mL/5mL.

12.5.23 Filter the acetonitrile extract through an Acrodisc LC25 filter assembly using a 10-cc disposable syringe into a new test tube. DO NOT READJUST THE VOLUME.

12.5.24 The acetonitrile extracts are stored in the freezer. A dilution of 1/4 is required for analysis to obtain a final concentration of 800mL/20mL.

12.5.24.1 For HPLC/UV, dilute 1.0 mL of the extract with 3.0 mL of distilled water in a 4-mL vial when ready for analysis.

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

12.6 **Soil/Sediment Samples.**

NOTE: Do NOT grind samples containing high concentrations of explosives. Lumps of material that have a chemical appearance should be suspect and not ground. Explosives are generally a very finely ground grayish-white material.

12.6.1 Subsample approximately 20 to 50 grams of soil into a 16-oz wide mouth jar for drying.

12.6.2 If the samples do not appear homogeneous and not suspicious, dry the samples in a cool, ventilated area and away from direct light. DO NOT HEAT the samples.

12.6.2.1 If samples do appear suspicious, stop work and consult with the project manager and client regarding to possible high levels of explosives.

12.6.3 After the samples are dried, grind thoroughly in mortar with pestle (both items previously rinsed with acetonitrile and dried).

12.6.4 Sift the sample through a sieve size 30 mesh. If samples are not to be extracted on the same day, store the sifted soil in the freezer and protect from light.

12.6.5 Label the sample tape with "8330-S" for 8330 by HPLC/UV, "NGU-S" for Nitroguanidine, "SPEX-S" for specialty explosives (Nitroglycerine, PETN, and Picric Acid), and 8330/SPEX-S for 8330 mix and specialty explosives by LC/TSP/MS.

12.6.5.1 Label the sample tape with 'PA-S' for picric acid as this is a separate extraction.

12.6.6 When the extractions are to be performed, turn on the recirculator cooler to cool the sonication bath to operating temperature (5 to 25 ^{o}C). The sonicator bath generates heat when the sonication bath is on.

12.6.7 Weigh 2.0 grams of homogeneous soil into a 40-mL vial.

12.6.7.1 For control, use Ottawa sand. If a larger sample size is desired, then the ratio of sample to solvent must be consistent to 1 gram/5 mL.

12.6.7.2 For picric acid, weigh out a separate set.

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

12.6.8.1 No surrogate is used for the nitroguanidine analysis.

12.6.8.2 For 8330 by HPLC/UV, add 80 uL of the 50 ug/mL surrogate solution to each 2.0 gram soil sample to yield 2.0 ppm.

12.6.8.3 For 8330/SPEX and SPEX by LC/TSP/MS, add 200 uL of the 50 ug/mL d_5-Nitrobenzene standard to each 2.0 gram soil sample to yield 5.0 ppm.

12.6.8.4 For picric acid, add 50 uL of the 50 ug/mL d_5-Nitrobenzene standard to each 2.0 gram soil sample to yield 1.25 ppm.

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

12.6.9.1 For nitroguanidine, fortify at 1.0 ppm with Nitroguanidine by adding 40 uL of the 50 ug/mL standard to the 2 gram sample.

12.6.9.2 For 8330-HPLC/UV, fortify at 1.0 ppm with 8330 mix by adding 40 uL of the 50 ug/mL standard to the 2 gram sample.

12.6.9.3 For 8330 by LC/TSP/MS, fortify at 2.0 ppm with 8330 mix by adding 80 uL of the 50 ug/mL standard to the 2 gram sample.

12.6.9.4 For SPEX-LC/TSP/MS, fortify at 5/5/2.5 ppm of NGl/PA/PETN with SPEX Mix by adding 0.50 mL of the 20/20/10 mix to the 2 gram samples.

12.6.9.5 For PA in soils using the method for picric acid, fortify at 1.25 ppm with SPEX Mix by adding 125 uL to the 2 gram samples.

12.6.10 Adjust the final volume to 10.0 mL with 0.1% (v/v) acetic acid in acetonitrile (except for 'PA-S' see below). Cap, and shake briefly to mix the contents well.

12.6.10.1 To the separate set of PA-S (picric acid in soil) adjust the final volume to 10.0 mL with 0.1% hydrochloric acid in 10% acetonitrile in water, cap, and shake briefly to mix the contents well.

12.6.11 Place the samples in the ultrasonic bath, and sonicate for 18 hours, keeping the bath at 35 °C or cooler.

12.6.11.1 Record the initial time, date, and temperature of the water bath on the benchsheet. Cover the sonication bath with aluminum foil or a cover to protect samples from light.

12.6.11.2 After sonication, record the final time, date, and temperature of the water bath.

12.6.12 Allow the samples to settle for 30 minutes or centrifuge the samples at approximately 1000 rpm for about 5 minutes.

12.6.13 Decant the extracts (minimally 4 mLs) into 8-mL test tubes. The extracts may be worked up immediately or stored in the freezer until ready for further prep.

12.6.14 Filter the required amount needed for analysis by transferring into a 10-cc disposable syringes fitted with 0.45 um PTFE 25-mm filters, and filter them with positive pressure using the plunger into a clean 8-mL screwcap test tubes.

12.6.15 For 8330-HPLC/UV, dilute 1.0 mL of the filtered extract in an 8-mL screwcap test tube with 3.0 mL of the 1.3 g/L calcium chloride solution. Shake briefly to mix the contents.

12.6.15.1 Filter the extracts by transferring into a 10-cc disposable syringes fitted with 0.45 um PTFE 25-mm filters, and filter them with positive pressure using the plunger into a clean 4-mL screwcap vials.

12.6.15.2 Store the 8330-HPLC/UV extracts in the freezer and protect from light until analysis. The final sample concentration is 0.20 g/4.0 mL.

12.6.16 For nitroguanidine (NGu-S), dilute 2.0 mL of the filtered extract into an 8-mL test tube with 0.5 mL of distilled water to obtain a 80:20 acetonitrile:water composition. Mix the contents well.

12.6.16.1 Filter the extracts by transferring into 10-cc disposable syringes fitted with 0.45 um PTFE 25-mm filters, and filter them with positive pressure using the plunger into clean 4-mL screwcap vials.

12.6.16.2 Store the NGu-S extracts in the freezer and protect from light until analysis. The final concentration is 0.4g/2.5 mL.

12.6.17 For SPEX-LC/TSP/MS and 8330/SPEX-LC/TSP/MS, dilute 1.0 mL of the filtered extracts in an 8-mL test tube with 3.0 mL of distilled water (do NOT add calcium chloride solution!).

12.6.17.1 Add 20 uL of the internal standard mix d_3-2,6-DNT and d_4-1,3-DNB, and mix the contents well.

12.6.17.2 Filter the extracts with 10-cc disposable syringes fitted with 0.45 um PTFE 25-mm filters into clean 4-mL screwcap vials. The final extract concentration is 0.2g/4.0mL.

12.6.17.3 Store the extracts in the freezer and protect from light until ready for analysis by LC/TSP/MS.

12.6.18 For PA-S, the extracts may be filtered or decanted into an 8-mL test tube, and the extracts may be worked up immediately or stored in the freezer until ready for further prep.

Since the extracting solvent is 0.1% hydrochloric acid in 10% acetonitrile in water, the organic content of the extract is less than 20% in water, therefore no dilution with water is required. The final extract concentration is 2.0g/10mL.

12.6.18.1 If the extracts has not been filtered, then filter the required amount needed for analysis with 10-cc disposable syringes fitted with 0.45 um PTFE 25-mm filters into clean 8-mL screwcap test tubes.

12.6.19 Transfer 2.0 mL of the filtered extracts into 4-mL vials, and add 10 uL of the internal standard mix d_3-2,6-DNT and d_4-1,3-DNB to yield 50 ng/mL in the final extract. Mix the contents well, and the final extract concentration is 0.4g/2.0mL.

12.6.20 Store the extracts in the freezer and protect from light until analysis by LC/TSP/MS.

12.7 **INSTRUMENTATION for nitroguanidine by HPLC/UV**

The conditions listed below are the recommended analytical conditions for nitroguanidine by HPLC/UV. If changes to the HPLC conditions are necessary, they will be noted on the chromatogram.

12.7.1 Primary Column: Phenomenex Zorbax C_8, 250 x 4.6 mm, 5 um, or equivalent.

12.7.2 Column Temperature: 30 °C

12.7.3 Mobile Phase (Reservoir)
A = Water
B = Acetonitrile
C = Methanol

Gradient Program:

Time	A	B	C
0.0	100	0	0
8.0	100	0	0
9.0	0	50	50
15.0	0	50	50

12.7.4 Flow rate: 1.0 mL/min.

12.7.5 Injection volume: 25 uL

12.7.6 UV Detection at 250 nm.

12.7.7 Approximate retention times on C_8 column:

Nitroguanidine 4.3 minutes

12.7.8 Preventive Maintenance:

Change in-line-frit as needed to retain proper pressure, peak shape and response.

If nitroguanidine is not retained or the retention time is close to the void volume on the C8 or C18 column, replace with another column.

12.8 INSTRUMENTATION for 8330 by HPLC/UV

The conditions listed below are the recommended analytical conditions for 8330 by HPLC/UV. If changes to the HPLC conditions are necessary, they will be noted on the chromatogram.

12.8.1 Primary Column: Phenomenex Hypersil C_8, 150 x 4.6 mm, 5 um, or equivalent.

12.8.2 Column Temperature: 20 °C (Column kept cooled to a constant tempeature with a recirculated cooler).

12.8.3 Mobile Phase (Reservoir)

A = Water
B = 98:2 (v/v) methanol/tetrahydrofuran
C = 98:2 (v/v) methanol/acetonitrile

Gradient program:

Time	A	B	C
0.0	75	25	0
2.0	75	35	0
22.0	35	0	65
24.0	0	0	100

12.8.4 Flow rate: 1.5 mL/min.

12.8.5 Injection volume: 200 uL on UV-9050
250 uL on UV-200

12.8.6 UV Detection at 250 nm.

12.8.7 Elution order and approximate retention times on C_8 column:

HMX	3.90 minutes	
TNB	6.59	
RDX	7.16	
2,4-DNFB	8.58	(Surrogate)
DNB	9.43	
NB	10.95	
TNT	12.03	
TETRYL	13.09	
2,4-DNT	14.22	
2,6-DNT	14.92	
2-AM-4,6-DNT	15.47	
4-AM-2,6-DNT	15.75	
2-NT	16.24	
4-NT	16.52	
3-NT	17.09	

12.8.8 Preventive Maintenance:

Change in-line-frit as needed to retain proper pressure, peak shape and response. The use of guard column causes poor resolution between RDX and TNB; therefore, guard columns are not used.

Co-elution of RDX and TNB and of 2-AM and 4-AM on the primary column depends on the type and condition of the column and the solvent composition of THF in methanol.

Loss of resolution between TNB and RDX may be caused by column deterioration.

If the solvent composition of THF in methanol is changed, RDX and TNB elution order may be reversed or co-elution may occur.

12.9 **INSTRUMENTATION for Confirmation of 8330 by HPLC/UV**

The conditions listed below are the recommended analytical conditions for confirmation of 8330 by HPLC/UV. If changes to the HPLC conditions are necessary, they will be noted on the chromatogram.

12.9.1 Confirmation Column: Zorbax Cyano CN, 250 x 4.6 mm, 5 um, or equivalent.

12.9.2 Column Temperature: 20 °C (Column kept cooled to a constant temperature with a recirculated cooler)

12.9.3 Mobile Phase (Reservoir) and Gradient Program

OPTION 1

A = Water
B = 98:2 (v/v) methanol/THF
C = 98:2 (v/v) methanol/MeCN

Time	A	B	C
0.0	60	0	40
17.0	40	60	0
19.0	0	100	0
35.0	0	100	0

OPTION 2

A = water
B = acetonitrile
C = methanol

Time	A	B	C
0.0	80	8	12
20.0	60	28	12
35.0	0	100	0

12.9.4 Flow rate: 1.0 mL/min.

12.9.5 Injection volume: 500 uL on UV-9050
250 uL on UV-200

12.9.6 UV Detection at 250 nm.

12.9.7 Elution order and approximate retention times on CN column:

OPTION 1			OPTION 2		
NB	12.20		NB	18.52	
DNB	13.16		DNB	20.07	
TNB	13.63		2,4-DNFB	20.94	Surrogate
2-NT & 4-NT	15.19		TNB	21.57	
3-NT	15.59		2-NT & 4-NT	23.82	
1C3NB	15.62	Surrogate	3-NT	24.25	
2,6-DNT	16.23		RDX	24.71	
2,4-DNT	16.68		2,4-DNT	25.78	
TNT	17.96		2,6-DNT	26.37	
4-AM-2,6-DNT	18.19		2-AM-4,6-DNT	27.29	
2-AM-4,6-DNT	18.79		4-AM-2,6-DNT	27.50	
RDX	20.42		TNT	28.73	
TETRYL	23.14		HMX	29.48	
HMX	24.52		TETRYL	31.00	

3-NT and 1C3NB co-elute on option 1.
1C3NB and RDX co-elute on option 2.
2-NT and 4-NT co-elute on both options.

12.10 **INSTRUMENTATION for 8330 by LC/TSP/MS**

The conditions listed below are the recommended analytical conditions for the analysis of specialty explosives and for the analysis or confirmation of 8330 compounds. If changes to the LC/TSP/MS conditions are necessary, they will be noted on the chromatogram.

12.10.1 Primary Column: Novapak C-8, 3.9 X 150 mm, 5 um, or equivalent.

12.10.2 Temperature: Ambient

12.10.3 Mobile Phase (Reservoir)

A = 0.20 M Ammonium acetate (aqueous) with 0.10% acetic acid
B = N/A
C = n-Propanol

Gradient program:

Time	Flow	A	C	Grad
init	1.00	95	5	--
4.0	1.00	85	15	6
7.0	1.00	70	30	6
7.1	0.80	70	30	6
14.0	0.80	50	50	6
15.0	0.80	95	5	11
19.0	1.00	95	5	11

(flow rate in ml/min)

The post column pump flow rate of solution A is set at 0.40 mL/min throughout the run.

12.10.4 Injection volume: 150 uL

12.10.5 Thermospray (TSP) conditions

The following thermospray conditions must be optimized whenever a new thermospray vaporizer is installed in order to provide good performance.

Vaporizer Temperature:	80 to 120 °C
Source Temperature:	280 to 350 °C
Repeller Voltage:	0 to 50 Volts
Discharge Voltage:	1200 Volts
Filament Current:	0 to 400 uA
Electron Energy:	0 to 500 eV

(Note: Negative ion analysis will usually require discharge voltage of 800 to 1000 V, filament current of 400 uA, and electron energy of 500 eV).

12.10.6 Mass Spectrometer (MS) scanning conditions

Conditions below describe analysis in negative-ion selective-ion-monitoring (SIM) mode. Some compounds (e.g. HMX and RDX) also respond in positive-ion mode. These compounds may be analyzed using positive-ion selective-reaction-monitoring (SRM) for confirmation purposes or when interfering compounds are present in negative-ion SIM analysis. Ions used in positive-ion SRM analysis will differ from those listed below and must be established by analysis of standard reference materials.

Compound (in approximate order of elution)	Ion	Retention Time (approximated)
HMX	355	3:39
Trinitrobenzene	213	5:53
RDX	281	6:12
1,3-Dinitrobenzene	168	8:54
d_4-1,3-Dinitrobenzene	172	8:48 (Int. Std)
Picric Acid	228	8:50
Trinitrotoluene	227	9:52
TETRYL	241	10:12
d_5-Nitrobenzene	128	10:49 (Surrogate)
Nitrobenzene	123	10:57
Nitroglycerine	241	11:06
2,4-Dinitrotoluene	182	11:48
2-Amino-4,6-dinitrotoluene	197	11:56
d_3-2,6-Dinitrotoluene	185	12:12 (Int. Std)
4-Amino-2,6-dinitrotoluene	197	12:12
2,6-Dinitrotoluene	182	12:14
2- & 4-Nitrotoluene	137	13:08
PETN	330	13:14
3-Nitrotoluene	137	13:23

2-NT and 4-NT co-elute by this method.

12.10.7 Preventive Maintenance:

Change in-line-frit as needed to retain proper pressure, peak shape and response.

13. DATA ANALYSIS AND CALCULATIONS

13.1 Results are reported in ug/g or mg/Kg (ppm) for soil, unless otherwise instructed.

13.2 Results are reported as dry weight since soil samples are air dried, ground with motar and pestle, and sieved, unless specified by the client.

13.3 The concentration of each analyte and surrogate in a sample is calculated by using the appropriate formula:

Without using internal standard correction for HPLC/UV;

$$\text{Amount Found (ppb)} = \frac{CF_a \cdot A_x \cdot V_x}{W_s}$$

where CF_a = Average Calibration Factor in ng/mL.
A_x = Sample Total Area Counts.
V_x = Sample Extract Final volume in milliliters.
W_s = Initial Sample weight in grams.

Using internal standard correction for LC/TSP/MS;

$$\text{Amount Found (ppb)} = \frac{CF_a \cdot A_x \cdot V_x}{W_s} \times \frac{C_{is}}{A_{is}}$$

where C_{is} = Internal Standard Concentration in ng/mL.
A_{is} = Internal Standard Total Area Counts.

The results of soils in ppb (ng/g) are converted to ppm (ug/g) by dividing the result in ng/g by 1000;

$$\text{ug/g} = \frac{X \text{ ng/g}}{1000 \text{ ng/ug}}$$

14. METHOD PERFORMANCE

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

15. POLLUTION PREVENTION

NONE

16. WASTE MANAGEMENT

16.1 Flammable Waste

16.1.1 Acetone, acetonitrile, hexane, and methanol waste generated from solvent rinsing of glassware is collected as "Flammable Waste" in 1-gallon plastic bottles.

16.1.2 The unused portion of the acetonitrile extracts from soil extraction may be decanted from the soils and discarded as flammable waste. For the soil portion see "solid waste" below.

16.1.3 When the 1-gallon plastic bottle is full, the waste is transferred to the 55-gallon drum labelled "Flammable Waste" in the H-3 room.

16.1.4 Methylene chloride should be collected separately, see "DCM Waste" below. Any organic waste that is less than 50% methylene chloride is flammable waste.

16.2 Methylene Chloride (DCM) Waste

16.2.1 Methylene chloride, or dichloromethane (DCM), generated from solvent rinsing of glassware is collected separately from the Flammable Waste into "DCM Waste" in 1-gallon plastic bottles.

16.2.2 Any organic waste that is in excess of 50% methylene chloride is considered DCM waste.

16.2.3 When the 1-gallon plastic bottle is full, the waste is transferred to the 55-gallon drum labelled "DCM Waste" in the H-3 room.

16.3 Liquid-liquid extraction (LLE) Waste

16.3.1 Aqueous samples from high level and low level extraction (liquid-liquid extraction) after completion have been saturated with organic solvent (such as acetonitrile) is collected into 5-gallon carboy containers, or if the pH of the water samples were neutral (pH between 6 to 9) they can be collected directly into 55-gallon drum labelled "LLE Waste" in the H-3 room.

16.3.2 Aqueous samples collected in 5-gallon carboy containers are transferred to 55-gallon drum labelled "LLE Waste" in the H-3 room after the pH is check and ajusted if necessary to between 6 to 9.

16.4 High Performance Liquid Chromatography (HPLC) Effluent

16.4.1 Waste generated from the HPLC is collected into 1-L plastic bottles labelled "HPLC Waste", it consists primarily of methanol, acetonitrile, water, and ammonium acetate.

16.4.2 When the 1-gallon plastic bottle is full, the waste is transferred to the 55-gallon drum labelled "HPLC/ETHER Waste" in the H-3 room.

16.5 Solid Waste

16.5.1 Residual soil are allowed to air-dry in the hood or in well ventilated area. After the soils are dried, they are discarded into the "Dry Hazard" trash.

16.5.2 Dry Hazard trash consists of glass, gloves, papers, and plastics that were in contact with samples. When the trash is full, it is double bagged, secured, and disposed in the 55-gallon drum labelled "Dry Hazards" in the waste room where they are compacted in the 55-gallon drums.

16.5.3 Empty glass solvent bottles are disposed of in the waste room after they have been air dried. They are placed in the 55-gallon drum where they are compacted and crushed.

17. REFERENCES

17.1 SW-846 Draft Method 8330, "Nitroaromatics and Nitramines by High Pressure Liquid Chromatography (HPLC)", Revision 0, November 1992, U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington, D.C.

17.2 Thomas F. Jenkins, et. al., "Comparison of Cartridge and Membrane Solid-Phase Extraction with Salting-Out Solvent Extraction for Preconcentration of Nitroaromatic and Nitramine Explosives from Water", Special Report 92-95, December 1992, US Army Corps of Engineers, Cold Regions Research & Engineering Laboratory, Hanover, New Hampshire.

17.3 "Quanterra Quality Control Program", QA-003, Quanterra Environmental Services, Revision No. 0, April 4, 1995.

17.4 "Laboratory Documentation Practices", LP-CAL-0001, Quanterra Environmental Services, Revision 3.0, December 5, 1991.

17.5 "Calibration and Calibration Check of Balances", SAC-QA-0041, Quanterra Environmental Services, Revision 1.1, December 30, 1994.

18. MODIFICATIONS TO SOP

18.1 This method deviates from EPA method 8330, November 1992 revision on the following:

Acetonitrile is our preferred solvent instead of methanol in the preparation of standards.

The final solvent for extracts for HPLC/UV analysis should be no more than 25% acetonitrile in water. Higher percentages of acetonitrile causes poor chromatography.

Sodium chloride is used in the salting out method of aqueous samples instead of sodium sulfate.

The primary column used in this method is C-8 reverse phase instead of C-18, and the column length is 15 cm instead of 25 cm.

A gradient program is run using Water/Methanol with THF and Acetonitrile as modifiers instead of isocratic at 50/50 Methanol/Water.

The initial calibration is performed by injecting five concentration levels singly instead of triplicate injections of five levels randomly.

The amount of calcium chloride solution (5g/L aqueous) added to the soil extracts is 2:8 and is less than the original method of 1:1. Calcium chloride solution is a flocculating agent that aids in the filtration of soil extracts. Higher concentrations may cause clogging of the frits and over-pressurizing the HPLC.

The UV detector is set at 250 nm instead of 254 nm.

The injection volume is 200 uL instead of 100 uL.

18.2 Changes made on the original revision to revision 1.0.

Improved the stability of TETRYL in soil by the addition of acetic acid at 0.1% by volume in acetonitrile as the extraction solvent.

Included the component Nitroguanidine by HPLC/UV.

Included the components Nitroglycerine, PETN, and Picric Acid by LC/TSP/MS.

8330 components can also be analyzed by LC/TSP/MS.

Nitroglycerine, PETN, and Picric acid can be extracted using 0.1% hydrochloric acid in acetonitrile instead of acetic acid in acetonitrile; however, nitroglycerine and PETN can be extracted using acetic acid along with 8330 components.

18.3 Changes made on revision 1.0 to revision 2.0.

Added sections to include extraction of 8330 components at high levels by the low level extraction.

Included the extraction of Nitroguanidine in water and in soil.

Included the extraction of Specialty Explosives (Nitroglycerine, Picric Acid, and PETN) in water and in soil.

Included the LC/TSP/MS conditions for Specialty Explosives.

Analysis may be performed by LC/TSP/MS.

Confirmation on positives measured by HPLC/UV may be performed by LC/TSP/MS.

18.4 Changes made on revision 2.0 to revision 3.0

Component 1-Chloro-3-nitrobenzene is added as a surrogate for 8330 by HPLC/UV analysis.

Component d_5-Nitrobenzene is added as a surrogate for 8330 by LC/TSP/MS and component d_4-1,3-Dinitrobenzene is added as another internal standard with d_3-2,6-Dinitrotoluene for LC/TSP/MS.

Combining method 8330 and specialty explosives as a single methodology by LC/TSP/MS (8330/SPEX).

Included flow diagrams.

18.5 Changes made on revision 3.0 to revision 4.0.

Updated to Quanterra format.

Correction on the naming of TETRYL as Methyl-2,4,6-trinitrophenylnitramine, not Methyl-2,4,6-trinitrophenylamine.

Analytical curve and midpoint standards for 8330 by HPLC/UV are prepared in 25:75 0.1% acetic acid in acetonitrile:HPLC grade water, and is valid for up to 45 days when properly stored in the refrigerator.

Addition of spiking soils for 8330-LCMS with 8330 mix at 2.0 ppm.

Changed final solvent composition of SPEX-A and 8330/SPEX-A to 20% acetonitrile in water (injection of samples greater than 20% acetonitrile in water adversely affects the chromatography).

Correction and clarity on the final prep of 8330/SPEX-LCMS-A.

Soil extracts are decanted into 8-cc test tubes for storage after extraction.

Addition of Method 8330 by SPE as Low Level and High Level in place of salting-out method.

Injection volume for cyano confirmation on UV 9050 to 250 uL and on UV 100 to 370 uL.

Injection volume for analytical column on UV 100 to 250 uL.

Separate extraction method for picric acid with 0.1% hydrochloric acid in 10% acetonitrile in water.

18.6 Changes made on revision 4.0 to revision 5.0.

SOP number changed from SAC-GC-0124 to SAC-LC-0001.

Other references related to quality added to References section.

Tetrahydrofuran added to list of flammable solvents.

Added TNT/RDX Soil Extrtaction Pac to test suspected soils before grinding.

New surrogate added, 2,4-dinitrofluorobenzene (2,4-DNFB) for HPLC/UV.

Changed the concentration range of the new surrogate for the initial standard curve on HPLC/UV.

Changed the standard range up to 1000 ng/mL for 8330 and eliminated level 4 standard on HPLC/UV.

Modify aqueous samples for the stability of TETRYL by adding 5 uL of concentrated acetic acid to the final 5.0 mL acetonitrile extracts.

Changed the concentration range of d5-Nitrobenzene for the initial standard curve on LC/TSP/MS.

Adjusted the spike level of d5-Nitrobenzene on aqueous samples to achieve 200 ng/mL in the final extracts.

Correction on UV-100 as UV-200.

Correct injection volume for cyano confirmation on UV 200 at 250 uL, not 370 uL.

Triplicate injection is no longer required at the begining of the batch but a single injection and it is to be less than or equal to 15% of the expected value from the initial calibration curve.

All continuing calibration standards in the batch are to be less than or equal to 15% of the initial calibration curve and not from the triplicate standard injections.

Soils are dried until no visible appearance of moisture present. Successful grinding and sifting verifies that soils are dried.

Updated instrument conditions for 8330 by HPLC/UV and for 8330 confirmation by HPLC/UV.

Added "LLE Waste" under Waste Management section to handle aqueous waste saturated with organic solvent (acetonitrile used in the high level and low level salting out methods).

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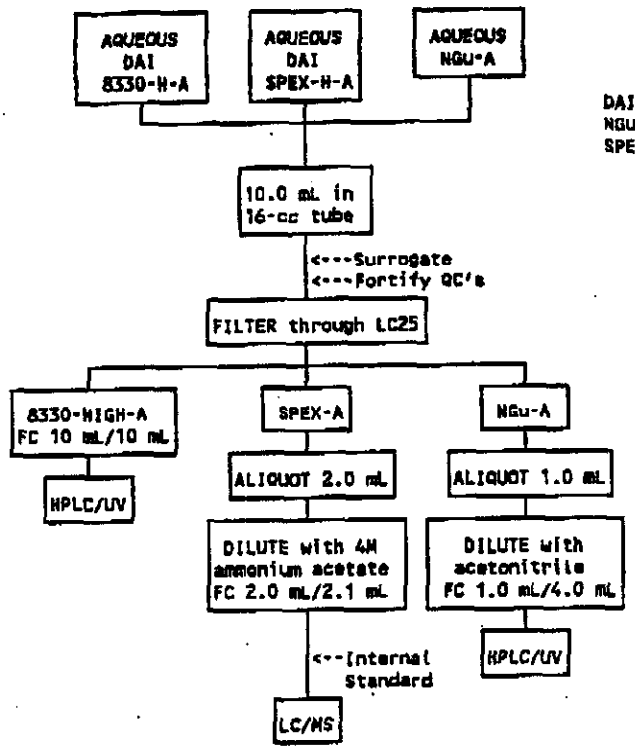
19. FLOW DIAGRAM

19.1 Aqueous by DAI for high levels.



DAI = Direct Aqueous Injection
NGU = Nitroguanidine
SPEX = Specialty Explosives (Nitroglycerine, Picric Acid, and PETN)

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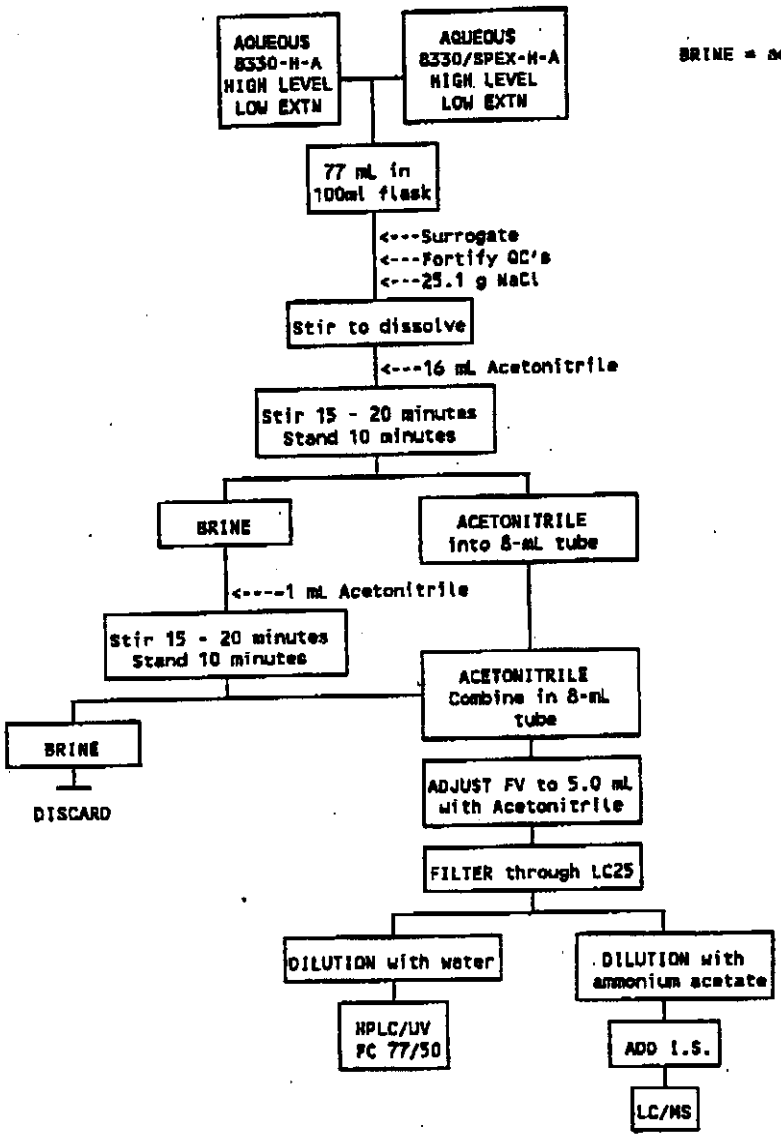
SOP No. SAC-LC-0001
Revision No. 5.0
Date January 3, 1996
Page 59 of 63

19.2 Aqueous by Salting Out for high levels.



C4-125

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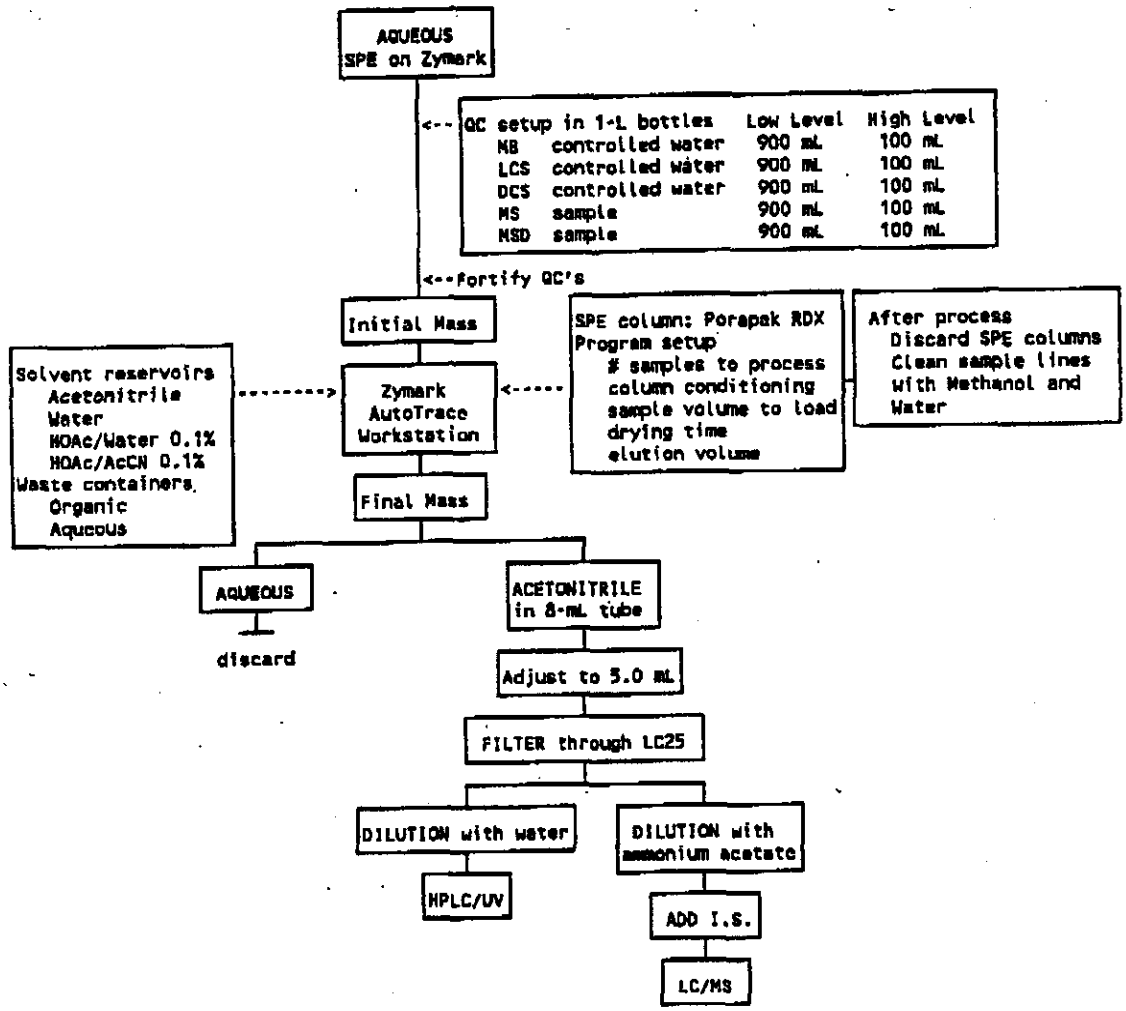
19.3 Aqueous by SPE using Zymark Autotrace Workstation for high levels or low levels.



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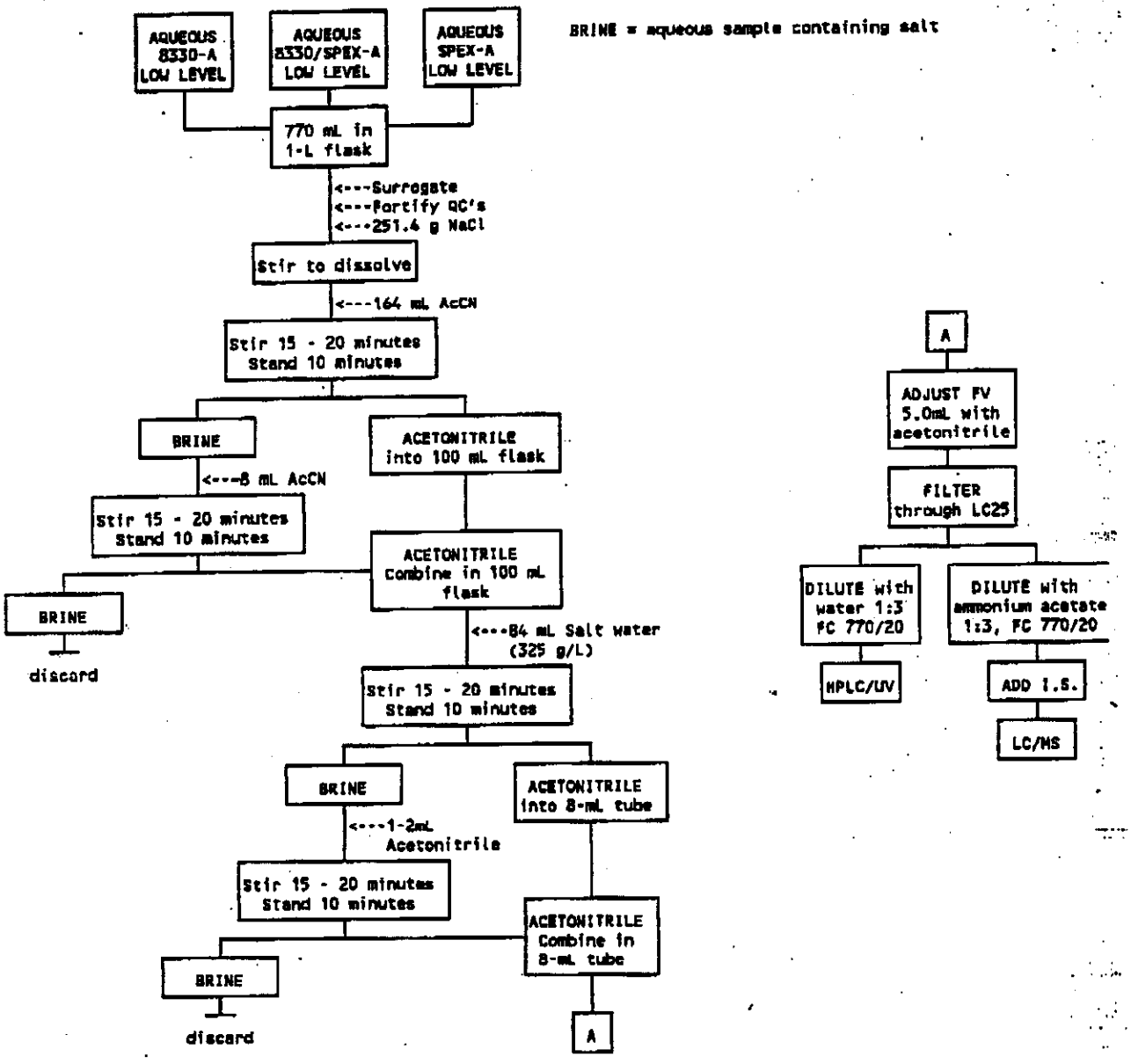
SOP No. SAC-LC-0001
Revision No. 5.0
Date January 3, 1996
Page 61 of 63

19.4 Aqueous by Salting Out for low levels.



C4-127

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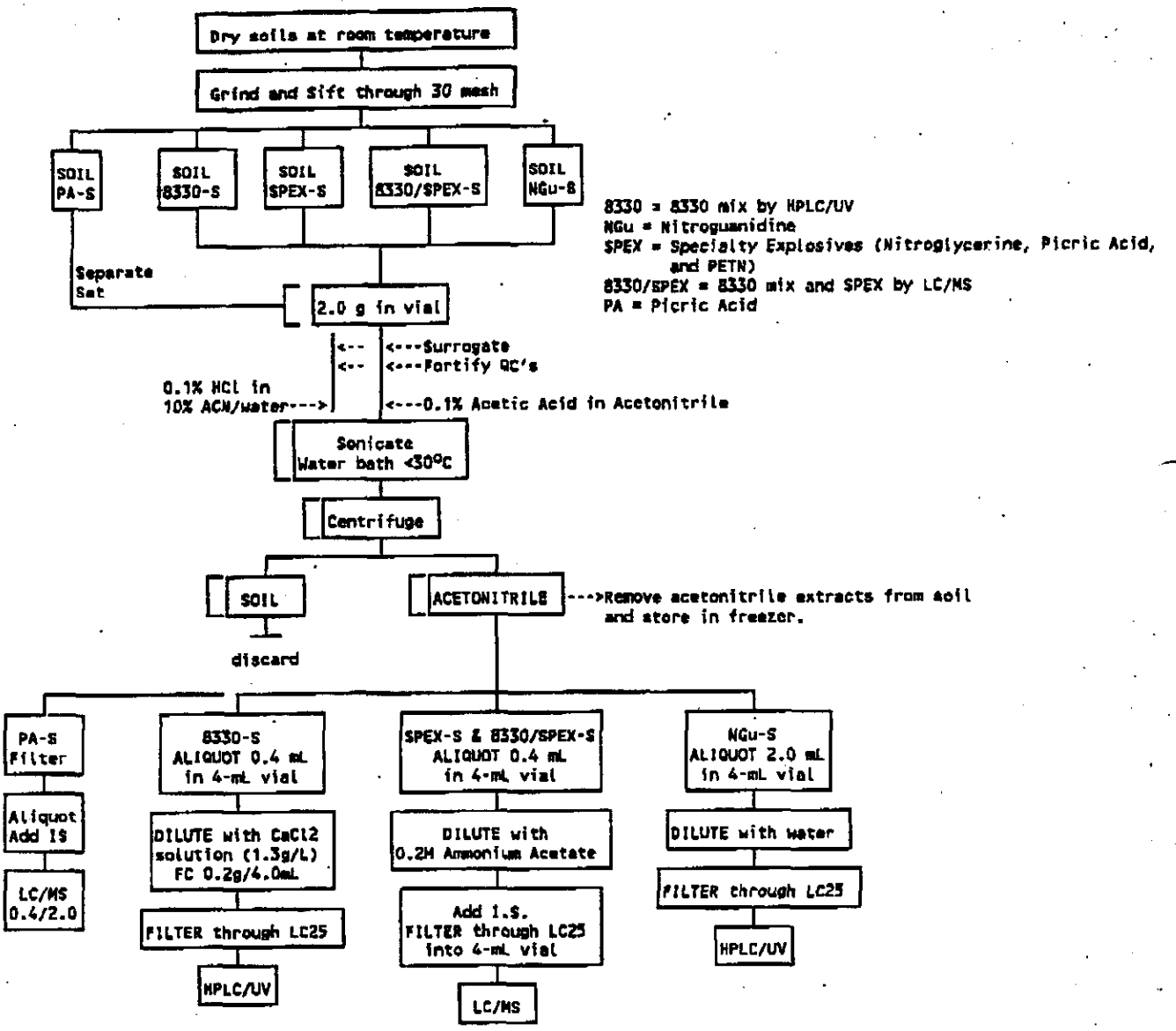
SOP No. SAC-LC-0001
Revision No. 5.0
Date January 3, 1996
Page 62 of 63

19.5 Soil for 8330 and specialty explosives.



C4-128

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Environmental Services

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20. APPENDIX

A copy of form - STAMDARD SOLUTION MADE FROM NEAT.

A copy of form - STANDARD SOLUTION MADE FROM SOLUTION(S).

A copy of form - MASTER SHEET

A copy of form - HPLC INSTRUMENT LOG

Quanterra / W. Sacramento

CHROMATOGRAPHY RUN LOG

Method: 8330_HY File: [MAY_SV] 4LC Inst: hplc #4 [109]

Calib: 4HY0513I 8330 Curve; 2.5 to 1000 ng/mL Hypersil C8 on 5/13/96

HPLC-UV #4 Phenomenex Hypersil C8 SN114198 150x4.6mm 5um, 8330 Analys

#	SAMPLE NAME	MULT DIVS	DATE	TIME	LVL
1	Water Blank	1.00 / 1.000	20-MAY-1996	13:35	
2					
3					
4					
5					
6					
7					
8					
9					
10					
11					
12					
13					
14					
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16					
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34					
35					
36					
37					
38					
39					
40					

COLUMN	A =	B =

Analyst: _____ Date: _____ Page: ____

Q/WS Elog 1.1 Printed: 05/20/96 15:14:59

C4-130

MASTER SHEET Holding time Due: _____ PROJECT Due: _____

PROJECT NO. : _____ Page ___ of ___ INITIATED BY: _____ DATE: _____

METHOD NO. : _____ COMPLETED BY: _____ DATE: _____

SAMPLE MATRIX: _____ REVIEWED BY : _____ DATE: _____

COMPOUND(S) : _____ APPROVED BY : _____ DATE: _____

STORAGE								

Comments: _____

CAL ID/SAMPLE DATA	SAMPLE SIZE	FORTIFICATION - STANDARD DATA PPB COMPOUND(S) ALIQUOT STD. CONC. STD. ND.			

DEQ; BOILER 5-26-94.

C4-131

MAY 05 '98 09:46 FR QUANTERRA 916 372 1059 TO 13606381685 P.63/66

PAGE # 96-_____

QUANTERRA ENVIRONMENTAL SERVICE, WEST SACRAMENTO

ENERGETICS AND SPECIALTY CHEMICALS GROUP (ESC)
STANDARD SOLUTION MADE FROM NEAT

SOLUTION ID#: _____

NAME OF STANDARD: _____

COMPONENT/SOURCE/STOCK#/LOT#/LOCATION/PURITY/EXP.	WEIGHT	FINAL CONC

(Additional components may be listed on back)

DESCRIPTION OF PREPARATION: BALANCE ID# _____

SPECIAL NOTES/COMMENTS:

ACCEPTABLE FOR USE? yes ___ no ___ QC'ed BY/DATE: _____

PREPARED BY/DATE: _____ REVIEWED BY/DATE: _____

STORAGE LOCATION: _____ EXPIRATION DATE: _____

DEG 2-1-96 / QA #359

C4-132

PAGE # 96-_____

QUANTERRA ENVIRONMENTAL SERVICE, WEST SACRAMENTO

ENERGETICS AND SPECIALTY CHEMICALS GROUP (ESC)
STANDARD SOLUTION MADE FROM SOLUTION(S)

SOLUTION ID#: _____

NAME OF MIX: _____

COMPONENT/ID#/LOCATION/CONCENTRATION/SOLVENT/ EXPIRATION	VOLUME USED	FINAL CONC

(Additional components may be listed on back)

DESCRIPTION OF PREPARATION:

_____

_____

_____

SPECIAL NOTES/COMMENTS:

_____

_____

ACCEPTABLE FOR USE? yes ___ no ___ QC'ed BY/DATE: _____

PREPARED BY/DATE: _____ REVIEWED BY/DATE: _____

STORAGE LOCATION: _____ EXPIRATION DATE: _____

DEG 2-1-96 / QA #360

C4-133

QUANTERRA ENVIRONMENTAL SERVICES (West Sacramento)
SOP CHANGE FORM

SOP Number:	SAC-LC-0001
SOP Title:	
SOP Sections Affected by Change:	SECTION 13.3
Reason for Addition or Change:	EQUATION WAS TYPED INCORRECTLY
Change Effective From [Date]:	SEPT. 3, 1997
Change or Addition (Specific Section; Use additional sheets if necessary):	HPLC/UV: AMOUNT FOUND (ppb) = $\frac{A_x \cdot V_x}{CF_c \cdot W_s}$

Submitted by/Date: Dennis E. Gall / 9-3-97
Approved by:*
Technical Reviewer: [signature] Date 9/3/97
EH&S Signature: [signature] Date 9/3/97
QA Signature: [signature] Date 9/3/97
Management Signature: [signature] Date 9-3-97
* Must be same signature authorities of SOP being revised.

C4-134

QUANTERRA INC. (West Sacramento)

STANDARD OPERATING PROCEDURE CHANGE FORM

SOP Number:	SAC-LM-0001, Revision 5.0, 07/08/96.
SOP Title:	Detemination of Nitroaromatics, Nitramines, and Specialty Explosives in Water and Soil/Sediments by HPLC/UV and by LC/TSP/MS
SOP Sections Affected by Change:	8.5, 12.1.4, 12.2.4, 12.4.4, 12.6.9, and 12.8.3.
Reason for Addition or Change:	Adding PETN with 8330 analytes by HPLC/UV.
Change Effective From [Date]:	May 4, 1998
Change or Addition (Specific Section; use additional sheets if necessary.):	8.5 Analytical Curve for 8330 by HPLC/UV. PETN is added to the 8330 Mix between 40 ng/mL to 1,000 ng/mL. 12.1.4 PETN is added to the LCS and MS/SD, or to the DCS, at 250 ppb by adding 25 uL of the 100 ug/mL standard to 10 mL water for DAI method 12.2.4 PETN is added to the LCS and MS/SD, or to the DCS, at 162 ppb by adding 125 uL of the 100 ug/mL standard to 77 mL water for high level salting method. 12.4.4 PETN is added to the LCS and MS/SD, or to the DCS, at 6.5 ppb by adding 50 uL of the 100 ug/mL standard to 770 mL water for low level salting method. 12.6.9 PETN is added to the LCS and MS/SD, or to the DCS at 5.0 ppm by adding 100 uL of the 100 ug/mL standard to 2.0 gram soil, and the sample is prep as per 8330. 12.8.3 The gradient program is modified for the elution of PETN. At 12 minutes, the solvent composition is 60:5:35, and at 15 minutes the solvent composition is 59:4:37, which is held till the end of the run. After the nitrotolunes elutes, the UV detection is switched at approximately 20.8 minutes to 214 nm to detect PETN at approximately 22.0 minutes.

Submitted by/Date:

C4-135

Approved by:*

Technical Reviewer: Karla Mueller *Date:* 5/4/98

EH&S Signature: *Date:*

QA Signature: *Date:*

Management Signature: *Date*

**Must be same signature authorities of SOP being revised.*

d:\qsscca01\secqs\sop\amend.dot

C4-1369

TABLE 1 REPORTING LIMITS FOR QUANTERRA, WEST SACRAMENTO

Parameter	Method W=Water S=Soil	Analyte	Water (ug/L)	Soil (mg/kg)
SW8321	Explosives by HPLC			
		HMX	13.0	0.25
		sym-Trinitrobenzene	7.3	0.25
		RDX	14.0	0.25
		1,3-Dinitrobenzene	4.0	0.25
		Nitrobenzene	6.4	0.25
		2,4,6-Trinitrotoluene	6.9	0.25
		Tetryl	4.0	0.25
		2,4-Dinitrotoluene	5.7	0.25
		2,6-Dinitrotoluene	9.4	0.25
		2-Am-DNT	10.0	0.25
		4-Am-DNT	10.0	0.25
		2-Nitrotoluene	12.0	0.25
		3-Nitrotoluene	8.5	0.25
		4-Nitrotoluene	7.9	0.25

* The following additional compounds may be added to this method by client request.

		Nitroglycerine	50	0.5
		PETN	50	0.5

C4-136b

TABLE 2 QUANTERRA, WEST SACRAMENTO LABORATORY CONTROL LIMITS FOR MATRIX SPIKES, MATRIX SPIKE DUPLICATES, AND SURROGATE SPIKES [1]

Analytical Method	Spiking Compounds	Spike Concentration Water (ug/L)	Spike Concentration Soil/Sediments (mg/kg)	Laboratory-Established Control Limits Percent Recovery (%) Water	Percent Recovery (%) Soil/Sediments	Relative Percent Difference (%) Water	Relative Percent Difference (%) Soil/Sediments
SW8321	HMX	50	1.0	65-135	65-135	35	35
	sym-Trinitrobenzene	50	1.0	65-135	65-135	35	35
	RDX	50	1.0	65-135	65-135	35	35
	1,3-Dinitrobenzene	50	1.0	65-135	65-135	35	35
	Nitrobenzene	50	1.0	65-135	65-135	35	35
	2,4,6-Trinitrotoluene	50	1.0	65-135	65-135	35	35
	Tetryl	50	1.0	65-135	65-135	35	35
	2,4-Dinitrotoluene	50	1.0	65-135	65-135	35	35
	2.6-Dinitrotoluene	50	1.0	65-135	65-135	35	35
	2-Am-DNT	50	1.0	65-135	65-135	35	35
	4-Am-DNT	50	1.0	65-135	65-135	35	35
	2-Nitrotoluene	50	1.0	65-135	65-135	35	35
	3-Nitrotoluene	50	1.0	65-135	65-135	35	35
	4-Nitrotoluene	50	1.0	65-135	65-135	35	35
	Surrogate: Nitrobenzene-d5	200	5.0	40-135	40-135	35	35

* The following additional compounds may be added to this method by client request.

	Nitroglycerine	800	2.0	65-135	65-135	35	35
	PETN	400	1.0	65-135	65-135	35	35

(1) The control limits are subject to change at the discretion of the laboratory.

QUANTERRA STANDARD OPERATING PROCEDURE

TITLE: INDUCTIVELY COUPLED PLASMA-ATOMIC EMISSION SPECTROSCOPY, SPECTROMETRIC METHOD FOR TRACE ELEMENT ANALYSES, SW-846 METHOD 6010B AND EPA METHOD 200.7

(SUPERSEDES: REVISION 1)

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TABLE OF CONTENTS

1. SCOPE AND APPLICATION 3
2. SUMMARY OF METHOD 3
3. DEFINITIONS 4
4. INTERFERENCES 4
5. SAFETY 5
6. EQUIPMENT AND SUPPLIES 6
7. REAGENTS AND STANDARDS 7
8. SAMPLE COLLECTION, PRESERVATION AND STORAGE 8
9. QUALITY CONTROL 8
10. CALIBRATION AND STANDARDIZATION 15
11. PROCEDURE 15
12. DATA ANALYSIS AND CALCULATIONS 19
13. METHOD PERFORMANCE 21
14. POLLUTION PREVENTION 22
15. WASTE MANAGEMENT 22
16. REFERENCES 22
17. MISCELLANEOUS (TABLES, APPENDICES, ETC.) 23

LIST OF APPENDICES:

APPENDIX A - TABLES 27
APPENDIX B - QUANTERRA ICP DATA REVIEW CHECKLIST 38
APPENDIX C - CROSS REFERENCE OF TERMS USED IN METHODSAND IN SOP 40
APPENDIX D - MSA GUIDANCE 42
APPENDIX E - TROUBLESHOOTING GUIDE 44
APPENDIX F - CONTAMINATION CONTROL GUIDELINES 46
APPENDIX G - PREVENTATIVE MAINTENANCE 48

1. SCOPE AND APPLICATION

1.1. This procedure describes the analysis of trace elements including metals in solution by Inductively Coupled Plasma -Atomic Emission Spectroscopy (ICP-AES) using SW-846 Method 6010B and EPA Method 200.7 . Table I of Appendix A lists the elements appropriate for analysis by Methods 6010B and 200.7. Additional elements may be analyzed under Methods 6010B and 200.7 provided that the method performance criteria presented in Section 13.0 are met.

1.2. ICP analysis provides for the determination of metal concentrations over several orders of magnitude. Detection limits, sensitivity and optimum concentration ranges of the metals will vary with the matrices and instrumentation used. For instance, in comparison to conventional ICP technique, ICP-Trace can achieve detection levels comparable to those determined using the graphite furnace atomic absorption spectroscopy (GFAAS) technique.

1.3. Method 6010B is applicable to the determination of dissolved, suspended, total recoverable and total elements in ground water, aqueous samples, soils, sludges, wastes, sediments, and TCLP, EP and other leachates/extracts. All matrices require digestion prior to analysis with the exception of analyses for dissolved metals in filtered and acidified aqueous samples. Although digestion is not specifically required by the method, some clients and regulators may require digestion of **dissolved samples** and this must be clarified and documented before project initiation. Silver concentrations must be below 2.0 mg/L in aqueous samples and 100 mg/kg in solid matrix samples. Precipitation may occur in samples where silver concentrations exceed these levels and lead to the generation of erroneous data.

1.4. Method 200.7 is applicable to the determination of dissolved, suspended, total recoverable, and total elements in water, waste water, and solid wastes. All matrices require digestion prior to analysis with the exception of analyses for dissolved metals in filtered and acidified aqueous samples if the criteria in Section 11.1 are met. Silver concentrations must be below 0.1 mg/L in aqueous samples and 50 mg/kg in solid matrix samples.

1.5. State-specific requirements may take precedence over this SOP for drinking water sample analyses

2. SUMMARY OF METHOD

2.1. This method describes a technique for the determination of multi elements in solution using sequential or simultaneous optical systems and axial or radial viewing of the plasma. The basis of the method is the measurement of atomic emission by an optical spectroscopic technique. Samples are nebulized and the aerosol that is produced is

transported to the plasma torch where excitation occurs. Characteristic atomic-line emission spectra are produced by a radio frequency inductively coupled plasma (ICP). The spectra are dispersed by a grating spectrometer and the intensities of the emission lines are monitored by photomultiplier tubes. The photocurrents from the photomultiplier tubes are processed and controlled by a computer system. A background correction technique is required to compensate for variable background contribution to the determination of trace elements. Background must be measured adjacent to analyte lines during analysis. The position selected for the background intensity measurement, on either or both sides of the analytical line, will be determined by the complexity of the spectrum adjacent to the analyte line. The position used must be free of spectral interferences and reflect the same change in background intensity as occurs at the analyte wavelength measured. Background correction is not required in cases of line broadening where a background correction measurement would actually degrade the analytical result. The possibility of additional interferences should also be recognized and appropriate actions taken. Alternatively, multivariate calibration methods may be chosen for which point selection for background correction is superfluous since whole spectral regions are processed.

2.2. Refer to the appropriate SOPs for details on sample preparation methods.

3. DEFINITIONS

3.1. Dissolved Metals: Those elements which pass through a 0.45 um membrane. (Sample is acidified after filtration).

3.2. Suspended Metals: Those elements which are retained by a 0.45 um membrane.

3.3. Total Metals: The concentration determined on an unfiltered sample following vigorous digestion.

3.4. Total Recoverable Metals: The concentration determined on an unfiltered sample following treatment with hot, dilute mineral acid.

4. INTERFERENCES

4.1. Spectral, physical and chemical interference effects may contribute to inaccuracies in the determinations of trace elements by ICP. Spectral interferences are caused by:

- Overlap of a spectral line from another element.
- Unresolved overlap of molecular band spectra.
- Background contribution from continuous or recombination phenomena.

- Stray light from the line emission of high concentration elements.

4.1.1. A background correction technique is required to compensate for variable background contribution to the determination of trace elements. Background correction is not required in cases where a background corrective measurement would actually degrade the analytical result.

4.1.2. Inter-element correction factors (IECs) are necessary to compensate for spectral overlap. Inter-element interferences occur when elements in the sample emit radiation at wavelengths so close to that of the analyte that they contribute significant intensity to the analyte channel. If such conditions exist, the intensity contributed by the matrix elements will cause an excessively high (or sometimes low) concentration to be reported for the analyte. Inter-element corrections IECs must be applied to the analyte to remove the effects of these unwanted emissions.

4.1.3. Physical interferences are generally considered to be effects associated with sample transport, nebulization and conversion within the plasma. These interferences may result in differences between instrument responses for the sample and the calibration standards. Physical interferences may occur in the transfer of solution to the nebulizer (e.g., viscosity effects), at the point of aerosol formation and transport to the plasma (e.g., surface tension) or during excitation and ionization processes within the plasma itself. Changes in viscosity and surface tension can cause significant inaccuracies, especially in samples containing high dissolved solids or high acid concentrations. If physical interferences are present, dilution of the sample, use of a peristaltic pump, mass flow controller, use of an internal standard and/or use of a high solids nebulizer can reduce the effect.

4.1.4. Chemical interferences are characterized by molecular compound formation, ionization effects and solute vaporization effects. Normally these effects are not significant with the ICP technique but if observed can be minimized by buffering the sample, matrix matching or standard addition procedures.

5. SAFETY

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

5.2. Eye protection that satisfies ANSI Z87.1 (as per the Chemical Hygiene Plan), laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.3. The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the Material Safety Data Sheets (MSDS) maintained in the laboratory.

5.3.1. The following materials are known to be **corrosive**:

sulfuric acid, hydrochloric acid, nitric acid and hydrofluoric acid. (NOTE: sulfuric and hydrofluoric acids are used in cleaning the ICP torch and hydrofluoric acid is also commonly used in air toxics preparations.)

~~1.1.2.~~5.3.2. The following materials are known to be **oxidizing agents**:

nitric acid and hydrogen peroxide.

~~1.1.3.~~5.3.3. The plasma emits strong UV light and is harmful to vision. **NOTE: AVOID looking directly at the plasma.**

5.3.4. The RF generator produces strong radio frequency waves, most of which are unshielded. People with pacemakers should not go near the instrument while in operation.

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

5.5. The preparation of standards and reagents will be conducted in a fume hood or well ventilated area.

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

5.7. The use of hydrofluoric acid requires special safety precautions. Consult the facility EH&S Manager and laboratory supervisor for guidance.

6. EQUIPMENT AND SUPPLIES

6.1. Inductively Coupled Plasma Atomic Emission Spectrometer equipped with autosampler and background correction.

6.2. Radio Frequency Generator.

6.3. Argon gas supply, welding grade or equivalent.

6.4. Coolflow or appropriate water cooling device.

6.5. Peristaltic Pump.

6.6. Calibrated automatic pipettes or Class A glass volumetric pipettes.

6.7. Class A volumetric flasks.

6.8. Autosampler tubes.

7. REAGENTS AND STANDARDS

7.1. Intermediate standards are purchased as custom Quanterra multi-element mixes or as single-element solutions. All standards must be stored in FEP fluorocarbon or unused polyethylene or polypropylene bottles. Intermediate standard solutions must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the intermediate solutions may be used for up to one year and must be replaced sooner if verification from an independent source indicates a problem. Expiration dates can be extended provided that the acceptance criteria described in laboratory-specific SOPs are met.

7.2. Working calibration and calibration verification solutions may be used for up to 3 months and must be replaced sooner if verification from an independent source indicates a problem. Standards should be prepared in a matrix of 5% hydrochloric and 5% nitric acids. An exception to this is in the event the Trace ICP is utilized without the internal standard. In this case, the standard acid matrix must be matched to the final preparation matrix as listed in Section 11.10.

7.3. Refer to Tables III, IV, IVA, V and VI (Appendix A) for details regarding the working standard concentrations for calibration, calibration verification, interference correction and spiking solutions.

7.4. Concentrated nitric acid (HNO_3), trace metal grade or better.

7.5. Concentrated hydrochloric acid (HCl), trace metal grade or better.

7.6. Reagent water must be produced by a Millipore DI system or equivalent. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Sample holding times for metals are six months from time of collection to the time of analysis.

8.2. Aqueous samples are preserved with nitric acid to a pH of <2 and may be stored in either plastic or glass. If boron or silica are to be determined, plastic containers are preferred. Refrigeration is not required. Preservation must be verified prior to analysis. For samples that will be analyzed by Method 200.7 for compliance with Safe Drinking Water regulations, the samples must be held for a minimum of 16 hours prior to verifying the pH.

8.3. Soil samples do not require preservation but must be stored at 4°C ± 2° until the time of preparation .

9. QUALITY CONTROL

Table VII (Appendix A) provides a summary of quality control requirements including type, frequency, acceptance criteria and corrective action.

~~1.1.~~9.1. Initial Demonstration of Capability

Prior to analysis of any analyte using either Method 200.7 or Method 6010B, the following requirements must be met.

~~1.1.1.~~9.1.1. Instrument Detection Limit (IDL) - The IDL for each analyte must be determined for each analyte wavelength used on each instrument. The IDL must be determined annually. If the instrument is adjusted in anyway that may affect the IDL, the IDL for that instrument must be redetermined. The IDL shall be determined by multiplying by 3, the standard deviation obtained from the analysis of a standard solution (each analyte in reagent water) at a concentration 3x - 5x the previously determined IDL, with seven consecutive measurements. Each measurement must be performed as though it were a separate analytical sample (i.e., each measurement must be followed by a rinse and/or any other procedure performed between the analysis of separate samples). The result of the IDL determination must be below the Quanterra reporting limit. The CLP IDL procedure can be used for this method.

9.1.2. Method Detection Limit (MDL) - An MDL must be determined for each analyte prior to the analysis of any client samples. The MDL is determined using seven replicates of reagent water, spiked with all the analytes of interest, that have been carried through the entire analytical procedure. MDLs must be redetermined on an annual basis in accordance with 40 CFR Part 136 Appendix B requirements as detailed in Quanterra QA Policy QA-005. The spike level must be between the calculated MDL and 10X the MDL to be considered valid. The result of the MDL determination must be below the Quanterra reporting limit (RL). MDL studies for the determination of metals in soil need not be performed; an appropriate soil MDL may be computed from the experimentally determined MDL for metals in aqueous solution.

9.1.3. Linear Range Verification (LR) - The linear range must be determined on an annual basis for each analyte wavelength used on each instrument. The linear range is the concentration above which results cannot be reported without dilution of the sample. The standards used to define the linear range limit must be analyzed during a routine analytical run. For the **initial** determination of the upper limit of the linear dynamic range (LDR) for each wavelength, determine the signal responses from a minimum of three to five different concentration standards across the estimated range. One standard should be near the upper limit of the estimated range. The concentration measured at the LDR must be no more than 10% less than the expected level extrapolated from lower standards. If the instrument is adjusted in any way that may affect the LRs, new dynamic ranges must be determined. The LR data must be documented and kept on file.

9.1.4. Background Correction Points - To determine the appropriate location for off-line background correction when establishing methods, the user must scan the area on either side adjacent to the wavelength and record the apparent emission intensity from all other method analytes. This spectral information must be documented and kept on file. The location selected for background correction must be either free of off-line interelement spectral interference or a

computer routine must be used for automatic correction on all determinations. Tests to determine spectral interference must be done using analyte concentrations that will adequately describe the interference. Background correction points must be set prior to determining IECs. Refer to the facility-specific instrument operation SOP and ICP instrument manual for specific procedures to be used in setting background correction points.

9.1.5. Inter-element Corrections (IECs) - ICP interelement correction factors must be determined prior to the analysis of samples and every six months thereafter. If the instrument is adjusted in any way that may affect the IECs, the IECs must be redetermined. When initially determining IECs for an instrument, wavelength scans must be performed to ensure that solutions in use are free from contaminants. If an IEC varies significantly from the previously determined IEC then the possibility of contamination should be investigated. The purity of the IEC check solution can be verified by using a standard from a second source or an alternate method (i.e., GFAA or ICP-MS). Published wavelength tables (e.g. MIT tables, Inductively Coupled Plasma-Atomic Spectroscopy: Prominent Lines) can also be consulted to evaluate the validity of the IECs. Refer to the facility specific instrument operation SOP and instrument manufacturer's recommendations for specific procedures to be used in setting IECs. An IEC must be established to compensate for any interelement interference which results in a false analyte signal greater than ± the RL as defined in Tables I, IA or II. To determine IECs, run a single element standard at the established linear range. To calculate an IEC, divide the observed concentration of the analyte by the observed concentration of the "interfering element."

Note: Trace ICP IECs are more sensitive to small changes in the plasma and instrument setup conditions. Adjustments in the IECs will be required on a more frequent basis for the Trace as reflected by the ICSA response.

~~1.1.6.~~9.1.6. Rinse Time Determination - Rinse times must be determined annually. To determine the appropriate rinse time for a particular ICP system, the linear range verification standard (see 9.1.3) should be aspirated as a regular sample followed by the analysis of a series of rinse blanks. The length of time required to reduce the analyte signals to < RL will define the rinse time for a particular ICP system. For some analytes it may be impractical to set the rinse time based on the linear range standard result (i.e., analyte not typically detected in environmental samples at that level and an excessive rinse time would be required at the linear range level). Until the required rinse time is established, the method recommends a rinse period of at least 60 seconds between samples and standards. If a memory effect is suspected, the sample must be reanalyzed after a rinse period of sufficient length. Rinse time studies can be conducted at additional concentration levels. These additional studies

must be documented and kept on file, if a concentration other than the linear range level is used to set the rinse time. The concentration levels used to establish the rinse time must be taken into consideration when reviewing the data.

9.2. Method Blank (MB) - One method blank must be processed with each preparation batch. The method blank consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. The method blank should not contain any analyte of interest at or above the reporting limit (exception: common laboratory contaminants, see below) or at or above 5% of the measured concentration of that analyte in associated samples, whichever is higher (sample result must be a minimum of 20x higher than the blank contamination level).

- If the analyte is a common laboratory contaminant (copper, iron, lead (Trace only) or zinc) the data may be reported with qualifiers if the concentration of the analyte in the method blank is less than two times the RL. **Such action must be taken in consultation with the client and must be addressed in the project narrative.**

- Repreparation and reanalysis of all samples associated with an unacceptable method blank is required when reportable concentrations are determined in the samples (see exception noted above).

- If there is no analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. **Such action must be taken in consultation with the client and must be addressed in the project narrative.**

- If the above criteria are not met and reanalysis is not possible, then the sample data must be qualified. **This anomaly must be addressed in the project narrative and the client must be notified.**

- For dissolved metals samples which have not been digested, a CCB result is reported as the method blank. The CCB run immediately prior to the start of the dissolved sample analyses must be used for this purpose. No more than 20 samples can be associated with one CCB.

9.3. Laboratory Control Sample (LCS) - One aqueous LCS must be processed with each preparation batch. The LCS must contain all analytes of interest and must be carried through the entire analytical procedure. Aqueous LCS spike levels are provided in Table III (Appendix A). The LCS is used to monitor the accuracy of the analytical process. On-going monitoring of the LCS results provides evidence that the

laboratory is performing the method within acceptable accuracy and precision guidelines.

- If any analyte is outside established control limits the system is out of control and corrective action must occur. Until in-house control limits are established, for method 6010B, a control limit of 80 - 120% (85-115% for 200.7) recovery must be applied.

- In the event that an MS/MSD analysis is not possible a Laboratory Control Sample Duplicate (LCSD) must be analyzed. The RPD of the LCS and LCSD must be compared to the matrix spike RPD limits.

- In the instance where the LCS recovery is greater than 120% (115% for 200.7) and the sample results are < RL, the data may be reported with qualifiers. **Such action must be taken in consultation with the client and must be addressed in the report narrative.**

- Corrective action will be repreparation and reanalysis of the batch unless the client agrees that other corrective action is acceptable.

- For dissolved metals samples which have not been digested, a CCV result is reported as the LCS. The CCV run immediately prior to the start of the dissolved sample analyses must be used for this purpose. No more than 20 samples can be associated with one CCV.

9.4. Matrix Spike/Matrix Spike Duplicate (MS/MSD) - One MS/MSD pair must be processed for each preparation batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Some client specific data quality objectives (DQO's) may require the use of sample duplicates in place of or in addition to MS/MSDs. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, these results may have immediate bearing only on the specific sample spiked. Samples identified as field blanks cannot be used for MS/MSD analysis. Spiking levels are provided in Tables III and VI (Appendix A).

- If any analyte recovery or RPD falls outside the acceptance range, the recovery of that analyte must be in control for the LCS. For both methods 200.7 and 6010B, control limits of 75 - 125% recovery and 20% RPD or historical acceptance criteria must be applied to the MS/MSD. If the LCS recovery is within limits, then the laboratory operation is in control and the results may be accepted. If the recovery of the LCS is outside limits corrective action must be taken. Corrective

action will include repreparation and reanalysis of the batch. MS/MSD results which fall outside the control limits must be addressed in the narrative.

- If the native analyte concentration in the MS/MSD exceeds 4x the spike level for that analyte, the recovery data are reported as NC (i.e., not calculated). If the reporting software does not have the ability to report NC then the actual recovery must be reported and narrated as follows: "Results outside of limits do not necessarily reflect poor method performance in the matrix due to high analyte concentrations in the sample relative to the spike level."

- If an MS/MSD is not possible due to limited sample volume then a laboratory control sample duplicate (LCSD) should be analyzed. The RPD of the LCS and LCSD must be compared to the matrix spike RPD limits.

- For dissolved metals samples which have not been digested, a MS/MSD must be performed per batch of up to 20 samples by spiking two aliquots of the sample at the levels specified in Table III (Appendix A).

9.5. Dilution test – A dilution test is performed to determine whether significant physical or chemical interferences exist due to the sample matrix. One sample per preparation batch must be processed as a dilution test. The test is performed by running a sample at a 5x (1:4) dilution. Samples identified as field blanks cannot be used for dilution tests. The results of the diluted sample, after correction for dilution, should agree within 10% of the original sample determination when the original sample concentration is greater than 50x the IDL. If the results are not within 10%, the possibility of chemical or physical interference exists.

9.6. Initial Calibration Verification (ICV/ICB) - Calibration accuracy is verified by analyzing a second source standard (ICV). For analyses conducted under Method 200.7, the ICV result must fall within 5% of the true value for that solution with relative standard deviation <3% from replicate (minimum of two) exposures. For Method 6010B, the ICV must fall within 10% of the true value for that solution with relative standard deviation <5% from replicate (minimum of two) exposures. An ICB is analyzed immediately following the ICV to monitor low level accuracy and system cleanliness. The ICB result must fall within +/- the RL from zero. If either the ICV or ICB fail to meet criteria, the analysis should be terminated, the problem corrected, the instrument recalibrated and the calibration reverified. (See Section 11.11 or 11.12 for required run sequence).

9.7. Continuing Calibration Verification (CCV/CCB) - Calibration accuracy is monitored throughout the analytical run through the analysis of a known standard after every 10 samples. The CCV is be a mid-range standard made from a dilution of the calibration standard. The CCV for both methods must fall within 10% of the true value for that solution with relative standard deviation <5% from replicate (minimum of two) exposures. A CCB is analyzed immediately following each CCV. (See

Section 11.11 or 11.12 for required run sequence.) The CCB result must fall within +/- RL from zero. If the blank is less than 1/10 the concentration of the action level of interest, and no sample is within 10% of the action limit, reanalysis and recalibration are not required before continuation of the run. Sample results may only be reported when bracketed by valid CCV/CCB pairs. If a mid-run CCV or CCB fails, the analysis for the affected element must be terminated, the problem corrected, the instrument recalibrated, the calibration verified and the affected samples reanalyzed. (Refer to Section 11.13 for an illustration of the appropriate rerun sequence).

9.8. Interference Check Analysis (ICSA/ICSAB) - The validity of the interelement correction factors is demonstrated through the successful analysis of interference check solutions. The ICSA contains only interfering elements, the ICSAB contains analytes and interferents. Refer to Table V (Appendix A) for the details of ICSA and ICSAB composition. Custom Quanterra multielement ICS solutions must be used. All analytes should be spiked into the ICSAB solution, therefore, if a non-routine analyte is required then it should be manually spiked into the ICSAB using a certified ultra high purity single element solution or custom lab-specific mix. If the ICP will display overcorrection as a negative number then the non-routine elements can be controlled from the ICSA as described in section 9.8.3. Elements known to be interferents on a required analyte must be included in the ICP run when that analyte is determined. Aluminum, iron, calcium and magnesium must always be included in all ICP runs.

9.8.1. The ICSA and ICSAB solutions must be run at the beginning of the run. (See Section 11.11 or 11.12 for required run sequence.)

9.8.2. The ICSAB results for the interferents must fall within 80 - 120% of the true value. If any ICSAB interferent result fails criteria, the analysis should be terminated, the problem corrected, the instrument recalibrated and the samples rerun.

9.8.3. ICSA results for the non-interfering elements with reporting limits ≤ 10 ug/L must fall within the Quanterra guidelines of ± 2x RL from zero. ICSA results for the non-interfering elements with RLs> 10 µg/L must fall within the Quanterra guidelines of ± 1x RL from zero. If the ICSA results for the non-interfering elements do not fall within +/- 2x RL (RL ≤10) or ± 1xRL (RL>10) from zero the field sample data must be evaluated as follows:

- If the non-interfering element concentration in the ICSA is the result of contamination versus a spectral interference, and this reason is documented, the field sample data can be accepted.

- If the affected element was not required then the sample data can be accepted.

- If the interfering elements are not present in the field sample at a concentration which would result in a false positive or negative result greater than +/- 2x RL from zero then the field sample data can be accepted.

- If the interfering element is present in the field sample at a level which would result in a false analyte signal greater than ± 2x RL from zero, the data can be accepted only if the concentration of the affected analyte in the field sample is more than 10x the analyte signal in the ICSA.

- If the data does not meet the above conditions then the IECs must be re-evaluated and corrected if necessary and the affected samples reanalyzed or the sample results manually corrected through application of the new IEC to the raw results. If the results are recalculated manually the calculations must be clearly documented on the raw data.

9.9. Method of Standard Addition (MSA) -This technique involves adding known amounts of standard to one or more aliquots of the processed sample solution. This technique compensates for a sample interferent that may enhance or depress the analyte signal, thus producing a different slope from that of the calibration standards. It will not correct for additive interferences which cause a baseline shift. Refer to Section 11.17 for additional information on when MSA is required as well as Appendix D for specific MSA requirements.

9.10. Quality Assurance/Project Summaries - Certain clients may require project- or program-specific QC which may supersede this SOP requirements. Quality Assurance Summaries (QASs) or equivalent documents providing project-specific requirements should be developed so that project staff clearly understand the special project requirements.

10. CALIBRATION AND STANDARDIZATION

10.1. Set up the instrument with the operating parameters recommended by the manufacturer. Allow the instrument to become thermally stable before beginning calibration (approximately 30 minutes of warm-up is required).

10.2. Profile and calibrate the instrument according to the instrument manufacturer's recommended procedures. Flush the system with the calibration blank between each standard or as the manufacturer recommends. The calibration curve must consist of a minimum of a blank and a standard. Refer to the facility-specific instrument SOP or ICP instrument manual for a detailed set up and operation protocols.

10.3. Calibration must be performed daily and each time the instrument is set up. Instrument runs may be continued over periods exceeding 24 hours as long as all

calibration verification (CCV) and interference check QC criteria are met. The instrument standardization date and time must be included in the raw data.

10.4. Refer to Section 9.0 for calibration verification procedures, acceptance criteria and corresponding corrective actions.

11. PROCEDURE

11.1. For 200.7 analyses, dissolved (preserved) samples must be digested unless it can be documented that the sample meets all of the following criteria:

A. Visibly transparent with a turbidity measurement of 1 NTU or less.

B. Is of one liquid phase and free of particulate or suspended matter following acidification.

C. Is NOT being analyzed for silver.

~~1.2.~~11.2. A minimum of two exposures for each standard, field sample and QC sample is required. The average of the exposures is reported. For Trace ICP analyses, the results of the sum channel must be used for reporting.

11.3. Prior to calibration and between each sample/standard the system is rinsed with the calibration blank solution. The minimum rinse time between analytical samples is 60 seconds unless following the protocol outlined in 9.1.6 it can be demonstrated that a shorter rinse time may be used. Triton-X can be added to the rinse solution to facilitate the rinse process.

11.4. The use of an autosampler for all runs is strongly recommended.

11.5. The use of automated QC checks through the instrument software is highly recommended for all calibration verification samples (ICV,CCV), blanks (ICB,CCB,PB), interference checks (ICSA,ICSAB) and field samples (linear range) to improve the data review process.

11.6. To facilitate the early identification of QC failures and samples requiring rerun it is strongly recommended that sample data be reviewed periodically throughout the run.

11.7. To facilitate the data review and reporting processes it is strongly recommended that all necessary dilutions be performed before closing out the instrument run.

11.8. For unattended overnight auto-runs it is strongly recommended that the frequency of ICSA/ICSAB analysis be increased to every 4 hours.

11.9. The use of an internal standard is recommended on the conventional, non-Trace ICPs as an alternative to using the method of standard additions. This technique is useful

in overcoming matrix interferences especially in high solids matrices. However, for conventional ICP techniques, internal standards may not be necessary provided that one of the following is performed to minimize physical interferences: (1) peristaltic pump is used, (2) high solids nebulizer is used, or (3) high solids samples are diluted and reanalyzed.

11.10. The use of an internal standard is **required** on the Trace ICP unless the calibration and QC standards are matrix matched to each digestion procedure used as follows:

Preparation Method	% HNO_3	% HCl
CLP Aqueous	1	5
CLP Soil	5	2.5
SW846 3050	5	10
SW846 3005	2	5
SW846 3010	3	5

The following procedural guidelines must be followed when using an internal standard:

~~1.1.1.~~11.10.1. Typically used internal standards are: yttrium or scandium. (Note: Any element can be used that is not typically found in environmental samples at a high rate of occurrence.)

11.10.2. The internal standard (IS) must be added to every sample and standard at the same concentration. It is recommended that the IS be added to each analytical sample automatically through use of a third pump channel and mixing coil. Internal standards should be added to blanks, samples and standards in a like manner, so that dilution effects resulting from the addition may be disregarded.

11.10.3. The concentration of the internal standard should be sufficiently high to obtain good precision in the measurement of the IS analyte used for data correction and to minimize the possibility of correction errors if the IS analyte is naturally present in the sample.

11.10.4. The internal standard raw intensity counts must be printed on the raw data.

11.10.5. The analyst must monitor the response of the internal standard throughout the sample analysis run. This information is used to detect potential problems and identify possible background contributions from the sample (i.e., natural occurrence of IS analyte).

11.10.5.1. If the internal standard counts fall within ±30% of the counts observed in the ICB then the data is acceptable.

11.10.5.2. If the internal standard counts in the field samples are more than ±30% higher than the expected level, the field samples must then be:

(1) Diluted and reanalyzed;

(2) The IS concentrations must be raised; or

(3) A different internal standard must be used.

~~1.11.~~11.11. The following analytical sequence must be used for Methods 6010B and 200.7:

Instrument Calibration
ICV
ICB
ICSA
ICSAB
8 samples
CCV
CCB
10 samples
CCV
CCB
Repeat sequence of up to 10 samples between CCV/CCB pairs as required to complete run
CCV
CCB

Refer to Quality Control Section 9.0 and Table VII (Appendix A) for Method 6010B and 200.7 quality control criteria.

~~1.12.~~11.12. Additional quality control analyses are necessary for analysis under the Contract Laboratory Program (CLP). If these are included then CLP, 6010 and 200.7 samples can be included in the same sequence. Refer to CORP-MT-002 for details.

11.13. Full method required QC must be available for each wavelength used in determining reported analyte results.

11.14. Guidelines are provided in the appendices on procedures to minimize contamination of samples and standards, preventive maintenance and troubleshooting.

11.15. All measurements must fall within the defined linear range where spectral interference correction factors are valid. Dilute and reanalyze all samples for required analytes that exceed the linear range or use an alternate wavelength for which QC data are established. If an interelement correction exists for an analyte which exceeds the linear range, the IEC may be inaccurately applied. Therefore, even if an overrange analyte may not be required to be reported for a sample, if that analyte is a interferent for any requested analyte in that sample, the sample must be diluted. Acid strength must be maintained in the dilution of samples.

11.16. For TCLP samples, full four-point MSA will be required if all of the following conditions are met:

1) recovery of the analyte in the matrix spike is not at least 50%,

2) the concentration of the analyte does not exceed the regulatory level, and,

3) the concentration of the analyte is within 20% of the regulatory level.

The reporting and regulatory limits for TCLP analyses as well as matrix spike levels are detailed in Table VI (Appendix A). Appendix E provides guidance on performing MSA analyses.

~~1.17.~~11.17. Any variation in procedure shall be completely documented using instrument run logs, maintenance logs, report narratives, a Nonconformance Memo, or an anomaly report and is approved by a Supervisor/Group Leader and QA Manager. If contractually required, the client shall be notified by the Project Manager.

11.18. Nonconformance documentation shall be filed in the project file.

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

12. DATA ANALYSIS AND CALCULATIONS

12.1. ICV percent recoveries are calculated according to the equation:

$$\%R = 100\left(\frac{Found(ICV)}{True(ICV)}\right)$$

~~1.2.~~12.2. CCV percent recoveries are calculated according to the equation:

$$\%R = 100\left(\frac{Found(CCV)}{True(CCV)}\right)$$

~~1.3.~~12.3. Matrix Spike Recoveries are calculated according to the following equation:

$$\%R = 100\left(\frac{SSR - SR}{SA}\right)$$

Where:
SSR = Spike Sample Result
SR = Sample Result
SA = Spike Added

~~1.4.~~12.4. The relative percent difference (RPD) of matrix spike/matrix spike duplicates are calculated according to the following equations:

$$RPD = 100\left[\frac{|MSD - MS|}{\left(\frac{MSD + MS}{2}\right)}\right]$$

Where:
MS = determined spiked sample concentration
MSD = determined matrix spike duplicate concentration

~~1.5.~~12.5. The final concentration for a digested aqueous sample is calculated as follows:

$$mg/L = \frac{C \times V1 \times D}{V2}$$

Where:
C = Concentration (mg/L) from instrument readout
D = Instrument dilution factor
V1 = Final volume in liters after sample preparation
V2 = Initial volume of sample digested in liters

~~1.6.~~12.6. The final concentration determined in digested solid samples when reported on a dry weight basis is calculated as follows:

$$mg / Kg, dry\ weight = \frac{C \times V \times D}{W \times S}$$

Where:

C = Concentration (mg/L) from instrument readout
D = Instrument dilution factor
V = Final volume in liters after sample preparation
W = Weight in Kg of wet sample digested
S = Percent solids/100

Note: A Percent Solids determination must be performed on a separate aliquot when dry weight concentrations are to be reported. If the results are to be reported on wet weight basis the "S" factor should be omitted from the above equation.

~~1.7.~~12.7. The LCS percent recovery is calculated according to the following equation:

$$\%R = 100\left(\frac{Found(LCS)}{True(LCS)}\right)$$

~~1.8.~~12.8. The dilution test percent difference for each component is calculated as follows:

$$\%Difference = \frac{|I - S|}{I} \times 100$$

Where:

I = Sample result (Instrument reading)
S = Dilution test result (Instrument reading × 5)

~~1.9.~~12.9. Appropriate factors must be applied to sample values if dilutions are performed.

12.10. Sample results should be reported with up to three significant figures in accordance with the Quanterra significant figure policy.

13. METHOD PERFORMANCE

13.1. Each laboratory must have initial demonstration of performance data on file for each analyte of interest as described in Section 9.0.

13.2. Refer to Tables I, IA & II in Appendix A for the list of Method 6010B and 200.7 analytes as well as additional analytes that may be analyzed using this SOP.

13.3. Method performance is determined by the analysis of MS and MSD samples as well as method blanks and laboratory control samples. The MS or MSD recovery should fall within +/- 25 % and the MS/MSD should compare within 20% RPD or within the laboratory's historical acceptance limits. These criteria apply to analyte concentrations greater than or equal to 10xIDL. Method blanks must meet the criteria specified in Section 9.2. The laboratory control samples should recover within 20% (15% for 200.7) of the true value or within the laboratory's historical acceptance limits.

13.4. Training Qualification:

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

14. POLLUTION PREVENTION

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

15. WASTE MANAGEMENT

15.1. Waste generated in the procedure must be segregated and disposed of according to the facility hazardous waste procedures and per the local, state, and federal regulations. The Environmental Health and Safety Director should be contacted, if additional information is required.

15.2. Standards should be purchased and prepared in volumes consistent with laboratory use to minimize the volume of expired standards to be disposed.

16. REFERENCES

16.1. 40 CFR Part 136, Appendix B, 7-5-95, Determination of Method Detection Limits.

16.2. Test Methods for Evaluating Solid Waste , Physical/Chemical Methods, SW-846, 3rd Edition, Final Update III, Revision 2, December 1996. Method 6010B.

16.3. Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma-Atomic Emission Spectrometry, Revision 4.4, May 1994. Method 200.7.

16.4. CORP-MT-0002, Inductively Coupled Plasma-Atomic Emission Spectroscopy, Method 200.7 & CLP-M, SOW ILMO3.0.

16.5. QA-003, Quanterra QC Program.

16.6. QA-004, Rounding and Significant Figures.

16.7. QA-005, Method Detection Limits.

17. MISCELLANEOUS (TABLES, APPENDICES, ETC.)

17.1. Modifications/Interpretations from reference method

17.1.1. Modifications/interpretations from both Methods 6010B and 200.7.

17.1.1.1. Quanterra laboratories use mixed calibration standard solutions purchased from approved vendors instead of using individual mixes prepared in house as recommended by the subject methods.

17.1.1.2. The alternate run sequence presented in Section 11.12 is consistent with method requirements. Additional QC (i.e., ICSA) analyses were added to accommodate the CLP protocol requirements.

17.1.1.3. Methods 200.7 and 6010B state that if the correction routine is operating properly, the determined apparent analyte(s) concentration from analysis of each interference solution should fall within a specific concentration range around the calibration blank. In determining IECs, because of lack of definition clarification for "concentration range around the calibration blank," Quanterra has adopted the procedure in EPA CLP ILMO4.0.

17.1.1.4. Section 8.5 of Method 6010B and Section 9.5 of Method 200.7 recommend that whenever a new or unusual matrix is encountered, a series of tests be performed prior to reporting concentration data for that analyte. The dilution test helps determine if a chemical or physical interference exists. Because Quanterra laboratories receive no prior information from clients regarding when to expect a new or unusual matrix, Quanterra may select to perform a dilution test on one sample in each prep batch. According to the method, the post digestion spike (PDS) determines any potential matrix interferences. At Quanterra labs, matrix interference is determined by evaluating data for the LCS and MS/MSD. Quanterra requires documented, clear guidance when a new or unusual matrix will be received for a project and a request to perform the dilution test or PDS on a client-identified sample.

17.1.2. Modifications from Method 200.7.

17.1.2.1. Method 200.7 defines the IDL as the concentration equivalent to a signal, due to the analyte, which is equal to three times the standard deviation of a series of ten replicate measurements of the calibration blank signal at the same wavelength. Quanterra labs utilize the CLP IDL definition as defined in Section 9.1.1 of this SOP.

17.1.2.2. The calibration blank is prepared in an acid matrix of 5% HNO_3/5% HCl instead of the specified 2% HNO_3/10% HCl matrix as the former matrix provides for improved performance relative to the wide variety of digestate acid matrices which result from the various EPA preparation protocols applied.

17.1.2.3. Method section 9.3.4 specifies that "Analysis of the IPC (ICSA/AB) solution immediately following calibration must verify that the instrument is within $\pm$ 5% of calibration with a relative standard deviation <3% from replicate integrations $\geq$ 4." Quanterra uses a minimum of two exposures.

17.1.2.4. Section 7.12 of 200.7 indicates that the QCS (ICV) should be prepared at a concentration near 1 ppm. The ICV specified in this SOP accommodates the 1 ppm criteria for the majority of analytes. For the remaining analytes, this SOP specifies ICV concentrations which are appropriate to the range of calibration. The intent of the ICV, verification of calibration standard accuracy, is independent of the ICV concentration used.

17.1.2.5. The ICS criteria applied by this SOP differ from those stated in the method. Method 200.7 section 10.4 states that results should fall within the established control limits of 3 times the standard deviation of the calibration blank for that analyte. The control limits listed in this SOP are those applicable to the EPA designed solution.

17.1.2.6. Method 200.7 section 9.3.4 states the CCB should be less than the IDL, but > the lower 3-sigma control limit of the calibration blank. The intent of this requirement is to ensure that the calibration is not drifting at the low end. Quanterra has adopted an absolute control limit of +/- RL from zero for calibration blank criteria. SOP section 9.7 provides the detailed corrective action criteria that must be followed.

17.1.3. Modifications from Method 6010B.

17.1.3.1. Chapter 1 of SW-846 states that the method blank should not contain any analyte of interest at or above the MDL. This SOP states that the method blank must not contain any analyte of interest at or above the reporting limit. Common lab contaminants are allowed up to two times the reporting limit in the blank following consultation with the client.

17.1.3.2. Method 6010B section 8.6.1.3 states that the results of the calibration blank are to agree within 3x the IDLIf not, repeat the analysis two or more times and average the results. If the average is not within three standard deviation of the background mean, terminate the analysis, correct the problem, recalibrate, and reanalyze the previous 10 samples. The intent of this requirement is to ensure that the calibration is not drifting at the low end. Quanterra has adopted an absolute control limit of +/- RL from zero for calibration blank criteria. See SOP Section 9.7 for a detailed description of the required corrective action procedures.

17.2. Modifications from previous SOP

Refer to revision 1 of this SOP.

~~1.3.~~17.3. Facility-Specific SOPs

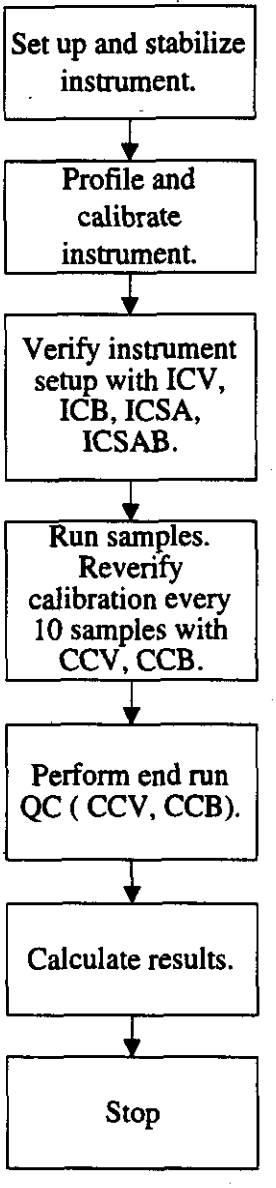
Each facility shall review and revise as appropriate this SOP to reflect any facility-specific requirementsIf no facility-specific amendments are required, the SOP can be adopted as is.

~~1.4.~~17.4. Documentation and Record Management

The following documentation comprises a complete ICP raw data package:

- Raw data (direct instrument printout).
- Relevant sample preparation benchsheets.
- Run log printout from instrument software where this option is available (TJA) or manually generated run log (i.e., Ward WSL printout).
- Data review checklist - See Appendix B.
- Standards documentation (including prep and expiration dates, source, and lot #).
- Nonconformance/anomaly documentation (if applicable).

17.5. Flow Diagram



APPENDIX A

TABLES

TABLE I. Method 200.7 and 6010B Target Analyte List

ELEMENT	Symbol	CAS #	6010B analyte	200.7 analyte	Reporting Limit (ug/L) Water	Reporting Limit (mg/kg) Soil
Aluminum	Al	7429-90-5	X	X	200	20
Antimony	Sb	7440-36-0	X	X	60	6
Arsenic	As	7440-38-2	X	X	300	30
Barium	Ba	7440-39-3	X	X	200	20
Beryllium	Be	7440-41-7	X	X	5.0	0.5
Boron	B	7440-42-8		X	200	20
Cadmium	Cd	7440-43-9	X	X	5.0	0.5
Calcium	Ca	7440-70-2	X	X	5000	500
Chromium	Cr	7440-47-3	X	X	10	1
Cobalt	Co	7440-48-4	X	X	50	5
Copper	Cu	7440-50-8	X	X	25	2.5
Iron	Fe	7439-89-6	X	X	100	10
Lead	Pb	7439-92-1	X	X	100	10
Lithium	Li	7439-93-2	X	X	50	5
Magnesium	Mg	7439-95-4	X	X	5000	500
Manganese	Mn	7439-96-5	X	X	15	1.5
Molybdenum	Mo	7439-98-7	X	X	40	4
Nickel	Ni	7440-02-0	X	X	40	4
Phosphorus	P	7723-14-0	X	X	300	30
Potassium	K	7440-09-7	X	X	5000	500
Selenium	Se	7782-49-2	X	X	250	25
Silicon	Si	7631-86-9		X	500	N/A
Silver	Ag	7440-22-4	X	X	10	1
Sodium	Na	7440-23-5	X	X	5000	500
Strontium	Sr	7440-24-6	X		50	5
Thallium	Tl	7440-28-0	X	X	2000	200
Vanadium	V	7440-62-2	X	X	50	5
Zinc	Zn	7440-66-6	X	X	20	2

TABLE IA. Method 200.7 and 6010B Trace ICP Target Analyte List

ELEMENT	Symbol	CAS #	Reporting Limit (ug/L) Water	Reporting Limit (mg/kg) Soil
Arsenic	As	7440-38-2	10	1.0
Lead	Pb	7439-92-1	3.0	0.3
Selenium	Se	7782-49-2	5.0	0.5
Thallium	Tl	7440-28-0	10	1.0
Antimony	Sb	7440-36-0	10	1.0
Cadmium	Cd	7440-43-9	2.0	0.2
Silver	Ag	7440-22-4	5.0	0.5
Chromium	Cr	7440-47-3	5.0	0.5

TABLE II. Non-Routine Analyte List

ELEMENT	Symbol	CAS #	Reporting Limit (ug/L) Water	Reporting Limit (mg/kg) Soil
Tin	Sn	7440-31-5	100	10
Titanium	Ti	7440-32-6	50	5
Bismuth	Bi	7440-06-99	200	20
Zirconium	Zr	7440-06-77	100	10
Tungsten	W	7440-03-37	500	50
Tellurium	Te	1349-48-09	500	50
Thorium	Th	7440-02-91	500	50
Uranium	U	7440-06-11	500	50
Palladium	Pd	7440-00-53	100	10

NOTE: Analysis of all elements listed may not be available at all Quanterra facilities.

TABLE III. Matrix Spike and Aqueous Laboratory Control Sample Levels

ELEMENT	LCS Level (ug/L)	Matrix Spike Level (ug/L)
Aluminum	2000	2000
Antimony	500	500
Arsenic	2000	2000
Barium	2000	2000
Beryllium	50	50
Cadmium	50	50
Calcium	50000	50000
Chromium	200	200
Cobalt	500	500
Copper	250	250
Iron	1000	1000
Lead	500	500
Lithium	1000	1000
Magnesium	50000	50000
Manganese	500	500
Molybdenum	1000	1000
Nickel	500	500
Phosphorous	10000	10000
Potassium	50000	50000
Selenium	2000	2000
Silver	50	50
Sodium	50000	50000
Strontium	1000	1000
Thallium	2000	2000
Vanadium	500	500
Zinc	500	500
Boron	1000	1000
Silicon	10000	10000
Tin	2000	2000
Titanium	1000	1000
Bismuth	1000	1000
Zirconium	1000	1000
Tellurium	1000	1000
Thorium	1000	1000
Uranium	1000	1000
Tungsten	1000	1000
Palladium	1000	1000

TABLE IV. ICP Calibration and Calibration Verification Standards

Element	Calibration Level	RL (ug/L)	ICV (ug/L)	CCV (ug/L)
Aluminum	100000	200	25000	50000
Antimony	10000	60	1000	5000
Arsenic	10000	300	1000	5000
Barium	10000	200	1000	5000
Beryllium	10000	5	1000	5000
Cadmium	10000	5	1000	5000
Calcium	100000	5000	25000	50000
Chromium	10000	10	1000	5000
Cobalt	10000	50	1000	5000
Copper	10000	25	1000	5000
Iron	100000	100	25000	50000
Lead	10000	100	1000	5000
Lithium	10000	50	1000	5000
Magnesium	100000	5000	25000	50000
Manganese	10000	15	1000	5000
Molybdenum	10000	40	1000	5000
Nickel	10000	40	1000	5000
Phosphorous	10000	300	1000	5000
Potassium	100000	5000	25000	50000
Selenium	10000	250	1000	5000
Silver	2000	10	500	1000
Sodium	100000	5000	25000	50000
Strontium	10000	50	1000	5000
Thallium	20000	2000	5000	10000
Vanadium	10000	50	1000	5000
Zinc	10000	20	1000	5000
Boron	10000	200	1000	5000
Silicon	10000	500	1000	5000
Tin	10000	100	1000	5000
Titanium	10000	50	1000	5000
Bismuth	10000	200	1000	5000
Zirconium	10000	100	1000	5000
Tellurium	10000	500	1000	5000
Thorium	10000	500	1000	5000
Uranium	10000	500	1000	5000
Tungsten	10000	500	1000	5000
Palladium	10000	100	1000	5000

TABLE IVA. Trace ICP Calibration and Calibration Verification Standards

Element	Calibration Level	RL (ug/L)	ICV (ug/L)	CCV (ug/L)
Aluminum	50000	200	12500	25000
Antimony	1000	10	250	500
Arsenic	1000	10	250	500
Barium	4000	10	1000	2000
Beryllium	4000	5	1000	2000
Cadmium	1000	2	250	500
Calcium	100000	5000	25000	50000
Chromium	4000	5	1000	2000
Cobalt	4000	50	1000	2000
Copper	4000	25	1000	2000
Iron	50000	100	12500	25000
Lead	1000	3	250	500
Magnesium	100000	5000	25000	50000
Manganese	4000	15	1000	2000
Molybdenum	4000	40	1000	2000
Nickel	4000	40	1000	2000
Potassium	100000	5000	25000	50000
Selenium	1000	5	250	500
Silver	2000	5	500	1000
Sodium	100000	5000	25000	50000
Thallium	2000	10	500	1000
Vanadium	4000	50	1000	2000
Zinc	4000	20	1000	2000

TABLE V. Interference Check Sample Concentrations*

Element	ICSA (ug/L)	ICSAB (ug/L)
Aluminum	500000	500000
Antimony	-	1000
Arsenic	-	1000
Barium	-	500
Beryllium	-	500
Cadmium	-	1000
Calcium	500000	500000
Chromium	-	500
Cobalt	-	500
Copper	-	500
Iron	200000	200000
Lead	-	1000
Magnesium	500000	500000
Manganese	-	500
Molybdenum	-	1000
Nickel	-	1000
Potassium	-	10000
Selenium	-	1000
Silver	-	1000
Sodium	-	10000
Thallium	-	10000**
Vanadium	-	500
Zinc	-	1000
Tin	-	1000

* Custom Quanterra solutions contain analytes common to all Quanterra facilities. Non-routine elements not listed above should be spiked into the ICSAB at 1000 ug/L.

** Thallium level for Trace ICP should be at 1000 ug/L.

TABLE VI. TCLP Reporting Limits, Regulatory Limits and Matrix Spike Levels

ELEMENT	Reporting Level (ug/L)	Regulatory Limit (ug/L)	Spike Level (ug/L)
Arsenic	500	5000	5000
Barium	10000	100000	50000
Cadmium	100	1000	1000
Chromium	500	5000	5000
Lead	500	5000	5000
Selenium	250	1000	1000
Silver	500	5000	1000

TABLE VII. Summary Of Quality Control Requirements

QC PARAMETER	FREQUENCY*	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Two-point Initial Calibration	Beginning of every analytical run, every 24 hours, whenever instrument is modified, or CCV criterion is not met	RSD between duplicate exposures ≤5%	Terminate analysis; Correct the problem; Prepare new standards; Recalibrate following system performance.
ICV	Beginning of every analytical run.	Method 200.7: 95 - 105 % recovery. Method 6010B: 90 - 110 % recovery.	Terminate analysis; Correct the problem; Recalibrate.
ICB	Beginning of every analytical run, immediately following the ICV.	The result must be within +/- RL from zero.	Terminate analysis; Correct the problem; Recalibrate.
CCV	Every 10 samples and at the end of the run.	Method 200.7 & 6010B: 90 - 110 % recovery.	Terminate analysis; Correct the problem; Recalibrate and rerun all samples not bracketed by acceptable CCV.
CCB	Immediately following each CCV.	The result must be within +/- RL from zero.	Terminate analysis; Correct the problem; Recalibrate and rerun all samples not bracketed by acceptable CCB.
ICSA	Beginning of every run	See Section 9.8.3	See Section 9.8.3.
ICSAB	Immediately following each ICSA.	Results must be within 80 - 120% recovery.	See Section 9.8.2.

* See Sections 11.11 and 11.12 for exact run sequence to be followed.

TABLE VII. Summary of Quality Control Requirements (Continued)

QC PARAMETER	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Dilution Test	One per prep batch.	For samples > 50x IDL, dilutions must agree within 10%.	Narrate the possibility of physical or chemical interference per client request.
Method Blank	One per sample preparation batch of up to 20 samples.	The result must be less than or equal to the RL. Common lab contaminants may be accepted up to 2x the RL after consultation with the client (See 9.2). Sample results greater than 10x the blank concentration are acceptable. Samples for which the contaminant is < RL may not require redigestion or reanalysis (see Section 9.2).	Redigest and reanalyze samples. Note exceptions under criteria section. See Section 9.2 for additional requirements.
Laboratory Control Sample (LCS)	One per sample preparation batch of up to 20 samples.	Aqueous LCS must be within 80 - 120% recovery or in-house control limits. (85-115% for 200.7) Samples for which the contaminant is < RL and the LCS results are > 120% (115% for 200.7) may not require redigestion or reanalysis (see Section 9.3)	Terminate analysis; Correct the problem; Redigest and reanalyze all samples associated with the LCS.

TABLE VII. Summary of Quality Control Requirements (Continued)

QC PARAMETER	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Matrix Spike	One per sample preparation batch of up to 20 samples.	75 - 125 % recovery or in-house control limits. I For TCLP See Section 11.17.	In the absence of client specific requirements, flag the data; no flag required if the sample level is > 4x the spike added. For TCLP see Section 11.17.
Matrix Spike Duplicate	See Matrix Spike	75 - 125 % recovery; RPD ≤ 20% .	See Corrective Action for Matrix Spike.

APPENDIX B

QUANTERRA ICP DATA REVIEW CHECKLIST

Quanterra ICP Data Review Checklist

Run/Project Information:

Run Date: _____ **Analyst:** _____ **Instrument:** _____
Prep Batches Run: _____

Circle Methods used: **6010B / 200.7: CORP-MT-0001 Rev 2**
CLP ILMO3.0/4.0 : CORP-MT-0002 Rev 1

Review Items

A. Calibration/Instrument Run QC	Yes	No	N/A	2nd Level
1. Instrument calibrated per manufacturer's instructions and at SOP specified levels ?				
2. ICV/CCV analyzed at appropriate frequency and within control limits ? (6010B, CLP = 90 - 110%, 200.7 = 95 -105%[ICV])				
3. ICB/CCB analyzed at appropriate frequency and within +/- RL or +/- CRDL (CLP) ?				
4. CRI analyzed? (for CLP only)				
5. ICSA/ICSAB run at required frequency and within SOP limits ?				
B. Sample Results				
1. Were samples with concentrations > the linear range for any parameter diluted and reanalyzed ?				
2. All reported results bracketed by in control QC ?				
3. Sample analyses done within holding time ?				
C. Preparation/Matrix QC				
1. LCS done per prep batch and within QC limits ?				
2. Method blank done per prep batch and < RL or CRDL (CLP) ?				
3. MS run at required frequency and within limits ?				
4. MSD or DU run at required frequency and RPD within SOP limits ?				
5. Dilution Test done per prep batch (or per SDG for CLP) ?				
6. Post digest spike analyzed if required (CLP only) ?				
D. Other				
1. Are all nonconformances documented appropriately ?				
2. Current IDL/LR/IEC data on file ?				
3. Calculations checked for error ?				
4. Transcriptions checked for error ?				
5. All client/project specific requirements met ?				
6. Date/time of analysis verified as correct ?				

Analyst: _____ **Date:** _____
Comments: _____

2nd Level Reviewer : _____ **Date:** _____
Comments: _____

APPENDIX C

CROSS REFERENCE OF TERMS USED IN METHODS 6010B, 200.7, AND BY QUANTERRA

CROSS REFERENCE OF TERMS COMMONLY USED IN METHODS EPA 200.7, SW6010B, AND QUANTERRA INC. SOP

EPA 200.7	SW6010B	Quanterra Inc. SOP
Calibration blank (CB)	Calibration blank	Initial and continuing calibration blanks (ICB/CCB)
Dilution test	Dilution test	Dilution Test
Instrument detection limit (IDL)	Instrument detection limit (IDL)	Instrument detection limit (IDL)
Instrument performance check (IPC)	Continuing calibration verification (CCV)	Continuing calibration verification (CCV)
Internal standard	Internal standard	Internal standard (IS)
Laboratory duplicates	n/a	n/a
Laboratory fortified blank (LFB)	n/a	Laboratory control sample (LCS)
Laboratory fortified sample matrix (LFM)	Matrix spike and matrix spike duplicate (MS/MSD)	Matrix spike and matrix spike duplicate (MS/MSD)
Laboratory reagent blank (LRB)	Method blank	Method or Prep blank (MB)
Linear dynamic range (LDR)	Linear dynamic range (LDR)	Linear dynamic range (LDR)
Method detection limit (MDL)	Method detection limit (MDL)	Method detection limit (MDL)
Quality control sample (QCS)	Check standard or Initial calibration verification (ICV)	Initial calibration verification (ICV)
Spectral interference check solution (SIC)	Interference check solution (ICS)	Interference check solution (ICSA/ICSAB)

APPENDIX D

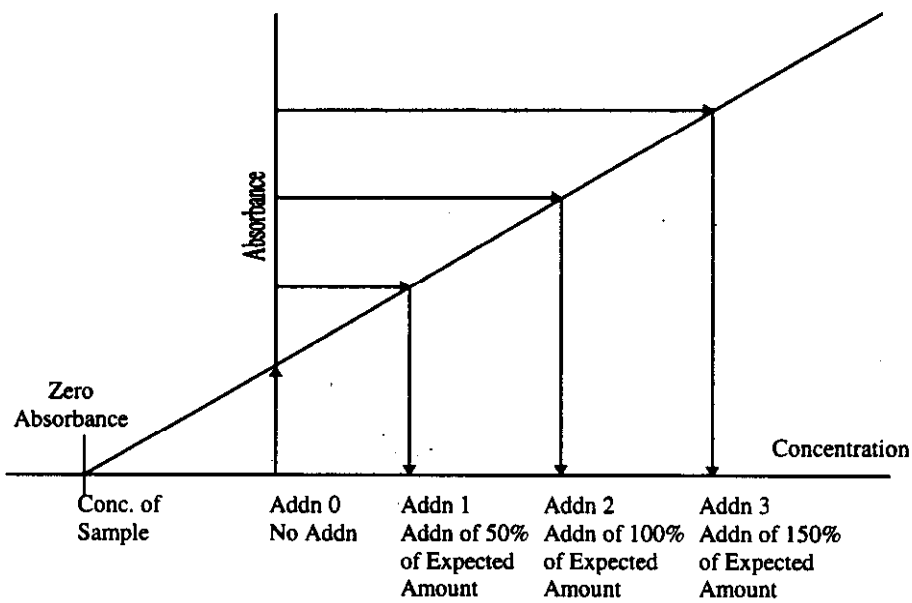
MSA GUIDANCE

Appendix D. MSA Guidance

Method of Standard Addition

Four equal volume aliquots of sample are measured and known amounts of standards are added to three aliquots. The fourth aliquot is the unknown and no standard is added to it. The concentration of standard added to the first aliquot should be 50% of the expected concentration. The concentration of standard added to the second aliquot should be 100% of the expected concentration and the concentration of standard added to the third aliquot should be 150% of the expected concentration. The volume of the unspiked and spiked standard should be the same.

In order to determine the concentration of analyte in the sample, the analytical value of each solution is determined and a plot or linear regression performed. On the vertical axis the analytical value is plotted versus the concentrations of the standards on the horizontal axis. An example plot is shown in Figure 1. When the resulting line is extrapolated back to zero absorbance, the point of interception of the horizontal axis is the concentration of the unknown.



For the method of standard additions to be correctly applied, the following limitations must be taken into consideration:

- The plot of the sample and standards must be linear over the concentration range of concern. For best results, the slope of the curve should be similar to that of a plot of the aqueous standard curve.
- The effect of the interference should not vary as the ratio of the standard added to the sample matrix changes.

APPENDIX E

TROUBLESHOOTING GUIDE

APPENDIX E. TROUBLESHOOTING GUIDE

Problem	Possible Cause/ Solution
High Blanks	Increase rinse time Clean or replace tip Clean or replace torch Clean or replace sample tubing Clean or replace nebulizer Clean or replace mixing chamber Lower Torch
Instrument Drift	RF not cooling properly Vacuum level is too low Replace torch (Crack) Clean or replace nebulizer (blockage) Check room temperature (changing) Replace pump tubing Room humidity too high Clean torch tip (salt buildup) Check for argon leaks Adjust sample carrier gas Reprofile Horizontal Mirror Replace PA tube
Erratic Readings, Flickering Torch or High RSD	Check for argon leaks Adjust sample carrier gas Replace tubing (clogged) Check drainage(back pressure changing) Increase uptake time (too short) Increase flush time (too short) Clean nebulizer, torch or spray chamber Increase sample volume introduced Check that autosampler tubes are full Sample or dilution of sample not mixed Increase integration time (too short) Realign torch Reduce amount of tubing connectors
Cu/Mn Ratio Outside Limits or Low Sensitivity	Plasma conditions changed Clean nebulizer, torch or spray chamber Replace tubing (clogged) Realign torch Check IECs
Standards reading twice normal absorbance or concentration	Incorrect standard used Incorrect dilution performed

APPENDIX F

CONTAMINATION CONTROL GUIDELINES

APPENDIX F. CONTAMINATION CONTROL GUIDELINES

The following procedures are strongly recommended to prevent contamination:

All work areas used to prepare standards and spikes should be cleaned before and after each use.

All glassware should be washed with detergent and tap water and rinsed with 1:1 nitric acid followed by deionized water.

Proper laboratory housekeeping is essential in the reduction of contamination in the metals laboratory. All work areas must be kept scrupulously clean.

Powdered or Latex Gloves must not be used in the metals laboratory since the powder contains silica and zinc as well as other metallic analytes. Only vinyl or nitrile gloves should be used in the metals laboratory.

Glassware should be periodically checked for cracks and etches and discarded if found. Etched glassware can cause cross contamination of any metallic analytes.

Autosampler trays should be covered to reduce the possibility of contamination. Trace levels of elements being analyzed in the samples can be easily contaminated by dust particles in the laboratory.

The following are helpful hints in the identification of the source of contaminants:

Yellow pipet tips and volumetric caps can sometimes contain cadmium.

Some sample cups have been found to contain lead.

The markings on glass beakers have been found to contain lead. If acid baths are in use for glassware cleaning, they should be periodically checked for contaminants since contaminant concentrations will increase over time.

New glassware especially beakers can be a source of silica and boron.

Reagents or standards can contain contaminants or be contaminated with the improper use of a pipette.

Improper cleaning of glassware can cause contamination.

Latex gloves contain over 500 ppb of zinc.

APPENDIX G

PREVENTIVE MAINTENANCE

APPENDIX G. PREVENTIVE MAINTENANCE

A maintenance log is used to record when maintenance is performed on instruments. When an instrument problem occurs indicate the date, time and instrument number, then identify the problem and corrective action in the maintenance log.

The following procedures are required to ensure that that the instrument is fully operational.

Daily	Change sample pump tubing and pump windings Check argon gas supply level Check rinse solution and fill if needed Check waste containers and empty if needed Check sample capillary tubing is clean and in good condition Check droplet size to verify nebulizer is not clogged. Check sample flow for cross flow nebulizer Check Cu/Mn ratio-should be 30% of value at date that IECs were performed Check pressure for vacuum systems
As Needed	Clean plasma torch assembly to remove accumulated deposits Clean nebulizer and drain chamber; keep free-flowing to maintain optimum performance Replace peristaltic pump tubing, sample capillary tubing and autosampler sipper probe
Weekly	Apply silicon spray on autosampler tracks Check water level in coolflow
Monthly	Clean air filters on back of power unit to remove dust Check D mirror for air instruments
Bi-yearly	Change oil for vacuum systems Replace coolant water filter (may require more or less frequently depending on quality of cooling water)

Quanterra
Environmental Services

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SOP No. SAC-MT-0001
Revision No. 1.
Revision Date: 9/23/96

OPERATION-SPECIFIC STANDARD OPERATING PROCEDURE

TITLE: ANALYSIS OF METALS BY INDUCTIVELY COUPLED PLASMA/MASS SPECTROMETRY

(SUPERSEDES: LM-CAL-2041 ORIGINAL, NOVEMBER 1, 1993)

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C-4-186

1. SCOPE AND APPLICATION

1.1. Analytes

1.1.1. This method is applicable to the determination of metals by inductively coupled plasma mass spectrometry (ICP-MS).

1.2. Applicable matrices.

1.2.1. This method is applicable to drinking, surface, and saline waters; soil and waste samples.

1.3. Reporting Limits

1.3.1. The Quanterra Inc. (West Sacramento) standard reporting limits for metals analyzed by ICP-MS are listed in Appendix A.

1.3.2. Upon client request, results below the standard reporting limit but above the current instrument detection limit (IDL) may be qualified using a "J" flag. The flag indicates that the flagged value is considered "estimated" as defined by U.S. EPA data validation guidelines.

1.4. Analysis Time

1.4.1. The analysis time varies depending on the number of analytes and the memory characteristics of those analytes quantitated in an analysis run.

2. SUMMARY OF METHOD

2.1. Aqueous samples, digestates or leachates are nebulized into a spray chamber where a stream of argon carries the sample aerosol through a quartz torch and injects it into a radio frequency plasma. There the sample is decomposed and desolvated. The ions produces are entrained in the plasma gas and by means of a water-cooled, differentially pumped interface, introduced into a high-vacuum chamber that houses a quadrupole mass spectrometer. The ions are sorted according to their mass-to-charge ratio and measured with a channel electron multiplier.

3. DEFINITIONS

4. INTERFERENCES

4.1.1. Isobaric interferences: Isobaric interferences associated with naturally occurring isotopes are automatically corrected by the instrument software.

4.1.2. Isobaric molecular interferences: Corrections for molecular interferences will be applied where appropriate based on known or suspected interferences.

4.1.2.1. See Appendix B for a list of common interferences and the isotopes affected.

Note: Not all possible interferences are listed.

4.1.3. Matrix interferences: Internal standards will be used to correct for some matrix interferences.

4.1.4. A representative sample(s) of the project or projects being analyzed will be screened to avoid using an analyte as an internal standard that is present in the samples at a level greater than 10% of the level in the internal standard.

4.1.5. Internal standards should be added at a level to give approximately 100,000 - 500,000 counts of raw signal intensity. The mass of the internal standard used should ideally be within ±50 amu of the mass of the affected analyte.

4.1.6. Severe matrix effects will be monitored by comparing the internal standard intensity in the sample to the internal standard intensity of the initial calibration blank (ICB). If the internal standard intensity is less than 30% or greater than 120% of the internal standard intensity of the ICB, a fivefold dilution will be performed on the sample to correct for severe matrix effects, and the sample reanalyzed.

5. SAFETY

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

5.2. Eye protection that satisfied ANSI Z87.1 (as per the Chemical Hygiene Plan), laboratory coat, and chemically resistant gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been

contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.2.1. Neoprene, natural rubber, nitrile, and TRIonic® Cleanroom gloves provide varying degrees of protection against those chemicals listed. Refer to permeation/degradation charts for the actual data.

5.3. The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the Material Safety Data Sheets (MSDS) maintained in the laboratory.

5.3.1. The following materials are known to be **corrosive:**

sulfuric acid; hydrochloric acid; nitric acid; hydrofluoric acid (NOTE: sulfuric and hydrofluoric acids are used in cleaning the ICP torch and hydrofluoric acid is also commonly used in air toxics preparations.)

5.3.2. The following materials are known to be **oxidizing agents**:

nitric acid; hydrogen peroxide.

5.3.3. The plasma emits strong UV light and is harmful to vision. AVOID looking directly at the plasma.

5.3.4. The RF generator produces strong radio frequency waves, most of which are unshielded. People with pacemakers should not go near the instrument while in operation.

5.3.5. Refer to Perkin Elmer/Sciex ICP-MS Operator's Manual and the SOP LM-CAL-2000 ICP-MS Operation and Maintenance for information on instrument safety and precautions.

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

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

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

5.7. The use of hydrofluoric acid requires special safety precautions. Consult the facility EH&S Manager and laboratory supervisor for guidance.

NOTE: Contact may not be painful at first. The fluoride ion rapidly penetrates the skin and may cause delayed effects: lasting ulcerations, bone degeneration, pulmonary edema, muscle paralysis, and cardiac arrest. Any suspected exposure to HF liquid/fumes must be immediately evaluated by appropriate medical staff (i.e. U.C. Davis Medical Group Emergency Room.) Review Appendix G for a detailed first aid plan before work with HF begins.

6. EQUIPMENT AND SUPPLIES

6.1. Perkin Elmer/Sciex ELAN 5000 ICP-MS

6.2. Argon gas: High-purity grade (99.99%)

7. REAGENTS AND STANDARDS

7.1. Reagent grade water from a Milli-Q, Barnstead, or equivalent polishing system reading 18 Mohm or higher.

7.2. Calibration Standards: Prepare single or multi-element calibration standards as appropriate for the analysis.

7.3. ICV Stock Solution: Prepare calibration verification standards from a source other than that used for the calibration.

7.4. Calibration Check Standard (CCV): The CCV is a mutlielement solution and can be the either the ICV or one of the calibration standards.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Samples are to be collected in plastic or glass containers.

8.2. All soils must be refrigerated to 4°C ± 2°C.

8.3. The analytical holding time for metals is 6 months by ICP-MS.

8.4. Aqueous samples for total metals must be digested before analysis using an appropriate digestion procedure. The procedure in SOP LM-CAL-2060 for furnace analysis is the preferred digestion procedure. The ICP digestate may also be used, but this may increase the effect of interferences due to the presence of hydrochloric acid. SW846 Preparation Methods 3005, 3010, and 3020 and 3051 may also be used when appropriate. Upon consultation with the client very clean samples can forego digestion to help prevent contamintion when very low detection limits are required.

8.5. Soil or waste samples should be digested before analysis using an appropriate digestion procedure. The furnace digestion in SOP LM-CAL-2060 is preferred and generally gives acceptable results. SW846 Method 3050 and 3051 may also be used.

9. QUALITY CONTROL

9.1. Initial Demonstration of Capability

Prior to analysis of any analyte using Method 6020, the following requirements must be met.

9.1.1. Instrument Detection Limit (IDL) - The IDL for each analyte must be determined for each analyte wavelength used on each instrument. The IDL must be determined quarterly for the standard analytes listed in Appendix A. All other elements will be determined annually. If the instrument is adjusted in any way that may affect the IDL, the IDL for that instrument must be redetermined.

9.1.1.1. IDLs shall be determined by multiplying by three the average of the standard deviations obtained on three non-consecutive days from the analysis of a blank solution with seven consecutive measurements per day.

9.1.1.2. Each measurement must be performed as though it were a separate analytical sample.

9.1.1.3. Each measurement must be followed by a rinse and/or any other procedure normally performed between the analysis of separate samples.

9.1.1.4. The IDL measurement must consist of the same number of replicates used for analytical samples with the average result used for reporting.

9.1.2. Method Detection Limit (MDL) - An MDL must be determined for each analyte/matrix prior to the analysis of any samples. The MDL is determined using seven replicates of reagent water, spiked with all the analytes of interest, that have been carried through the entire analytical procedure. MDL's must be redetermined on an annual basis in accordance with 40 CFR Part 136 Appendix B requirements as detailed in Quanterra QA Policy QA-005. The spike level should be between the calculated MDL and 10X the MDL to be valid. At a minimum, the result of the MDL determination must be below the Quanterra reporting limit.

9.1.3. Linear Range Verification (LR) -The linear range is determined semi-annually (2x/year) for each element on the standard analyte list. Linearity is usually in the range of 0 to 1 million counts. For elements not on the standard analyte list, linearity will be from 0 to 1 million counts.

9.1.3.1. The linear range for routinely reported elements must be verified quarterly. Standards must be run at increasing concentration until elements are no longer within 10% of true value. The last concentration where the element was within 10 % of true value is considered the upper linear range. For those elements not analyzed routinely the upper linear range can be considered to be the concentration at 1,000,000 counts.

9.1.4. Interelement Corrections (IEC)-The ICSA and ICSAB solutions must be run daily. There are no published ICP-MS acceptance criteria for either the ICSA or the ICSAB. Analyses of these samples are used to assess the validity of the elemental equations used for the analysis.

9.2. Method Blank (MB) - One method blank must be processed with each preparation batch. The method blank consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The method blank is used to identify any system and

process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. The method blank should not contain any analyte of interest at or above the reporting limit (exception: common laboratory contaminants, see below) or at or above 5% of the measured concentration of that analyte in associated samples, whichever is higher (sample result must be a minimum of 20x higher than the blank contamination level).

- If the analyte is a common laboratory contaminant (copper, iron, or zinc) the data may be reported with qualifiers if the concentration of the analyte in the method blank is less than two times the RL. **Such action must be taken in consultation with the client and must be addressed in the project narrative.**

- Repreparation and reanalysis of all samples associated with an unacceptable method blank is required when reportable concentrations are determined in the samples (see exception noted above).

- If there is no analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. **Such action must be taken in consultation with the client and must be addressed in the project narrative.**

- If the above criteria are not met and reanalysis is not possible, then the sample data must be qualified. **This anomaly must be addressed in the project narrative and the client must be notified.**

- For dissolved metals samples which have not been digested or matrix matched, a CCB result is reported as the method blank. The CCB run immediately prior to the start of the dissolved sample analyses must be used for this purpose. No more than 20 samples can be associated with one CCB.

9.3. Laboratory Control Sample (LCS) - One aqueous LCS must be processed with each preparation batch. The LCS must contain all analytes of interest and must be carried through the entire analytical procedure. The LCS is used to monitor the accuracy of the analytical process. On-going monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines.

- If any analyte is outside established control limits the system is out of control and corrective action must occur. Until in-house control limits are established, a control limit of 80 - 120% recovery must be applied.

- In the event that an MS/MSD analysis is not possible a Laboratory Control Sample Duplicate (LCSD) must be analyzed. The RPD of the LCS and LCSD must be compared to the matrix spike RPD limits.

- In the instance where the LCS recovery is greater than 120% and the sample results are < RL, the data may be reported with qualifiers. **Such action must be taken in consultation with the client and must be addressed in the case narrative.**

- Corrective action will be repreparation and reanalysis of the batch unless the client agrees that other corrective action is acceptable.

- For dissolved metals samples which have not been digested or matrix matched, a CCV result is reported as the LCS. The CCV run immediately prior to the start of the dissolved sample analyses must be used for this purpose. No more than 20 samples can be associated with one CCV.

9.4. Matrix Spike/Matrix Spike Duplicate (MS/MSD) - One MS/MSD pair must be processed for each preparation batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Some client specific data quality objectives (DQO's) may require the use of sample duplicates in place of or in addition to MS/MSD's. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, these results may have immediate bearing only on the specific sample spiked. Samples identified as field blanks cannot be used for MS/MSD analysis.

- If any analyte recovery or RPD falls outside the acceptance range, the recovery of that analyte must be in control for the LCS. Control limits of 80 - 120 % recovery and 20% RPD (± RL if concentration is <10x the RL) must be applied to the MS/MSD. If the LCS recovery is within limits, then the laboratory operation is in control and the results may be accepted. If the recovery of the LCS is outside limits corrective action must be taken. Corrective action will include repreparation and reanalysis of the batch. MS/MSD results which fall outside the control limits must be addressed in the narrative.

- If the native analyte concentration in the MS/MSD exceeds 4x the spike level for that analyte, the recovery data are reported as NC (i.e., not calculated). If the

reporting software does not have the ability to report NC then the actual recovery must be reported and narrated as follows: "Results outside of limits do not necessarily reflect poor method performance in the matrix due to high analyte concentrations in the sample relative to the spike level."

- If an MS/MSD is not possible due to limited sample volume then a laboratory control sample duplicate (LCSD) should be analyzed. The RPD of the LCS and LCSD must be compared to the matrix spike RPD limits.

- For dissolved metals samples which have not been digested or matrix matched, a MS/MSD must be performed per batch of up to 20 samples by spiking two aliquots of the sample.

9.5. Initial Calibration Verification (ICV/ICB) - Calibration accuracy is verified by analyzing a second source standard (ICV). The ICV must fall within 10% of the true value for that solution. An ICB is analyzed immediately following the ICV to monitor low level accuracy and system cleanliness. The ICB result must fall within +/- the reporting limit (RL) from zero. If either the ICV or ICB fail to meet criteria, the analysis should be terminated, the problem corrected, the instrument recalibrated and the calibration reverified.

9.6. Continuing Calibration Verification (CCV/CCB) - Calibration accuracy is monitored throughout the analytical run through the analysis of a known standard after every 10 samples. The CCV must fall within 10% of the true value for that solution. A CCB is analyzed immediately following each CCV. The CCB result must fall within +/- RL from zero. Sample results may only be reported when bracketed by valid ICV/CCV and ICB/CCB pairs. If a mid-run CCV or CCB fails, the CCV or CCB may be reanalyzed once to and accepted if there is a reason for the initial out-of-control event such as carryover from a high concentration sample. Otherwise, if the CCV or CCB fails, the analysis for the affected element must be terminated, the problem corrected, the instrument recalibrated, the calibration verified and the affected samples reanalyzed. (Refer to Section 11.9 for an illustration of the appropriate rerun sequence).

9.7. The calibration curve must have a correlation coefficient of 0.995 or greater (assuming that more than 2 points are used to generate the linear curve).

9.8. The internal standard intensities must be 30% < [IS intensities for the ICB] < 120%. If this criteria is not met, the sample will be diluted and reanalyzed until the IS recoveries are within the limits. If the upper control limit is exceeded, the analyst

should review the data for the presence of the out-of-control internal standard in the native sample. Narrate any findings.

9.8.1. The internal standard intensity in the ICV and ICB should be within 20% of the IS intensity in the instrument standardization solution. If not, the analyst should check for any instrument anomalies and continue if none are noted.

9.9. Analytical matrix spikes on samples after preparation may be done at the discretion of a senior ICP-MS operator or Technical Manager to further assess the effect of the matrix on the results or as an indication of the performance of the interference equations used in the sample matrix.

9.10. Quality Assurance Summaries - Certain clients may require specific project or program QC which may supersede the SOP requirements. Quality Assurance Summaries (QAS) should be developed to address these requirements.

10. CALIBRATION AND STANDARDIZATION

10.1. Instrument start-up

10.1.1. Follow the instrument start-up procedure outlined in the ICP-MS OPERATION AND MAINTENANCE SOP LP-CAL-2000).

10.2. Instrument Tuning

10.2.1. Follow the instrument tuning procedure outlined in the ICP-MS OPERATION AND MAINTENANCE SOP (LP-CAL-2000).

10.2.2. Mass calibration and resolution checks must be documented and included as part of the raw data package.

10.2.2.1. Resolution must be < 1.0 amu at 10% peak height.

10.2.2.2. Mass calibration must be within ± 0.1 amu from the actual value or the mass calibration must be adjusted.

10.3. Standardization

10.3.1. From the APPLICATIONS menu load the QUANTITATIVE ANALYSIS application.

10.3.2. Load the appropriate parameter set if one already exists or create a new one for the analytes to be quantitated in the run. Solicit the assistance of a senior ICP-MS operator in creating a new parameter file.

10.3.3. See Appendix B for recommended isotopes and interference equations for commonly analyzed elements.

10.3.4. If no recommended isotopes are given for the element to be analyzed, consult a senior ICP-MS operator or appropriate reference (see Section 13.2).

10.3.5. See Appendix C or commonly used internal standards.

10.3.6. All masses which could affect data quality should be monitored to determine potential interferences either simultaneously during an analytical run or in a separate scan.

10.3.7. Open a new data set for a new analytical run. Title the data set with an appropriate title such as the date.

10.3.8. In order to simplify data archival, data set names should be limited to eight characters if possible.

10.3.9. Under the data set description enter the statement "ANALYST: (your initials)" and any other necessary information.

10.3.10. Two standards are routinely used for instrument calibration using a simple linear calibration curve.

- Low standard - Reagent water matching the sample digestate and standards, i.e. 1% HNO_3/0.5% HCl for water samples.
- High standard - the level should be appropriate for the analysis being performed.

10.3.11. If the use of more than two standards is desired, either the weighted linear or blank subtracted calibration types should be selected. The use of a simple

linear curve with more than two standards may cause a positive y-intercept leading to artificially elevated blank levels.

10.3.12.The first line in the data set should be used for the Low Standard. The standard name must be the same as what the standard concentration file was named. The Low Standard must be classified as a "standard" (as opposed to a "blank" which would signal the software to perform blank subtraction, which is not routinely used). The concentrations entered in the concentration file should be zero for all elements. Based on this standardization technique a Simple Linear curve is the most appropriate.

10.3.13.The second line should be used for the high standard. Again, the standard name must match the standard file name. The appropriate concentrations should be entered in the standard concentration file under the same name and saved.

10.3.14.Add internal standard to all standards and samples either by spiking or pumping. Both methods are acceptable.

10.3.14.1.If the pumping method is used, an additional line for the pump and a mixing tee are required. The internal standard line must be placed in the internal standard solution before any analyses are started.

10.3.15.A read delay of 45 seconds (60 seconds or longer if using the autosampler) should be entered in the configuration portion of the software which will allow the sample to travel through the sample uptake tubing and equilibrate through the plasma into the vacuum chamber before the read process is initiated. This allows all standards and samples to be processed in an identical manner.

10.3.16.Insert the sampler tube into the blank solution and press **PF12** (READ) to begin the analysis. After the "Measurements Complete" message appears at the top left-hand corner of the screen remove the sample uptake tube and return it to the rinse solution.

10.3.17.After the standard data has been collected and processed the data will automatically be stored and printed.

10.3.18.Repeat step 10.3.16 for the high standard. When complete the instrument is

standardized

11. PROCEDURE

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

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

11.3. An instrument or team backlog is generated from the LIMS data system. This backlog lists the Quanterra (WSAC) project numbers that were assigned to each batch or QC lot of samples (a batch or QC lot consists of a maximum of 20 customer samples). The backlog also shows project due date, number of samples and sample matrix.

11.4. An analytical run will consist of all customer samples and quality control samples analyzed under a single standardization. Each new standardization will begin a new analytical run.

11.5. A new ANALYSIS RUN LOG will be filled out for each analytical run. An alternative is to print out the "Sample Entry Report" from the Elan computer and include that.

11.6. Type in the QC and sample information into the sample ID column. You can change the default dilution factor (1) and enter any additional sample information (such as QC lots) by pressing **SHIFT F5** and filling out the detailed sample information.

11.7. In order to use the ICP-MS data upload program into LIMS, the following naming conventions must be followed:

11.7.1. Samples are identified by project number-sample number and aliquot. For example, Sample 1 from project 28977 is identified as: 28977-01.

11.7.2. Matrix spikes, duplicates, and matrix spike duplicates are identified as 28977-

01MS, 28977-01DU, and 28977-01SD, respectively.

11.7.3. Prep Blanks are identified by BLANK.

11.7.4. LCSs are identified as LCS, LCS DUP.

11.8. The order of analysis for the initial QC samples should be:

- ICV
- ICB
- Elemental Equation verification or interference check solution (if required).
- Prep Blank
- LCS
- LCSDUP (if required).
- Samples

11.9. Analysis sequence when out-of-control run QC is observed:

- CCV
- CCB
- Restandardize (see 10.3.16 through 10.3.18)
- CCV
- CCB
- Samples

12. DATA ANALYSIS AND CALCULATIONS

12.1. All pertinent calculations are performed by the ELAN software.

12.2. Reporting Requirements

12.2.1. Units equal mg\L for aqueous samples and mg\kg for soil samples.

12.2.2. If dilutions were required due to insufficient sample interferences, or other problems, the reporting limits are multiplied by the dilution factor.

12.2.3. For results less than 10 mg\L, two significant figures will be reported. For results greater than or equal to 10 mg\L, three significant figures will be reported. Refer to Quanterra Policy QA-0004 for additional information on significant figures and rounding.

12.2.4. Document any non-standard procedures or anomalies by using the anomaly program in LIMS.

12.3. Data Package Requirements

12.3.1. A complete data package consists of: the initial masscal, resolution, and sensitivity checks; run log, copy of parameter file used, level 1 checklist, and all raw data.

12.3.2. Level I review will be completed by the analyst.

12.3.3. Level II review will be completed by a senior level laboratory analyst familiar with the technical aspects of ICP-MS and in accordance with the ICP-MS DATA REVIEW checklists. The instrument operator of an analytical run may not perform the Level II review for that run.

13. METHOD PERFORMANCE

13.1. Each laboratory must have initial demonstration of performance data on file for each analyte of interest as described in Section 9.0.

13.2. Refer to Appendix A for the list of analytes using this SOP.

13.3. Method performance is determined by the analysis of matrix spike and matrix spike duplicate samples as well as method blanks and laboratory control samples. The matrix spike recovery should fall within ± 20% and the matrix spike duplicates should

compare within ± 20% RPD (± RL if concentration is < 10x RL). Method blanks must meet the criteria specified in Section 9.2. The laboratory control samples should recover within 20% of the true value until in-house control limits are established.

13.4. Training Qualification

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

14. POLLUTION PREVENTION

14.1. Standards or solutions are not approved for disposal to the sink.

15. WASTE MANAGEMENT

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

15.2. Standards should be purchased and prepared in volumes consistent with laboratory use to minimize the volume of expired standards to be disposed.

15.3. All waste must be disposed of according to the facility hazardous waste managment procedures, Attachment C of the Quanterra Inc. Chemical Hygiene Plan, Section WS002, Table 1.

15.4. Samples and other solutions containing high concentrations of toxic materials must be disposed of according to the facility hazardous waste management procedures, Attachment C of the Quanterra Inc. Chemical Hygiene Plan, Section WS003, Disposal of Samples After Analysis.

16. REFERENCES

16.1. Source Method: EPA Method 6020 CLP-M, Version 8.

16.2. Quanterra Inc., Quality Assurance Management Plan, Revision 1.0, May 1996.

16.3. Quanterra Corporate Policy QA-003 QC Program

16.4. LS-CAL-2002, Standards Preparation-Metals.

16.5. Perkin Elmer/Sciex ELAN model 5000 Users Manual

16.6. SW846 Method 6020, Revision 0 (3rd Update, September 1994).

16.7. EPA Method 200.8 EMSL office of Research & Development, Cincinnati, OH (Draft Method, Revision 4.3, August 1990).

16.8. Recommended ICP-MS References

16.8.1. K. E. Jarvis, A. L. Gray, and R. S. Honk, Handbook of Inductively Coupled Plasma Mass Spectrometry; Chapman & Hall, New York 1992.

16.8.2. A. R. Date and A. L. Gray, Applications of Inductively Coupled Mass Spectrometry, Chapman & Hall, New York 1989. ISBN # 0-41201721-0

17. MISCELLANEOUS (TABLES, APPENDICES, ETC...)

17.1. Deviations From Source Method and Rationale

17.1.1. As a conservation of space, the raw data will only contain the mean and standard deviation results on standard ELAN summary report. As long as standard deviation data is shown the analyst can make decisions on replicate quality on standard ELAN summary report.

17.1.2. The ICP-MS is not required to be restandardized every 8 hours as long as all continuing calibration criteria continue to be met.

17.1.3. The continuing calibration standards are not required to be reanalyzed within 2 hours as long as they continue to meet acceptability criteria.

17.1.4. Each CCV and CCB may be reanalyzed once to verify the instrument is not out of calibration providing that the next sample has not yet been analyzed and the analyst has an indication that the CCV or CCB result could have been affected by air bubbles, sample carryover, or other problems.

17.1.5. A serial dilution is not performed. The internal standard is used to monitor matrix interference.

17.1.6. This SOP may be used to analyze for elements not included in Method 6020, as long as appropriate QC samples are run with acceptable results.

17.1.7. For reporting consistency, an ICB/CCB is acceptable if the result is <RL.

17.2. Modifications from Previous SOP

17.2.1. Format was updated to be consistent with Quanterra Inc. QA-001.

17.2.2. ICSA/ICSAB analysis frequency was increased to "daily." Section 9.1.4.

17.2.3. Linear range studies are conducted semiannually instead of quarterly. Section 9.1.3.

17.2.4. Control limits and corrective actions for the MBLK, LCS and MS/MSD were updated to reflect Quanterra Inc. QA Policy QA-003. Sections 9.2, 9.3, 9.4.

17.2.5. Safety sections were updated to reflect the Quanterra Inc. Chemical Hygiene Plan. Sections 5.0, 14.0, 15.0.

17.2.6. Acceptance criteria for the internal standard was updated. Section 9.8.

APPENDICES

Appendix A	Standard Analyte List and Reporting Limits
Appendix B	Common Molecular Ion Interferences-Background and Matrix
Appendix C	Recommended Isotopes and Additional Masses Which May be Monitored
Appendix D	Elemental Equations for Data Calculations
Appendix E	Internal Standards and Limitations for Use
Appendix F	Forms and Documentation
Appendix G	Hydrofluoric Acid First Aid Plan
Appendix H	Flow Diagram

Appendix A

Standard Analyte List and Reporting Limits

Note: These are the routine reporting limits for most sample types. Lower reporting limits may be achievable for special projects. Difficult sample matrices may cuase reporting limits to be raised.

Element	Symbol	CAS #	Aqueous Report Limit, mg/L	Soil Reporting Limit, mg/Kg
Aluminum	Al	7429-90-5	0.05	5.0
Antimony	Sb	7440-36-0	0.002	0.2
Arsenic	As	7440-38-2	0.002	0.2
Barium	Ba	7440-39-3	0.001	0.1
Beryllium	Be	7440-41-7	0.001	0.1
Cadmium	Cd	7440-43-9	0.001	0.1
Chromium	Cr	7440-47-3	0.001	0.1
Cobalt	Co	7440-48-4	0.001	0.1
Copper	Cu	7440-50-8	0.001	0.1
Iron	Fe	7439-89-6	0.05	5.0
Lead	Pb	7439-92-1	0.001	0.1
Magnesium	Mg	7439-95-4	0.01	1.0
Manganese	Mn	7439-96-5	0.001	0.1
Molybdenum	Mo	7439-98-7	0.001	0.1
Nickel	Ni	7440-02-0	0.001	0.1

Element	Symbol	CAS #	Aqueous Report Limit, mg/L	Soil Reporting Limit, mg/Kg
Potassium	K	7440-09-7	0.05	5.0
Selenium	Se	7782-49-2	0.002	0.2
Silver	Ag	7440-22-4	0.001	0.1
Sodium	Na	7440-23-5	0.05	5.0
Tin	Sn	7440-31-5	0.002	0.2
Titanium	Ti	7440-03-26	0.001	0.1
Thallium	Tl	7440-28-0	0.001	0.1
Vanadium	V	7440-62-2	0.005	0.5
Zinc	Zn	7440-66-6	0.002	0.2

APPENDIX B (FROM 13.2.6, METHOD 200.8)

COMMON MOLECULAR ION INTERFERENCES IN ICP-MS

BACKGROUND MOLECULAR IONS

Molecular Ion	Mass	Element Interferences*
NH^+	15	
OH^+	17	
OH_2^+	18	
C_2^+	24	
CN^+	26	
CO^+	28	
N_2^+	28	
N_2H^+	29	
NO^+	30	
NOH^+	31	
O_2^+	32	
O_2H_+	33	
$^{36}ArH^+$	37	
$^{38}ArH^+$	39	

C4-208

Molecular Ion	Mass	Element Interferences*
$^{40}ArH^+$	41	
CO_2^+	44	
CO_2H^+	45	Sc
ArC^+, ArO^+	52	Cr
ArN^+	54	Cr
$ArNH^+$	55	Mn
ArO^+	56	
$ArOH^+$	57	
$^{40}Ar^{36}Ar^+$	76	Se
$^{40}Ar^{38}Ar^+$	78	Se
$^{40}Ar_2^+$	80	Se

* Method elements or internal standards affected by the molecular ions.

MATRIX MOLECULAR IONS

CHLORIDE

Molecular Ion	Mass	Element Interference
$^{35}ClO^+$	51	V
$^{35}ClOH^+$	52	Cr
$^{37}ClO^+$	53	Cr
$^{37}ClOH^+$	54	Cr
$Ar^{35}Cl^+$	75	As
$Ar^{37}Cl^+$	77	Se

SULFATE

Molecular Ion	Mass	Element Interference
$^{32}SO^+$	48	
$^{32}SOH^+$	49	
$^{34}SO^+$	50	V, Cr
$^{34}SOH^+$	51	V
SO_2^+, S_2^+	64	Zn
$Ar^{32}S^+$	72	
$Ar^{34}S^+$	74	

PHOSPHATE

Molecular Ion	Mass	Element Interference
PO^+	47	
POH^+	48	
PO_2^+	63	Cu
ArP^+	71	

GROUP I, II METALS

Molecular Ion	Mass	Element Interference
$ArNa^+$	63	Cu
ArK^+	79	
$ArCa^+$	80	

MATRIX OXIDES*

Molecular Ion	Mass	Element Interference
TiO	62-66	Ni, Cu, Zn
ZrO	106-112	Ag, Cd
MoO	108-116	Cd

* Oxide interferences will normally be very small and will only impact the method elements when present at relatively high concentrations. Some examples of matrix oxides are listed of which the analyst should be aware. It is recommended that Ti and Zr isotopes are monitored in solid waste samples, which are likely to contain high levels of these elements. Mo is monitored as a method analyte.

RECOMMENDED ANALYTICAL ISOTOPES AND ADDITIONAL MASSES WHICH MAY BE MONITORED *

Isotope	Element of Interest
27	Aluminum (1)
121,123	Antimony (1)
75	Arsenic (1)
138,137,136,135,134,132,130	Barium (1)
9	Beryllium (1)
114,112,111,110,113,116,106,108	Cadmium (1)
42,43,44,46,48	Calcium (1)
52,53,50,54	Chromium (1)
59	Cobalt (1)
63,65	Copper (1)
56,54,57,58	Iron (1)
206,207,208	Lead (1)
24,25,26	Magnesium (1)
55	Manganese (1)
202,200,199,201	Mercury
98,96,92,97,94	Molybdenum

Isotope	Element of Interest
58,60,62,61,64	Nickel (1)
39	Potassium (1)
80,78,82,76,77,74	Selenium
107,109	Silver (1)
23	Sodium (1)
203, 205	Thallium (1)
51,50	Vanadium (1)
66, 67, 68	Zinc (1)
83	Krypton
72	Germanium
139	Lanthanum
140	Cerium
129	Xenon
118	Tin
105	Palladium
47,49	Titanium
125	Tellurium
69	Gallium
35,37	Chlorine

*FROM Table 9 of Method 6020 CLP-M)

(1) Element approved for ICP-MS determination by SW846 Method 6020 CLP-M

NOTE: Isotopes recommended for analytical determination are underlined.

APPENDIX D

RECOMMENDED ELEMENTAL EQUATIONS FOR DATA CALCULATIONS

Element	Elemental Equation	Note
Al	(1.000) (^{27}C)	
Sb	(1.000) (^{121}C)	
As	(1.000) (^{75}C) - (3.1278)[^{77}C) - (1.0177)(^{78}C)]	(1)
Ba	(1.000) (^{137}C)	
Be	(1.000) (^{9}C)	
Cd	(1.000) (^{111}C) - (1.073) [(^{108}C) - (0.712) (^{106}C)]	(2)
Cr	(1.000) (^{52}C)	(3)
Co	(1.000) (^{59}C)	
Cu	(1.000) (^{63}C)	
Pb	(1.000) (^{206}C) + (1.000) (^{207}C) + (1.000) (^{208}C)	(4)
Mn	(1.000) (^{55}C)	
Mo	(1.000) (^{98}C) - (0.146) (^{99}C)	(5)
Ni	(1.000) (^{60}C)	

C4-216

Element	Elemental Equation	Note
Se	(1.000) (^{82}C)	(6)
Ag	(1.000) (^{107}C)	
Tl	(1.000) (^{205}C)	
Th	(1.000) (^{232}C)	
U	(1.000) (^{238}C)	
V	(1.000) (^{51}C) - (3.127) [(^{53}C) - (0.113) (^{52}C)]	(7)
Zn	(1.000) (^{66}C	

* Method elements or internal standards affected by the molecular ions.

INTERNAL STANDARDS

Element	Elemental Equation	Note
Bi	(1.000) (^{209}C)	
In	(1.000) (^{115}C) - (0.0149) (^{118}C)	(8)
Sc	(1.000) (^{45}C)	
Tb	(1.000) (^{159}C)	
Y	(1.000) (^{89}C)	

C Calibration blank subtracted counts at specified mass.

(1) Correction for chloride interference with adjustment for Se77. ArCl 75/77 ratio may be determined from the reagent blank.

(2) Correction of MoO interference. An additional isobaric elemental correction should be made if palladium is present.

(3) In 0.4% v/v HCl, the background from ClOH will normally be small. However the contribution may be estimated from the reagent blank.

(4) Allowance for isotopic variability of lead isotopes.

(5) Isobaric elemental correction for ruthenium.

(6) Some argon supplies contain krypton as an impurity. Selenium is corrected for Kr82 by background subtraction.

(7) Correction of chloride inference with adjustment for Cr53. Cl0 51/53 ration may be determined from the reagent blank.

(8) Isobaric elemental correction for tin.

APPENDIX E

INTERNAL STANDARDS AND LIMITATIONS OF USE

Internal Standard	Mass	Possible Limitation
^{6}Lithium	6	a
Scandium	45	Polyatomic Ion Interference
Germanium	72	
Yttrium	89	a, b
Rhodium	103	
Indium	115	Isobaric Interference by Sn
Terbium	159	
Holmium	165	
Lutetium	175	
Bismuth	209	a

a May be present in environmental samples.

b In some instruments Yttrium may form measurable amounts of YO^+ (105 amu) and YOH^+ (106 amu). If this is the case, care should be taken in the use of the cadmium elemental correction equation.

APPENDIX F

FORMS AND DOCUMENTATION

QUANTERRA INC.
West Sacramento

SOP#_____ ICP-MS ANALYSIS RUN LOG METHOD_____

ANALYST: FILENAME: DATE:

NO.	SAMPLE I.D.	D.F.	NO.	SAMPLE I.D.	D.F.	NO.	SAMPLE I.D.	D.F.
1			26			51		
2			27			52		
3			28			53		
4			29			54		
5			30			55		
6			31			56		
7			32			57		
8			33			58		
9			34			59		
10			35			60		
11			36			61		
12			37			62		
13			38			63		
14			39			64		
15			40			65		
16			41			66		
17			42			67		
18			43			68		
19			44			69		
20			45			70		
21			46			71		
22			47			72		
23			48			73		
24			49			74		

C4-221

25			50			75		

Standard Verifications: Cal Stock: # ICV/DCS Stock #:

Deviations from SOP:_____

_____Approved:_____

QUANTERRA INC. (West Sacramento)

Anomalies

TEST	SAMPLE(s)	ANOMALIES

1. Prep blank contamination for _____.
2. MDL's used to meet required reporting limits.
3. QC out of limits for non-required elements.
4. Furnace results taken from ICP data.
5. Sample(s) is/are hazardous due to (*characteristic*) for (*element*).
6. Sample(s) diluted due to matrix interference, detection limit raised.
7. Detection limit raised due to matrix interference (no dilution required).
8. Total and dissolved analysis not consistent.
9. Other (explain): _____

_____

_____

_____

Comments: _____

_____

_____

_____

Approved:_____ Date:_____

QUANTERRA INC. (West Sacramento)

ICP-MS DATA REVIEW CHECKLIST

LEVEL I

FILE NO.	PROJECT NOS.	ANALYST:	DATE:	YES	NO	NA

	YES	NO	NA
1. Copy of analysis protocol used included?			
2. ICVs & CCVs within 10% of true value?			
3. ICB & CCBs < reporting limit?			
4. 10 samples or less analyzed between calibration checks?			
5. All parameters within linear range. No results reported from intensity reading with a > sign?			
6. LCS/DCS within limits?			
7. Prep blank value < reporting limit or all samples >10x blank?			
8. Internal standard intensities for samples (unless followed by dilution) are > 30% and < 120% of the ICB intensities?			
9. Appropriate dilution factors applied to data?			
10. Matrix spike and spike dup within customer defined limits			
11. Each batch checked for presence of internal standard in samples?			
12. Anomalies entered using LIMS anomaly program.			

COMMENTS: _____

REVIEWED BY:	DATA ENTERED BY:
DATE:	DATE:

QAQC/BENCHSHEETS/ICPMS

QUANTERRA INC. (West Sacramento)

PREVIEW DATA REVIEW

LEVEL II

FILE NO.	PROJECT NOS.	ANALYST:	DATE:	YES	NO	NA
1. Prep and analysis anomalies acceptable?						
2. Bench sheets complete?						
3. Anomalies noted on Camera Ready Report Cover Sheet?						
4. Reanalysis data included?						
5. Error corrections made in appropriate manner?						
6. QC checked and acceptable?						
7. PJ report checked for transcription errors?						
8. Reporting limits correct?						
9. Appropriate flags entered?						
10. Analysis date reflects date of accepted data?						
11. Corrections entered and verified?						
12. Data released?						

COMMENTS: _____

_____

_____

LEVEL II REVIEWED BY:	CORRECTIONS ENTERED BY:
DATE:	DATE:

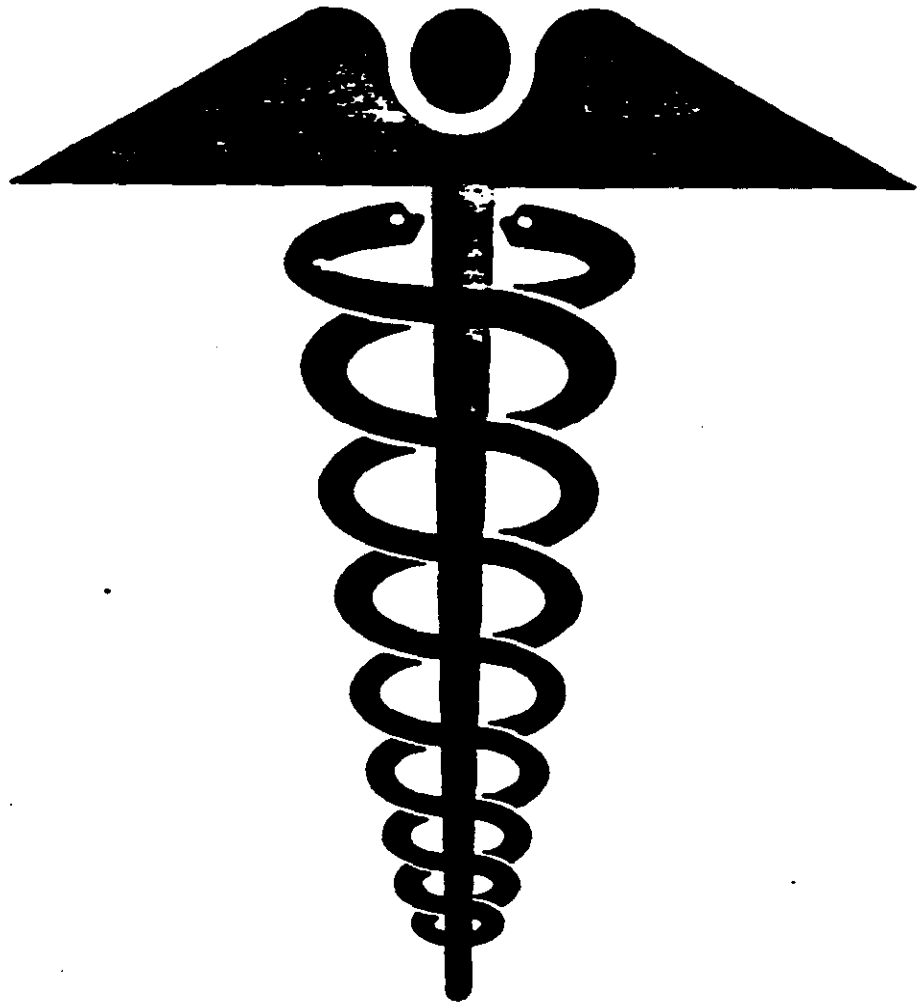
APPENDIX G

HYDROFLUORIC ACID FIRST AID PLAN

[redacted]

~~Appendix I~~ 1-18-96

Recommended Medical Treatment for Hydrofluoric Acid Exposure



C4-229

The HF Products Group

Allied Signal

The HF Products Group

Allied Signal

For more information including technical information on any aspect of hydrofluoric acid write: Allied-Signal Inc., Hydrofluoric Acid Products Group, 101 Columbia Road, P.O. Box 1053, Morristown, NJ 07962-1053.

In Canada, write: Allied-Signal Canada Inc., Manager, Hydrofluoric Acid Products Group, 201 City Centre Drive, Suite 205, Mississauga, Ontario L5B 2T4.

To place an order, obtain prices or availability information from any location in the continental United States, call toll free 800-522-8001.

Your HF Resource

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C4-230

Hydrofluoric acid is a very strong inorganic acid. Both anhydrous hydrofluoric acid (hydrogen fluoride) and its solutions are clear, colorless liquids. When exposed to air, concentrated solutions and anhydrous hydrofluoric acid produce pungent fumes which are especially dangerous. Unless heated, dilute concentrations of hydrofluoric acid in water do not produce vapors.

NOTE: Persons unfamiliar with hydrofluoric acid often mistake it or confuse it with hydrochloric acid. Although hydrofluoric acid (HF) and hydrochloric acid (HCl) sound similar, the toxicity of these two acids is very different. To decrease or avoid confusion, we recommend that *HYDROFLUORIC ACID*, and *HYDROGEN FLUORIDE* be referred to as "HF".

HF is primarily an industrial raw material. It is used in stainless steel manufacturing, iron and steel foundries, metal finishing, aluminum manufacturing, inorganic and organic chemical manufacturing, petroleum refining, mineral processing, glassmaking and electronic components manufacturing. It is also used in certain industrial and consumer cleaning compounds. However, its use in consumer products is discouraged because of the hazards described herein.

Most non-industrial burns are caused by dilute concentrations of HF. Most of the HF used in the electronics industry is less than 50% concentration. However, many industrial uses of HF involve concentrated (50-100%) HF.

WARNING: BURNS WITH CONCENTRATED HF ARE USUALLY VERY SERIOUS, WITH THE POTENTIAL FOR SIGNIFICANT COMPLICATIONS DUE TO FLUORIDE TOXICITY. CONCENTRATED HF, LIQUID OR VAPOR, MAY CAUSE SEVERE BURNS, METABOLIC IMBALANCES, PULMONARY EDEMA AND LIFE THREATENING CARDIAC ARRYTHMIAS. EVEN *MODERATE* EXPOSURES TO CONCENTRATED HF MAY RAPIDLY PROGRESS TO A FATALITY IF LEFT UNTREATED.

Allied-Signal is the world's leading supplier of hydrofluoric acid. The recommended medical procedures described below are based on many years experience in dealing with the unique hazards of this product, in addition to a review of the medical literature. Every effort must be made to prevent exposure to hydrofluoric acid. If exposure does occur, the specialized procedures which follow are recommended to avoid the very serious consequences that might otherwise occur.

Because the medical treatment of hydrofluoric acid exposure is so specialized and differs from the treatment of other inorganic acid exposures, not all physicians may be aware of appropriate treatment measures. It is recommended that HF users make arrangements ahead of time with local medical resources to be sure that users are familiar with first aid measures and that professional personnel are familiar with the toxicity of HF and the treatment of HF posure. This would include, at a minimum, thoroughly reviewing this booklet and making sure that treatment facilities and supplies are available.

Hydrofluoric acid (HF) can cause serious, painful burns of the skin. Specialized first aid and medical treatment is required. Burns larger than 25 square inches (160 square cm) may result in serious systemic toxicity.

Hydrofluoric acid is a highly corrosive acid which can severely burn skin, eyes, and mucous membranes. The *vapors* from anhydrous hydrofluoric acid or its concentrated solutions can also burn these tissues.

Hydrofluoric acid is similar to other acids in that the initial extent of a burn depends on the concentration, the temperature and the duration of contact with the acid. Hydrofluoric acid differs, however, from other acids because the fluoride ion readily penetrates the skin, causing destruction of deep tissue layers including bone. Unlike other acids which are rapidly neutralized, this process may continue for days.

Strong acid concentrations (over 50%), and particularly anhydrous HF (AHF or 100% HF), usually cause immediate, severe, burning pain and a whitish discoloration of the skin which usually proceeds to blister formation. Exposure to HF vapors can also result in similar burns.

In contrast to the immediate effects of concentrated HF, the effects of contact with more dilute hydrofluoric acid or its vapors may be delayed, and this is one of the problems with the recognition of some HF burns. Contact with acid concentrations in

tracheostomy. It is recommended that all patients with such exposures be hospitalized for observation.

Because of the strong irritant nature of hydrofluoric acid, an individual inhaling HF vapors or fumes will usually experience upper respiratory injury, with mucous membrane irritation and inflammation as well as cough. All individuals suspected of inhalation should be observed for pulmonary effects. This would include those individuals with significant upper respiratory irritation, broncho-constriction by pulmonary auscultation or spirometry, and any individual with HF exposure to the head, chest or neck areas. It has been reported that pulmonary edema may be delayed for several hours and even up to two days. If there is no initial upper respiratory irritation, significant inhalation exposure can generally be ruled out.

The Permissible Exposure Limit (PEL) set by the U.S. Occupational Safety and Health Administration (OSHA) is a ceiling level of 3 ppm and a 15 minute short term exposure limit (STEL) of 6 ppm (24). The National Institute for Occupational Safety and Health (NIOSH) has established a level that is immediately dangerous to life and health (IDLH) at 30 ppm (25). The American Industrial Hygiene Association has published Emergency Response Planning Guidelines setting 50 ppm as the maximum level below which nearly all individuals could be exposed for one hour without experiencing or developing life-threatening health effects (ERPG-3), 20 ppm as the maximum level below which nearly all individuals could be exposed for one hour without developing irreversible health effects or symptoms which would impair taking protective action (ERPG-2), and 5 ppm as the maximum level below which nearly all individuals could be exposed up to one hour without experiencing other than mild, transient adverse health effects (ERPG-1) (26).

If hydrofluoric acid is ingested, severe burns to the mouth, esophagus and stomach may occur. Severe systemic effects usually also occur. Ingestion of even small amounts of dilute HF have resulted in death (27).

Chronic toxicity from overexposure to fluoride ion has been reported to result in tooth mottling in children, bone fluorosis and sometimes osteosclerosis in adults and children.

Skeletal fluorosis is known to be associated with excessive exposure to fluoride compounds. Cases of skeletal fluorosis have been reported in populations exposed to naturally occurring drinking water containing greater than 10 ppm of fluoride ion and in individuals exposed to high levels of fluoride containing dusts. However, skeletal fluorosis has not been reported as a consequence of hydrofluoric acid exposure.

Fluorides are not carcinogenic, and have not been reported to cause male or female reproductive effects. Fluoride exposures should be kept below recommended levels to assure no adverse effects to the developing fetal skeletal system.

Monitoring of urine for fluorides is an accepted method of determining exposure (28). Urine fluoride levels above 3 mg/liter at the beginning of a workshift, or above 10 mg/liter at the end of a workshift, may indicate excessive absorption of fluoride. It should be noted that fluorides are often present in significant amounts in persons not occupationally exposed (because of dietary sources of fluoride such as tea), and that the urine fluoride determination is not specific for HF (29, 30).

In Case of Contact or Suspected Contact with Hydrofluoric Acid:

1. Move victim immediately under safety shower or other water source and flush affected area thoroughly with large amounts of cool running water. Speed in washing off the acid is of primary importance.
2. Remove all contaminated clothing while flushing with water.
3. Rinse with large amounts of cool running water. If 0.13% ZEPHIRAN® solution or 2.5% calcium gluconate gel are available, the rinsing may be limited to 5 minutes, with the soaks or gel applied as soon as the rinsing is stopped. If ZEPHIRAN® or calcium gluconate gel are not available, rinsing must continue until medical treatment is rendered.
4. While the victim is being rinsed with water, someone should alert first aid or medical personnel and arrange for subsequent treatment.

3. If a physician is not immediately available, apply one or two drops of 0.5% PONTOCAINE® hydrochloride solution or other aqueous, topical ophthalmic anesthetic and continue irrigation. Use no other medications unless instructed to do so by a physician. Rubbing of the eyes is to be avoided.

1. Immediately move victim to fresh air and get medical attention.
2. Keep victim warm, quiet and comfortable.
3. If breathing has stopped, start artificial respiration at once. Make sure mouth and throat are free of foreign material.
4. Oxygen should be administered as soon as possible by a trained individual. Continue oxygen while awaiting medical attention unless instructed otherwise by a physician.
5. A nebulized solution of 2.5% calcium gluconate may be administered with oxygen by inhalation.
6. Do not give stimulants unless instructed to do so by a physician.
7. The victim should be examined by a physician and held under observation for at least a 24 hour period.
8. Vapor exposures can cause skin mucous membrane burns as well damage to pulmonary tissue. Vapor burns to the skin are treated the same as liquid HF burns.

1. Have the victim drink large amounts of water as quickly as possible to dilute the acid. Do not induce vomiting. Do not give emetics or baking soda. Never give anything by mouth to an unconscious person.
2. Give several glasses of milk or several ounces of milk of magnesia, MAALOX®, MYLANTA® etc. The calcium or magnesium in these compounds may act as an antidote.
3. Get immediate medical attention.

Burns from dilute acid are difficult to distinguish from other chemical burns and usually appear as areas of erythema. However, they may progress, if not treated, to areas of blistering, necrosis or ulceration. Burns from more concentrated acid have a rather characteristic appearance and present as severely reddened, swollen areas with blanched, whitish regions which rapidly progress to blistering and necrosis. A thick granular exudate usually appears under these blisters which requires debridement and removal.

Hydrofluoric acid burns cause extreme pain. The pain is thought to result from nerve ending irritation due to increased levels of potassium ions in extracellular spaces to compensate for the reduced levels of calcium ions which have been bound by the fluoride. Relief of pain is an excellent indication of the success of treatment and, therefore, local anesthetics should be avoided.

Many different types of therapies have been suggested for HF burns. The aim of all treatment is to chemically sequester the fluoride ion and to prevent extensive, deep-tissue destruction (31).

After treatment of burned areas is begun, the victim should be carefully examined to insure there are no other burned areas which may have been overlooked.

Most HF burns can be satisfactorily treated by immersion of the burned part in an iced, aqueous (or aqueous-alcohol) solution of a quaternary ammonium compound. Two solutions have been clinically successful, 0.13% ZEPHIRAN® (benzalkonium chloride) or 0.2% HYAMINE® 1622 (benzethonium chloride). *Because of its availability as a non-prescription drug, ZEPHIRAN® is recommended in the United States.*

The solutions should be cooled with ice cubes. (Shaved or crushed ice may cause excessive cooling, with the danger of frostbite.)

If immersion in the solution is not practical, soaked compresses of the same iced solution should be applied to the burned area. The immersion or compresses should be used for at least two hours. Compresses should be changed or soaked with additional solutions approximately every two to four minutes.

If blisters are present, they should be opened and drained and necrotic tissue should be debrided prior to use of ZEPHIRAN® immersion or compresses.

Prolonged immersion in the iced ZEPHIRAN® bath may result in discomfort due to excess chilling; relief may be obtained by removing the part from the bath every ten to fifteen minutes for a few minutes and then reimmersing it. After the initial 30-60 minutes of treatment, less ice can be used so the bath is cool rather than cold.

treatment to be effective. One author has cautioned that removal of the nail should rarely be necessary in the case of dilute HF acid (less than 10%) burns (32). The treating physician must consider the morbidity associated with removal of the nail versus the need to treat the HF exposure.

If immersion in ZEPHIRAN® solution is started immediately, it may be possible to avoid removing the nail. Sometimes better penetration under the nail can be successfully accomplished by splitting the nail or by drilling several burr holes in the nail using a large gauge needle or a nail drill. If calcium gluconate injection is used as treatment, the nail may still need to be split or removed. Some authorities recommend the use of general anesthesia or a regional nerve block, rather than local anesthesia, to remove the nail so that pain relief by calcium gluconate injections may be used as an indicator of effective treatment. When using calcium gluconate injections in the digits, care must be taken to inject the solution slowly so as to avoid any compromise to the circulation in these areas.

Reports in the literature have described the use of intra-arterial injection or infusion of dilute calcium gluconate solutions to treat HF burns of the hand and digits which do not respond to other methods, either due to inadequate or improper treatment, or in cases where treatment has been greatly delayed. The method is described as follows:

> "A long catheter was inserted percutaneously into the radial artery using standard aseptic technique. Intra-arterial catheter placement was confirmed by pressure transducer and oscilloscope. If the burn involved only the thumb, index, or long fingers, the catheter was advanced only a few centimeters proximally in preparation for digital subtraction arteriography. If the burn involved the ring or small fingers, the catheter was advanced proximally into the brachial artery because access to the ulnar circulation was necessary.
>
> Following satisfactory placement of the arterial catheter, we performed digital subtraction arteriography on all patients in our series to identify the origin of vascular supply to digits involved.
>
> Once the tip of the arterial catheter was in the desired location, a dilute preparation of calcium [gluconate] (10 ml of a 10% solution mixed in 40 to 50 ml 5% dextrose) was infused with a pump apparatus into the catheter over four hours. We generally have used calcium gluconate.... Each patient was observed closely during the infusion period for progression of symptoms and potential complications of the procedure, such as alterations of distal vascular supply.
>
> Following the four-hour infusion, the arterial catheter was maintained in place in the usual manner while the patient underwent an observation period. If typical HF pain returned within four hours, a second calcium infusion was repeated until the patient was pain free four hours following completion of the calcium infusion. (13)"

This method, although rather involved, should be considered in selected cases, especially where inadequate or delayed treatment has occurred.

Where blistering and/or necrosis occur, early debridement may facilitate healing.

In instances of extensive burns, skin grafting has occasionally been required, but the need for this treatment should be markedly reduced by immediate and aggressive primary treatment.

Follow-up care requires monitoring to prevent secondary infections, and the use of antibiotic creams such as SILVADENE® or GARAMYCIN® has proven effective. HF burns may heal slowly, but if properly treated most heal with little or no scarring in 14 to 28 days.

Significant amounts of fluoride ion may be absorbed by skin contact, inhalation, or by ingestion. If systemic absorption of fluoride occurs, hypocalcemia, hypomagnesemia and hyperkalemia may also occur. All of these parameters need to be monitored and appropriate therapeutic measures instituted. The patient should be observed for clinical signs of hypocalcemia following ingestion or inhalation or following extensive burns greater than 25 square inches. Serum calcium determinations must be performed immediately and periodically to monitor and treat hypocalcemia. Severe lowering of serum calcium levels can occur within one to two hours even with HF burns covering less than 2.5% of body surface area (7). Continuous EKG monitoring to observe prolongation of the Q-T interval may be useful to detect early changes in serum calcium, although profound hypocalcemia following HF exposure has been reported in the absence of EKG changes.

The fall in serum calcium may occur precipitously following HF exposure. In two reported cases of exposure to anhydrous HF, the serum calcium fell to

Pulmonary function testing may be helpful in assessing the degree and progress of pulmonary injury.

Specific measures may be needed to treat pulmonary edema. High doses of parenteral steroids may be needed along with the administration of appropriate diuretics. Caution should be taken not to administer excessive fluid. Hemoconcentration may require treatment by phlebotomy. The management of pulmonary edema may result in renal failure due to reduced fluid volume, and this may be another indication for renal dialysis.

If it is necessary to relieve anxiety, use general measures and do not use sedatives which could cause central nervous system depression or hypoventilation. Although right heart failure is uncommon in chemically-induced pulmonary edema, monitoring of pulmonary pressure, arterial pressure, and central venous pressure may be indicated.

Secondary infections must be treated. It is preferable to start antibiotics at the first signs of infection such as fever or tachycardia. Periodic blood cultures may be advisable. Prophylactic use of antibiotics is not advised.

Ingestion Injuries

After first aid is completed (drinking several glasses of water followed by two glasses of milk or two ounces of milk of magnesia, MYLANTA®, or other calcium or magnesium containing antacids), the stomach may be lavaged with lime water. The Levin tube must be passed with care to prevent perforation. Treatment is the same as for ingestion of other strong acids. Systemic toxicity is very likely to occur and may require aggressive treatment.

C4-235

drug. Care should be taken that HYAMINE® 1622 is used, not HYAMINE® with other numeric or alphanumeric modifiers.

CALCIUM GLUCONATE GEL is not available commercially in the United States as a pharmaceutical agent. It is hoped that a commercial preparation of calcium gluconate gel will be approved by the U.S. Food and Drug Administration, and become available in the U.S. as it now is in Canada, Great Britain, and other countries. At the present time in the U.S., however, calcium gluconate gel requires a prescription either for the compounded gel or for the 10% solution. The shelf life is uncertain, but it is recommended that calcium gluconate gel be replaced on an annual basis.

CALCIUM GLUCONATE INJECTION, USP (one gram in 10 ml, 10% solution) is labelled for intravenous use only. Experience has shown that when diluted to 5% with normal saline, and used as described in this booklet, it is a safe and effective treatment for HF skin exposure. When diluted to 2.5% and used as described, it is safe for nebulization and inhalation, and when diluted to 1.0% and used as described, it is safe for eye irrigation.

GARAMYCIN® is a Registered Trademark of Schering Corporation, Kenilworth, NJ 07033

HYAMINE® 1622 is a Registered Trademark of Lonza, Inc., Fairlawn, NJ 07410

K-Y® Jelly is a Registered Trademark of Johnson & Johnson Products, Inc., New Brunswick, NJ 08903

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	Skin Burns		Eye Exposure	Inhalation		Ingestion
	Conc. HF	Dilute HF	All HF	Conc. HF	Dilute HF	All HF
First Aid	Water Wash THEN ZEPHIRAN® [A] 0.13% soaks OR Calcium Gluconate 2.5% Gel	Water Wash THEN ZEPHIRAN® [A] 0.13% soaks OR Calcium Gluconate 2.5% Gel	Water Wash THEN 1% Calcium Gluconate Irrigation	Oxygen AND 2.5% Calcium Gluconate by Nebulizer	Oxygen AND 2.5% Calcium Gluconate by Nebulizer	Do Not Induce Vomiting Milk or Water THEN Milk of Magnesia or MYLANTA® [B]
Medical Treatment	Debride THEN Continue Soaks or Calcium Gluconate 2.5% Gel OR Calcium Gluconate 5% Injection[2] AND Observe For/And Treat Systemic Effects[3] (especially if > 25sq. in.)	Debride THEN Continue Soaks or Calcium Gluconate 2.5% Gel OR Calcium Gluconate 5% Injection[2] Systemic Effects[3] Unlikely	Topical Tetracaine Hydrochloride THEN 1% Calcium Gluconate Irrigation AND Consult Ophthalmologist	Observe AND Treat Bronchoconstriction, Pulmonary Edema and Systemic Effects[3]	Observe (Serious Effects Unlikely)	Lavage with Limewater AND Treat Systemic Effects[3]

1) This is a brief summary of First Aid and Treatment measures. The text of the booklet "RECOMMENDED MEDICAL TREATMENT FOR HYDROFLUORIC ACID EXPOSURE" must be consulted for more complete information.

2) 5% Calcium gluconate injections must be used if the gel does not significantly relieve pain in 30-60 minutes. Injections may also be used as the primary treatment, especially for larger and/or deeper burns.

3) Systemic effects include hypocalcemia, hypomagnesemia, hyperkalemia, cardiac arrhythmias, and altered pulmonary hemodynamics. TREATMENT includes cardiac monitoring, monitoring serum calcium, magnesium, and electrolytes; administration of intravenous calcium gluconate, correcting magnesium and electrolyte imbalance, and dialysis.

NOTE: In absence of the usual medical history, the physician will find it helpful to obtain the following information: concentration of Hydrofluoric Acid, date and time of exposure, distance of exposure, how exposure occurred, body parts exposed/affected, first aid measures initiated (what, when, how long), figures that relate Hydrofluoric Acid solutions or fume concentrations of vapors may result in delays in clinical presentation up to 24 hours following exposure.

[A] Registered Trademark, Winthrop Laboratories, New York, NY 10016

[B] Registered Trademark, Stuart Pharmaceuticals, Wilmington, DE 19899

For additional reference charts or information on properties, storage and handling, or medical treatment for hydrofluoric acid, contact:

The HF Products Group
Allied-Signal Inc.
P.O. Box 1053
Morristown, NJ 07962-1053

In the event of an emergency with this product, call the 24-hour Allied-Signal Emergency telephone number: (201) 455-2000.

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24.237

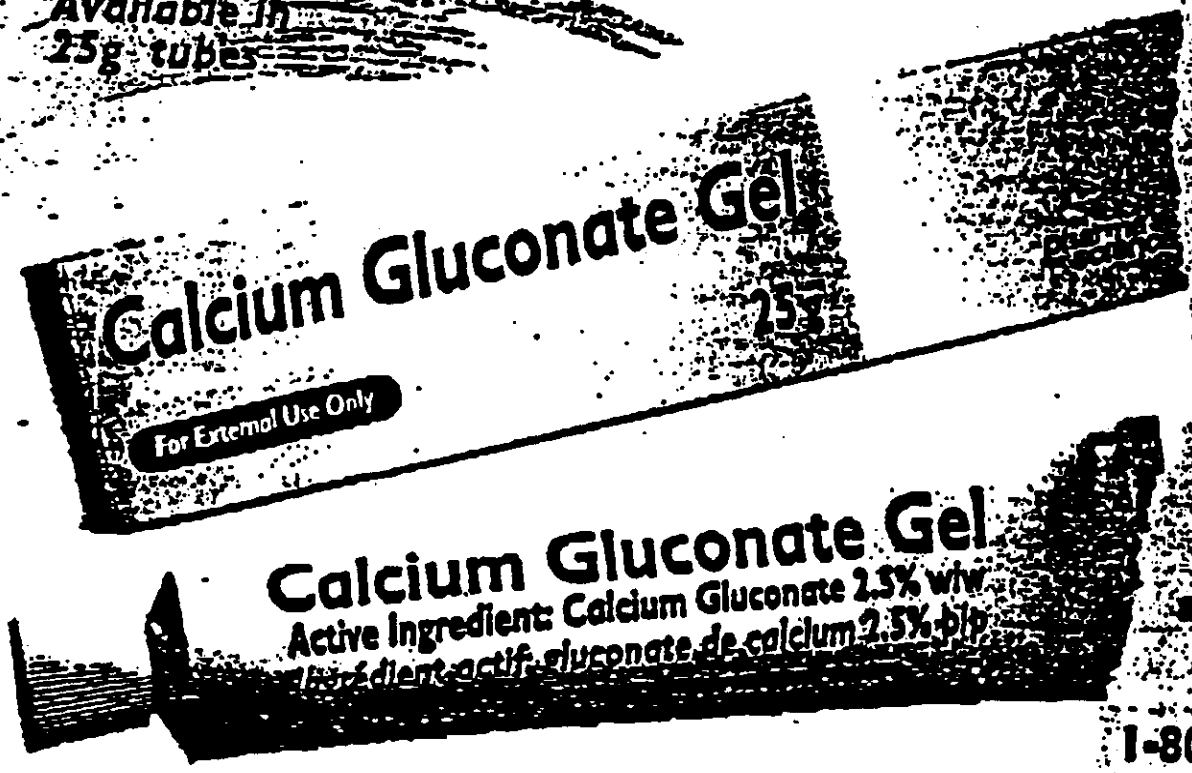
Calcium Gluconate 2.5%

Topical Gel

"Topical Calcium Gluconate is an efficacious treatment for dermal hydrofluoric acid burns."

(Am. J. Emerg. Med., Jan. '94, Vol. 24, No. 1, pg. 9)

Available in 25g tubes



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1-800-363-8805

Pharmascience Inc., 8400 Darnley Road, Montreal, Canada, H4T 1M4
Tel.: (514) 340-9735 Fax.: (514) 340-9290

C4-238

Coping with HF

Reports on the Internet mailing list SAFETY tell of the extreme hazards associated with hydrofluoric acid. They also provide some valuable insights into dealing with HF hazards.

A cautionary story appeared in *The West Australian* last year. Exposure to hydrofluoric acid killed an employee of a small laboratory in Western Australia. The victim accidentally splashed about 100 mL of a 70% solution on his leg. Though the leg was unscarred, the acid had poisoned the man's blood and he died agonizingly. According to a health expert on the case, the lab worker would have had little chance of surviving an HF spill as big as the one that killed him even if every precautionary measure had been taken. The acid has to come in contact with only 2% of the body to be fatal.

HF exposure causes profound hypocalcemia, a lowering of blood calcium. HF burns are deep and excruciatingly painful (though burns from exposure to dilute solutions of less than 25% may not be felt for more than an hour). They can be treated through intravenous administration of calcium gluconate solutions. Immediate attention in the laboratory should entail flushing with water followed by soaking in 0.13% Zephiran solution or topical application of calcium gluconate gel which ties up the fluoride ion and thus reduces its damaging effect. Lab personnel should not use HF without Zephiran (Winthrop Laboratories) or calcium gluconate gel being readily available. Calcium gluconate gel can be prepared by mixing calcium gluconate with a water-soluble lubricant such as Johnson & Johnson's K-Y Jelly. Tubes of prepared gel are available commercially; one supplier is Pharmascience, Inc., in Montreal).

Labs that use HF also should inform local medical facilities and make sure that they have in place a protocol and materials for dealing with patients exposed to HF. For more information on first aid and medical treatment of HF exposures, request a copy of AlliedSignal's publication "Recommended Medical Treatment for Hydrofluoric Acid Exposure" by calling 800/522-8001. This booklet is a must for labs using HF! MSDSs for HF-containing products may provide first aid information as well.

Labs with HF on hand should also be prepared for a spill. Silicate-based absorbents (such as kitty litter) are not useful, because they can react with HF to form SiF_4, a toxic and corrosive gas. Instead, stock a supply of gypsum ($CaSO_4$) to absorb HF spills.

Check around to see if HF is employed in your lab. Also, check old chemical supplies and discard HF solutions that are not in use. Finally, review the MSDSs for such lab supplies as commercial cleaners; HF may turn up as an ingredient.

Personnel using HF should understand the hazards associated with exposure and know the steps to take in the event exposure occurs. The OSHA Lab Standard (29 CFR 1910.1450) mandates that such a hazardous chemical be mentioned in a lab's chemical hygiene plan and discussed in the required annual training.

LSEM has a new editorial office. See staff box on page 2 for new mail and E-mail addresses. Direct comments, inquiries and contributions there, and subscription requests to Burbank.

Waste Management Guide for Clinical Labs

The National Committee for Clinical Laboratory Standards (NCCLS) offers a collection of documents on issues in lab safety and environmental management. One of these, "Clinical Laboratory Waste Management; Approved Guideline" (Document GP5-A, December 1993), provides much important information on waste management in less than 100 pages. According to Gerald A. Hoskyn, MD, of the Cleveland [Ohio] Clinic Foundation, who chairs the NCCLS Subcommittee on Laboratory Waste, the guideline will be revised periodically to keep it current.

NCCLS describes the document thus: "GP5-A, Clinical Laboratory Waste Management; Approved Guideline, was written for use by laboratory managers and is intended to provide approaches to controlling laboratory-generated hazardous and nonhazardous waste. A brief summary of the relevant federal regulations and laws is included. The types of waste addressed include chemical, infectious, radioactive, sharps, and nonhazardous. Options for handling, packing, labeling, storing, treating, transporting, and disposal of each type of waste are also described."

To learn more about the lab waste subcommittee, contact Dr. Hoskyn at 216/444-2830. For a copy of the guideline or list of other relevant publications, write NCCLS at 771 E. Lancaster Ave., Villanova, PA 19085.

Free NSC Training

The National Safety Council is offering a two-day course covering health and safety standards applicable to small-business owners. The training is funded through a grant received by OSHA and, therefore, is free. Topics include ergonomics, personal protective equipment, recordkeeping, incident reporting and program development. While not lab-specific, the course still can benefit lab personnel. The training is given each month through February in Itasca, IL. To learn more, or to register, call NSC at 800/621-7615, ext. 2356.

FR REVIEW

An overview of relevant notices and proposed and final rules published in the Federal Register (60 FR) August 31–October 24, 1995.

Aug. 31: EPA proposed streamlined procedures for revising stationary source operating permits issued under Title V of the CAAA (45530). Contact Michael Trutna, EPA, 919/541-5435. See story on page 1.

Sept. 1: EPA finalized its NESHAP for aerospace manufacturing and rework facilities (45948). Contact Vickie Bowles, EPA, 919/541-0164. See story on page 2.

Sept. 5: EPA proposed to delete diethyl phthalate from the list of chemicals subject to reporting requirements under Section 313 of the Emergency Planning and Community Right-to-Know Act (46076). Contact the EPCRA Hotline, 800/535-0202.

Sept. 5: EPA rescinded Subpart I of 40 CFR 61 (radionuclide air emissions limits) as it applies to nuclear power reactors (46206). Contact Fran Jenner, EPA, 202/233-9300.

Sept. 7: EPA issued a rule limiting regulatory obligations of lending institutions or other persons holding a security interest in property containing an underground storage tank (46692). Contact the RCRA Hotline, 800/424-9346.

Sept. 8: NRC proposed a one-time 5-year extension of certain by-product, source, and special nuclear materials licenses (46784). Contact John Pelchat, NRC Region II, 404/331-5083.

Sept. 20: EPA terminated the reporting requirements for 234 chemicals under the TSCA Section 8(d) model health and safety data reporting rule (48660). Contact the TSCA Hotline, 202/554-0551.

Sept. 20: DOT's Research and Special Programs Administration (RSPA) revised its regulations pertaining to infectious substances, including regulated medical waste (48780). Contact Jennifer Antonetti, RSPA, 202/366-8553.

Sept. 27: EPA announced that revisions to North Carolina's hazardous waste program satisfy requirements necessary to qualify for final authorization under RCRA (49800). Contact Al Hanke, EPA Region 4, 404/347-3234.

Sept. 28: EPA proposed rescinding 40 CFR 61, Subpart I (radionuclide NESHAP), as it applies to NRC and Agreement State licensees other than nuclear power reactors (50161). It believes rescission is acceptable since NRC has committed to propose a rule to constrain air emissions from such licensees to a level that would result in a dose of no

Continued on page 7

C4-239

QUANTERRA INC. (West Sacramento)

STANDARD OPERATING PROCEDURE CHANGE FORM

SOP Number:	LM-CAL-2041
SOP Title:	Analysis of Metals by Inductively Coupled Plasma/Mass Spectrometry
SOP Sections Affected by Change:	9.3.2
Reason for Addition or Change:	Limits were updated to reflect current method requirements.
Change Effective From [Date]:	
Change or Addition (Specific Section; use additional sheets if necessary.):	9.3.2 Average DCS recovery must be within ± 20% of the expected value.

Submitted by/Date: John Barnett

Approved by:*

Technical Reviewer: *Date:*

EH&S Signature: *Date:*

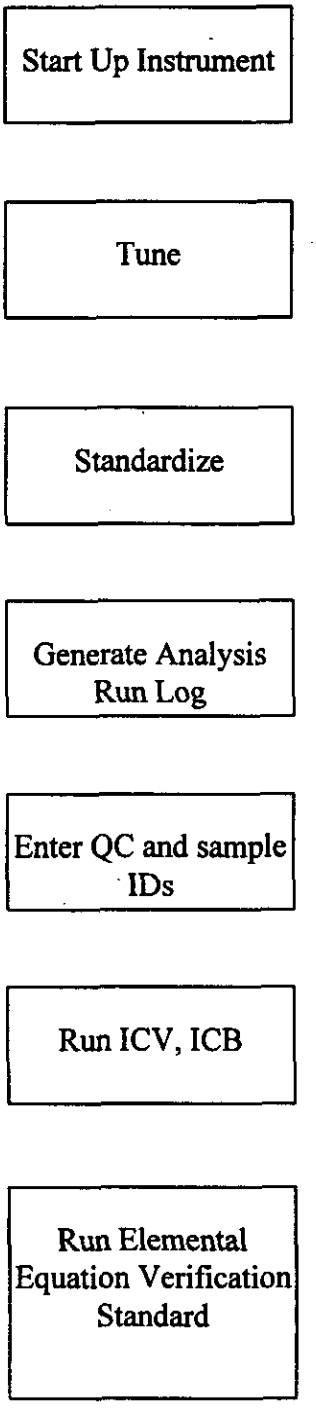
QA Signature: *Date:*

Management Signature: *Date*

Must be same signature authorities of SOP being revised.
d:\\qsacca01\sacqa\sop\amend.dot

C4-240

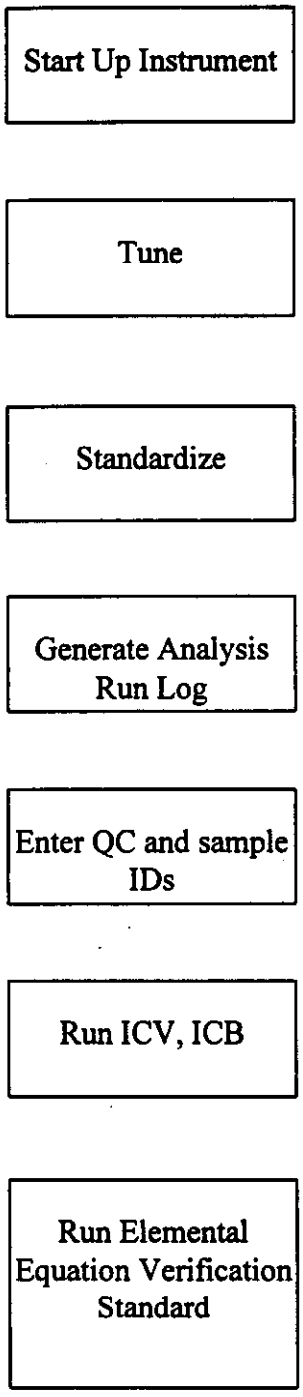
Flow Diagram



Run batch QC and samples.

Run CCV, CCB

Flow Diagram



Run batch QC and samples.

Run CCV, CCB

Quanterra Environmental Services

QUANTERRA ENVIRONMENTAL SERVICES (West Sacramento)
SOP CHANGE FORM

SOP Number:	
SOP Title:	
SOP Sections Affected by Change:	
Reason for Addition or Change:	
Change Effective From [Date]:	
Change or Addition (Specific Section; Use additional sheets if necessary):	

Submitted by/Date:_____

Approved by:*

Technical Reviewer:_____ Date_____

EH&S Signature:_____ Date_____

QA Signature:_____ Date_____

Management Signature:_____ Date_____

* Must be same signature authorities of SOP being revised.

C4-245

Controlled Copy
Copy No._____

QUANTERRA STANDARD OPERATING PROCEDURE

TITLE: PREPARATION AND ANALYSIS OF MERCURY IN SOLID SAMPLES BY COLD VAPOR ATOMIC ABSORPTION SPECTROSCOPY, SW846 7471A AND MCAWW 245.5

(SUPERSEDES: REVISION 0)

Prepared by: _____
Principal Scientist, Inorganics

Reviewed by: _____
Technology Standardization Committee

Approved by: _____
Director, Quality Assurance

Approved by: _____
Director, Environmental Health and Safety

Approved by: _____
Management

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C4-246

TABLE OF CONTENTS

1. SCOPE AND APPLICATION 4
2. SUMMARY OF METHOD 4
3. DEFINITIONS 4
4. INTERFERENCES 4
5. SAFETY 6
6. EQUIPMENT AND SUPPLIES 7
7. REAGENTS AND STANDARDS 8
8. SAMPLE COLLECTION, PRESERVATION AND STORAGE 10
9. QUALITY CONTROL 10
10. CALIBRATION AND STANDARDIZATION 14
11. PROCEDURE 15
12. DATA ANALYSIS AND CALCULATIONS 20
13. METHOD PERFORMANCE 22
14. POLLUTION PREVENTION 22
15. WASTE MANAGEMENT 22
16. REFERENCES 22
17. MISCELLANEOUS (TABLES, APPENDICES, ETC. . .) 23

LIST OF APPENDICES:

APPENDIX A - TABLES 27
APPENDIX B - QUANTERRA Hg DATA REVIEW CHECKLIST 31
APPENDIX C - MSA GUIDANCE 33
APPENDIX D - TROUBLESHOOTING GUIDE 36
APPENDIX E- CONTAMINATION CONTROL GUIDELINES 38
APPENDIX F - PREVENTATIVE MAINTENANCE 40

1. SCOPE AND APPLICATION

1.1. This procedure describes the preparation and analysis of mercury (Hg, CAS # 7439-97-6) by Cold Vapor Atomic Absorption Spectroscopy (CVAA) using SW-846 Method 7471A and MCAWW Method 245.5.

1.2. CVAA analysis provides for the determination of total mercury (organic and inorganic). The combination of the oxidants, potassium permanganate and potassium persulfate, has been found to give 100% recovery with both types of compounds. Detection limits, sensitivity and optimum concentration ranges for mercury analysis will vary with the matrices, instrumentation and volume of sample used.

1.3. Methods 7471A and 245.5 are applicable to the preparation and analysis of mercury in soils, sediments, bottom deposits and sludge-type materials. All matrices require sample preparation prior to analysis.

1.4. The Quanterra reporting limit for mercury in solid matrices is 0.1 mg/kg based a 0.2 g sample aliquot (wet weight).

2. SUMMARY OF METHOD

2.1. This SOP describes a technique for the determination of mercury in solution. The procedure is a physical method based on the absorption of radiation at 253.7 nm by mercury vapor. A representative portion of the sample is digested in sulfuric and nitric acids. Organic mercury compounds are oxidized with potassium permanganate and potassium persulfate and the mercury reduced to its elemental state with stannous chloride and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer. Absorbance is measured as a function of mercury concentration. Concentration of the analyte in the sample is determined by comparison of the sample absorbance to the calibration curve (absorbance vs. concentration).

3. DEFINITIONS

3.1. Total Metals: The concentration determined on an unfiltered sample following digestion.

4. INTERFERENCES

Chemical and physical interferences may be encountered when analyzing samples using this method.

4.1. Potassium permanganate which is used to breakdown organic mercury compounds also eliminates possible interferences from sulfide. Concentrations as high as 20 mg/L of sulfide as sodium sulfide do not interfere with the recovery of inorganic mercury from reagent water.

4.2. Copper has also been reported to interfere; however, copper concentrations as high as 10 mg/L had no effect on the recovery of mercury from spiked samples.

4.3. Chlorides can cause a positive interference. Samples high in chlorides require additional permanganate (as much as 25 mL) because, during the oxidation step, chlorides are converted to free chlorine, which also absorbs radiation at 253.7 nm. Care must be taken to ensure that free chlorine is absent before the mercury is reduced and swept into the cell. This is accomplished by adding excess hydroxylamine reagent (25 mL) and purging the sample headspace before stannous chloride is added. Both inorganic and organic mercury spikes have been quantitatively recovered from seawater using this technique.

Note: Sufficient addition of permanganate is apparent when the purple color persists at least 15 minutes. Some samples may require dilution prior to digestion due to extremely high concentrations of chloride.

4.4. Interference from certain volatile organic materials that absorb at this wavelength may also occur. If suspected, a preliminary run without stannous chloride can determine if this type of interference is present. While the possibility of absorption from certain organic substances present in the sample does exist, this problem is not routinely encountered. This is mentioned only to caution the analyst of the possibility. If this condition is found to exist, the mercury concentration in the sample can be determined by subtracting the result of the sample run without the reducing reagent (stannous chloride) from that obtained with the reducing reagent.

4.5. Samples containing high concentrations of oxidizable organic materials, as evidenced by high COD levels, may not be completely oxidized by this procedure. When this occurs the recovery of mercury will be low. The problem can be eliminated by reducing the volume of original sample used.

4.6. The most common interference is laboratory contamination which may arise from impure reagents, dirty glassware, improper sample transfers, dirty work areas, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them.

5. SAFETY

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

5.2. Eye protection that satisfies ANSI Z87.1 (as per the Chemical Hygiene Plan), laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.3. The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the Material Safety Data Sheets (MSDS) maintained in the laboratory. The following specific hazards are known:

5.3.1. The following materials are known to be **corrosive:**

hydrochloric acid, nitric acid and sulfuric acid.

5.3.2. The following materials are known to be **oxidizing agents**:

nitric acid, potassium permanganate, potassium persulfate and magnesium perchlorate.

5.3.3. Mercury is a highly toxic element that must be handled with care. The analyst must be aware of the handling and clean-up techniques before working with mercury. Since mercury vapor is toxic, precaution must be taken to avoid its inhalation, ingestion or absorption through skin. All lines should be checked for leakage and the mercury vapor must be vented into a hood or passed through a mercury absorbing media such as:

5.3.3.1. Equal volumes of 0.1 M $KMnO_4$ and 10% H_2SO_4, or

5.3.3.2. Iodine, 0.25%, in a 3% KI solution.

5.3.4. Magnesium sulfate is known to be a reproductive toxin (mutagen).

5.4. Exposure to chemicals must be maintained **as low as reasonably achievable.** Therefore, unless they are known to be non-hazardous, all samples should be opened, transferred and prepared in a fume hood, or under other means of mechanical

ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

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

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

5.7. Do not look directly into the beam of the Hg lamp. The UV light that these lamps radiate is harmful to the eyes.

5.8. Cylinders of compressed gas must be handled with caution, in accordance with local regulations. It is recommended that, wherever possible, cylinders be located outside the laboratory and the gas led to the instrument through approved lines.

5.9. The CVAA apparatus must be properly vented to remove potentially harmful fumes generated during sample analysis.

6. EQUIPMENT AND SUPPLIES

6.1. Temperature controlled water bath (capable of maintaining temperature of 90- 95 °C) or autoclave capable of obtaining 15 lbs., 120 °C.

6.2. Atomic Absorption Spectrophotometer equipped with:

6.2.1. Absorption Cell with quartz end windows perpendicular to the longitudinal axis. Dimensions of the cell must result in sufficient sensitivity to meet the SOP defined reporting limit. The quartz windows must be maintained to provide accurate measurements. Any scratches or fingerprints can alter the absorption of UV radiation.

6.2.2. Mercury specific hollow cathode lamp (HCL) or electrodeless discharge lamp (EDL).

6.2.3. Peristaltic pump which can deliver 1 L/min air.

6.2.4. Flowmeter capable of measuring an airflow of 1 L/min.

6.2.5. Recorder or Printer.

6.2.6. Aeration Tubing: A straight glass frit having a course porosity and Tygon tubing is used for the transfer of mercury vapor from the sample bottle to the absorption cell and return.

6.2.7. Drying device (a drying tube containing magnesium perchlorate or magnesium sulfate and/or a lamp with a 60 W bulb) to prevent condensation in cell. The lamp is positioned to shine on the absorption cell maintaining the air temperature in the cell about 10 °C above room temperature. Other drying devices that acheive the same purpose are also acceptable (i.e., Gortex filter).

Note: Instruments designed specifically for the measurement of mercury using the cold vapor technique may be substituted for the atomic absorption spectrophotometer.

6.3. BOD bottles or equivalent.

6.4. Nitrogen or argon gas supply, welding grade or equivalent.

6.5. Calibrated automatic pipettes or Class A glass volumetric pipettes.

6.6. Class A volumetric flasks.

6.7. Top-loading balance, capable of reading up to two decimal places.

6.8. Thermometer (capable of accurate readings at 95 °C).

6.9. Disposable cups or tubes.

7. REAGENTS AND STANDARDS

7.1. Reagent water must be produced by a Millipore DI system or equivalent. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

7.2. Stock (1000 ppm) mercury standards (in 10% HNO_3) are purchased as custom Quanterra solutions. All standards must be stored in FEP fluorocarbon or previously unused polyethylene or polypropylene bottles. Stock standard solutions must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the stock solutions may be used for up to one year and must be replaced sooner if verification from an independent source indicates a problem.

7.3. Intermediate mercury standard (10 ppm): Take 1 mL of the stock mercury standard (7.2) and dilute to 100 mL with reagent water. The intermediate standard must be made monthly and must be prepared in a matrix of 2% HNO_3. This acid (2 mL of concentrated HNO_3) must be added to the flask/bottle before the addition of the stock standard aliquot.

7.4. Working mercury standard (0.1 ppm): Take 1 mL of the intermediate mercury standard (7.3) and dilute to 100 mL with reagent water. The working mercury standard must be made daily and must be prepared in a matrix of 0.15% HNO_3. This acid (150 uL of concentrated HNO_3) must be added to the flask/bottle before the addition of the stock standard aliquot.

7.5. The calibration standards must be prepared fresh daily from the working standard (7.4) by transferring 0, 0.2, 0.5, 1.0, 2.0, 5.0 and 10.0 mL aliquots of the working mercury standard into sample prep bottles and proceeding as specified in Section 11.1

Note: Alternate approaches to standard preparation may be taken and alternate volumes of standard may be prepared as long as the accuracy and final standard concentrations as detailed in Table I are maintained. For example, automated mercury systems do not require 100 mL of standard and therefore smaller volumes may be generated to reduce waste generation.

7.6. The initial calibration verification standard must be made from a different stock solution than that of the calibration standards.

7.7. Refer to Table I (Appendix A) for details regarding the working standard concentrations for calibration, calibration verification and spiking solutions. All standards must be processed through the entire analytical procedure including sample preparation.

7.8. Nitric acid (HNO_3), concentrated, trace metal grade or better.

Note: If a high reagent blank is obtained, it may be necessary to distill the nitric acid.

7.9. Sulfuric acid (H_2SO_4), concentrated, trace metal grade or better.

7.9.1. Sulfuric acid, 0.5 N: Dilute 14.0 mL of concentrated H_2SO_4 to 1 liter with reagent water.

7.10. Hydrochloric acid (HCl), concentrated, trace metal grade or better.

7.11. Aqua Regia: Prepare immediately before use by carefully adding three volumes of concentrated HCl to one volume of concentrated HNO_3.

7.12. Stannous sulfate solution: Add 25 g of stannous sulfate to 250 mL of 0.5 N sulfuric acid. This mixture is a suspension and should appear cloudy. This solution should be made daily and should be stirred continuously during use.

Note: Stannous chloride may be used in place of stannous sulfate. Prepare the stannous chloride solution according to the recommendations provided by the instrument manufacturer.

7.13. Sodium chloride-hydroxylamine hydrochloride solution: Add 12 g of sodium chloride and 12 g of hydroxylamine hydrochloride to every 100 mL of reagent water.

Note: Hydroxylamine sulfate may be used in place of hydroxylamine hydrochloride.

7.14. Potassium permanganate, 5% solution (w/v): Dissolve 5 g of potassium permanganate for every 100 mL of reagent water.

7.15. Potassium persulfate, 5% solution (w/v): Dissolve 5 g of potassium persulfate for every 100 mL of reagent water.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Sample holding time for mercury is 28 days from time of collection to the time of sample analysis.

8.2. Soil samples do not require preservation but must be stored at 4° C ± 2° C until the time of analysis.

9. QUALITY CONTROL

Table II (Appendix A) provides a summary of quality control requirements including type, frequency, acceptance criteria and corrective action.

9.1. Initial Demonstration of Capability

Prior to the analysis of any analyte using 7471A or the 245.5, the following requirements must be met.

9.1.1. Method Detection Limit (MDL) - An MDL must be determined for each analyte/matrix prior to the analysis of any samples. The MDL is determined using seven replicates of reagent water, spiked with all the analytes of interest,

that have been carried through the entire analytical procedure. MDLs must be redetermined on an annual basis in accordance with 40 CFR Part 136 Appendix B requirements. The spike level must be between the calculated MDL and 10X the MDL to be valid. The result of the MDL determination must be below the Quanterra reporting limit.

9.1.2. Initial Demonstration Study - This requires the analysis of four QC check samples. The QC check sample is a well characterized laboratory generated sample used to monitor method performance. The results of the initial demonstration study must be acceptable before analysis of samples may begin.

9.1.2.1. Four aliquots of the check sample (LCS) are prepared and analyzed using the procedures detailed in this SOP and the determinative SOPs.

9.2. Preparation Batch - A group of up to 20 samples that are of the same matrix and are processed together using the same procedures and reagents. The preparation batch must contain a method blank, a LCS and a matrix spike/matrix spike duplicate. In some cases, at client request, it may be appropriate to process a matrix spike and sample duplicate in place of the MS/MSD. If clients specify specific samples for MS/MSD, the batch may contain multiple MS/MSD pairs.

9.3. Sample Count - Laboratory generated QC samples (method blanks, LCS) are not included in the sample count for determining the size of a preparation batch. MS/MSD are not included in the sample count unless there are multiple sets of MS/MSD per batch. In other words, the first MS/MSD are not counted; all additional MS and MSDs are counted as samples.

9.4. Method Blank (MB) - One method blank must be processed with each preparation batch. The method blank consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. The method blank should not contain any analyte of interest at or above the reporting or at or above 5% of the measured concentration of that analyte in associated samples, whichever is higher (sample result must be a minimum of 20 times higher than the blank contamination level).

- Repreparation and reanalysis of all samples associated with an unacceptable method blank is required when reportable concentrations are determined in the samples (see exception noted above).

- If there is no analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. **Such action must be taken in consultation with the client and must be addressed in the project narrative.**

- If the above criteria are not met and reanalysis is not possible, then the sample data must be qualified. **This anomaly must be addressed in the project narrative and the client must be notified.**

9.5. Laboratory Control Sample (LCS) - One LCS must be processed with each preparation batch. The LCS is used to monitor the accuracy of the analytical process. On-going monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines. The LCS must be carried through the entire analytical procedure. The CCV results can be reported as the LCS results since all CCVs (as well as all other standards) are processed through the sample preparation step with the field samples. No more than 20 samples can be associated with one CCV used for the purpose of reporting LCS data.

- If the LCS is outside established control limits the system is out of control and corrective action must occur. Until in-house control limits are established, a control limit of 80 - 120% recovery must be applied.

- In the instance where the LCS recovery is > 120% and the sample results are < RL, the data may be reported with qualifiers. Such action must be taken in consultation with the client and must be addressed in the case narrative."

- In the event that an MS/MSD analysis is not possible, a Laboratory Control Sample Duplicate (LCSD) must be analyzed. The RPD of the LCS and LCSD must be compared to the matrix spike RPD limits.

- Corrective action will be repreparation and reanalysis of the batch unless the client agrees that other corrective action is acceptable.

9.6. Matrix Spike/Matrix Spike Duplicate (MS/MSD) - One MS/MSD pair must be processed for each preparation batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Some client specific data quality objectives (DQO's) may require the use of sample duplicates in place of or in addition to MS/MSD's. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, these results may have immediate bearing

only on the specific sample spiked. Samples identified as field blanks cannot be used for MS/MSD analysis. Spiking levels are provided in Table I (Appendix A).

- If analyte recovery or RPD falls outside the acceptance range, the recovery of that analyte must be in control for the LCS. Until in-house control limits are established, a control limit of 75 - 125 % recovery and 20% RPD must be applied to the MS/MSD. If the LCS recovery is within limits, then the laboratory operation is in control and the results may be accepted. If the recovery of the LCS is outside limits, corrective action must be taken. Corrective action will include repreparation and reanalysis of the batch. MS/MSD results which fall outside the control limits must be addressed in the narrative.

- If the native analyte concentration in the MS/MSD exceeds 4 times the spike level for that analyte, the recovery data are reported as NC (i.e., not calculated). If the reporting software does not have the ability to report NC then the actual recovery must be reported and narrated as follows: "Results outside of limits do not necessarily reflect poor method performance in the matrix due to high analyte concentrations in the sample relative to the spike level."

- If an MS/MSD is not possible due to limited sample volume, then a laboratory control sample duplicate (LCSD) should be analyzed. The RPD of the LCS and LCSD must be compared to the matrix spike RPD limits.

9.7. Initial Calibration Verification (ICV/ICB) - Calibration accuracy is verified by analyzing a second source standard (ICV). The ICV result must fall within 20% of the true value for that solution. An ICB is analyzed immediately following the ICV to monitor low level accuracy and system cleanliness. The ICB result must fall within +/- the reporting limit (RL) from zero. If either the ICV or ICB fail to meet criteria, the analysis should be terminated, the problem corrected and the instrument recalibrated. (See Section 11.2.10 and Section 11.2.11 for required run sequence). If the cause of the ICV or ICB failure was not directly instrument related the corrective action will include repreparation of the associated samples.

9.8. Continuing Calibration Verification (CCV/CCB) - Calibration accuracy is monitored throughout the analytical run through the analysis of a known standard after every 10 samples. The CCV must be a mid-range standard at a concentration other than that of the ICV. The CCV result must fall within 20% of the true value for that solution. A CCB is analyzed immediately following each CCV. (See Section 11.2.10 and 11.2.11 for required run sequence.) The CCB result must fall within +/- RL from zero. Each CCV and CCB analyzed must reflect the conditions of analysis of all associated samples. Sample results may only be reported when bracketed by valid ICV/CCV

and ICB/CCB pairs. If a mid-run CCV or CCB fails, the analysis must be terminated, the problem corrected, the instrument recalibrated, the calibration verified and the affected samples reanalyzed. If the cause of the CCV or CCB failure was not directly instrument related the corrective action will include repreparation of the associated samples.

9.9. Method of Standard Addition (MSA) -This technique involves adding known amounts of standard to one or more aliquots of the sample prior to preparation. This technique compensates for a sample interferent that may enhance or depress the analyte signal, thus producing a different slope from that of the calibration standards. It will not correct for additive interferences which cause a baseline shift. Refer to Section 11.2.12 for additional information on when full 4 point MSA is required as well as Appendix C for specific MSA requirements.

10. CALIBRATION AND STANDARDIZATION

10.1. Calibration standards must be processed through the preparation procedure as described in Section 11.1.

10.2. Due to the differences in preparation protocols separate calibration and calibration verification standards must be prepared for aqueous and solid matrices.

10.3. Calibration must be performed daily (every 24 hours) and each time the instrument is set up. The instrument calibration date and time must be included in the raw data.

10.4. Set up the instrument with the operating parameters recommended by the manufacturer. Allow the instrument to become thermally stable before beginning calibration (approximately 30 minutes of warm-up is required). Refer to the facility specific instrument SOP and CVAA instrument manual for detailed setup and operation protocols.

10.5. Calibrate the instrument according to instrument manufacturer's instructions, using a minimum of five standards and a blank. One standard must be at the Quanterra reporting limit. Analyze standards in ascending order beginning with the blank. Refer to Section 7.5 and Table I for additional information on preparing calibration standards and calibration levels.

10.6. The calibration curve must have a correlation coefficient of ≥0.995 or the instrument shall be stopped and recalibrated prior to running samples. Sample results can not be reported from a curve with an unacceptable correlation coefficient.

10.7. Refer to Section 9.0 for calibration verification procedures, acceptance criteria and corrective actions.

11. PROCEDURE

11.1. Standard and Sample Preparation:

11.1.1. All calibration and calibration verification standards (ICV, ICB, CCV, CCB) are processed through the digestion procedure as well as the field samples.

11.1.2. Transfer 0, 0.2, 0.5, 1.0, 2.0, 5.0 and 10.0 mL aliquots of the working standard (7.5) into a series of sample digestion bottles.

Note: Alternate volumes of standard may be prepared as long as the accuracy and final standard concentrations as detailed in Table I are maintained.

11.1.3. Add reagent water to each standard bottle to make a total volume of 23.5 mL. Continue preparation as described under 11.1.5 or 11.1.6 below.

11.1.4. Transfer triplicate 0.2 g or 0.6g total portions of a well mixed sample into a clean sample digestion bottle. Continue preparation as described under 11.2.2 or 11.2.3 below.

11.1.5. Water Bath protocol:

11.1.5.1. To each **standard** bottle: Add 5 mL of aqua regia.
To each **sample** bottle: Add 5 mL of reagent water and 5 mL of aqua regia.

11.1.5.2. Heat for 2 minutes in a water bath at 90 - 95 ° C.

11.1.5.3. Cool.

11.1.5.4. Add 50 mL of distilled water.

11.1.5.5. Add 15 mL of potassium permanganate solution.

11.1.5.6. Add 8 mL of potassium persulfate solution, mix thoroughly

11.1.5.7. Heat for 30 minutes in the water bath at 90 - 95 °C.

11.1.5.8. Cool.

11.1.5.9. Add 6 mL of sodium chloride-hydroxylamine sulfate solution to reduce the excess permanganate.

11.1.5.10. To each **standard** bottle: Add 50 mL of reagent water. To each **sample** bottle: Add 55 mL of reagent water.

11.1.5.11. Continue as described under Section 11.2.

11.1.6. Autoclave protocol:

11.1.6.1. Add 5 mL concentrated of H_2SO_4 and 2.5 mL of concentrated HNO_3.

11.1.6.2. Add 5 mL of saturated potassium permanganate solution.

11.1.6.3. Add 8 mL of potassium persulfate solution.

11.1.6.4. Cover digestion bottle with aluminum foil or screw cap loosely applied.

11.1.6.5. Heat samples at 121 °C and 15 lbs. pressure for 15 minutes.

11.1.6.6. Cool.

11.1.6.7. At this point, there should be 44ml total volume in each standard and sample bottle.

Note: Alternate final volumes may be used as long as the standards and sample are treated the same way.

11.1.6.8. Add 6 mL of sodium chloride-hydroxylamine sulfate solution to reduce excess permanganate.

11.1.6.9. Continue as described under Section 11.2 .

11.2. Sample Analysis:

11.2.1. Because of differences between various makes and models of CVAA instrumentation, no detailed operating instructions can be provided. Refer to

the facility specific instrument operating SOP and the CVAA instrument manual for detailed setup and operation protocols.

11.2.2. All labs are required to detail the conditions/programs utilized for each instrument within the facility specific instrument operation SOP.

11.2.3. Manual determination:

11.2.3.1. Treating each sample individually, purge the head space of the sample bottle for at least one minute.

11.2.3.2. Add 5 mL of stannous chloride solution and immediately attach the bottle to the aeration apparatus.

11.2.3.3. Allow the sample to stand quietly without manual agitation while the sample is aerated (1 L/min flow). Monitor the sample absorbance during aeration. When the absorbance reaches a maximum and the signal levels off, open the bypass valve and continue aeration until the absorbance returns to its baseline level. Close the bypass valve and remove the aeration device.

11.2.3.4. Place the aeration device into 100 mLs of 1% HNO_3 and allow to bubble rinse until the next sample is analyzed.

11.2.4. Automated determination: Follow instructions provided by instrument manufacturer.

11.2.5. Perform a linear regression analysis of the calibration standards by plotting maximum response of the standards vs. ug of mercury. Determine the mercury concentration in the samples from the linear regression fit of the calibration curve. Calibration using computer or calculation based regression curve fitting techniques on concentration/response data is acceptable.

11.2.6. All measurements must fall within the defined calibration range to be valid. Dilute and reanalyze all samples for analytes that exceed the highest calibration standard.

11.2.7. If the sample results are negative and the absolute value of the negative result is greater than the reporting limit, the sample must be diluted and reanalyzed.

11.2.8. The samples must be allowed to cool to room temperature prior to analysis or a decrease in the response signal can occur.

11.2.9. Baseline correction is acceptable as long as it is performed after every sample or after the CCV and CCB; resloping is acceptable as long as it is immediately preceded and followed by a compliant CCV and CCB.

11.2.10. The following analytical sequence must be used with 7471A and 245.5:

Instrument Calibration
ICV
ICB
Maximum 10 samples
CCV
CCB
Repeat sequence of 10 samples between CCV/CCB pairs as required to complete run
CCV
CCB

Refer to Quality Control Section 9.0 and Table II (Appendix A) for quality control criteria to apply to Methods 7471A and 245.5.

Note: Samples include the method blank, LCS, MS, MSD, duplicate, field samples and sample dilutions.

11.2.11. The following run sequence is consistent with 7471A, CLP and 245.5 and may be used as an alternate to the sequence in 11.2.10. This run sequence is recommended if multiple method requirements must be accommodated in one analytical run:

Instrument Calibration
ICV
ICB
CRA*
CCV
CCB
10 samples
CCV
CCB
Repeat sequence of 10 samples between CCV/CCB pairs as required to complete run.

CCV
CCB

Refer to the appropriate CLP SOP (CORP-MT-0008) for quality control requirements for QC samples.

* Refer to the CLP SOP for information on the CRA.

11.2.12. TCLP samples are always prepped as waters:

1) recovery of the analyte in the matrix spike is not at least 50%,

2) the concentration of the analyte does not exceed the regulatory level, and,

3) the concentration of the analyte is within 20% of the regulatory level.

Appendix E provides guidance on performing MSA analyses. For TCLP mercury determinations, MSA spikes must be added prior to sampe preparation.

11.3. To facilitate the early identification of QC failures and samples requiring rerun it is strongly recommended that sample data be reviewed periodically throughout the run.

11.4. Guidelines are provided in the appendices on procedures to minimize contamination of samples and standards, preventive maintenance and troubleshooting.

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

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

12. DATA ANALYSIS AND CALCULATIONS

12.1. ICV percent recoveries are calculated according to the equation:

$$\%R = 100\left(\frac{Found(ICV)}{True(ICV)}\right)$$

12.2. CCV percent recoveries are calculated according to the equation:

$$\%R = 100\left(\frac{Found(CCV)}{True(CCV)}\right)$$

12.3. Matrix spike recoveries are calculated according to the following equation:

$$\%\ R = 100\left(\frac{SSR - SR}{SA}\right)$$

Where:
SSR = Spike Sample Result
SR = Sample Result
SA = Spike Added

12.4. The relative percent difference (RPD) of matrix spike/matrix spike duplicates or sample duplicates are calculated according to the following equations:

$$RPD = 100\left[\frac{|MSD - MS|}{\left(\frac{MSD + MS}{2}\right)}\right]$$

Where:
MS = determined spiked sample concentration
MSD = determined matrix spike duplicate concentration

$$RPD = 100\left[\frac{|DU1 - DU2|}{\left(\frac{DU1 + DU2}{2}\right)}\right]$$

Where:
DU1 = Sample result
DU2 = Sample duplicate result

12.5. For automated determinations, the final concentration determined in solid samples when reported on a dry weight basis is calculated as follows:

mg/kg, dry weight = (C x V x D)/(W x S)

Where:

C = Concentration (ug/L) from instrument readout
V = Volume of digestate (L)
D = Instrument dilution factor
W = Weight in g of wet sample digested
S = Percent solids/100

Note: A Percent Solids determination must be performed on a separate aliquot when dry weight concentrations are to be reported. If the results are to be reported on a wet weight basis, the "S" factor should be omitted from the above equation.

12.6. For manual (total) determinations, the final concentration determined in solid samples when reported on a dry weight basis is calculated as follows:

$$mg/kg,\ dry\ weight = (C)/(W \times S)$$

Where:

C = Concentration (ug) from instrument readout
W = Weight in g of wet sample digested
S = Percent solids/100

Note: A Percent Solids determination must be performed on a separate aliquot when dry weight concentrations are to be reported. If the results are to be reported on a wet weight basis, the "S" factor should be omitted from the above equation.

12.7. The LCS percent recovery is calculated according to the following equation:

$$\%R = 100\left(\frac{Found(LCS)}{True(LCS)}\right)$$

12.8. Sample results should be reported with up to three significant figures in accordance with the Quanterra significant figure policy.

13. METHOD PERFORMANCE

13.1. Each laboratory must have initial demonstration of performance data on file for each analyte of interest as described in Section 9.0.

13.2. Method performance is determined by the analysis of method blank, laboratory control sample, matrix spike and matrix spike duplicate samples. The matrix spike recovery should fall within +/- 25 % and the matrix spike duplicates should compare within 20% RPD. The method blanks must meet the criteria in Section 9.3. The

laboratory control sample should recover within 20% of the true value until in house limits are established.

13.3. Training Qualification:

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

14. POLLUTION PREVENTION

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

15. WASTE MANAGEMENT

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

16. REFERENCES

16.1. Test Methods for Evaluating Solid Waste , Physical/Chemical Methods, SW-846, 3rd Edition, Final Update II, Revision I, September 1994, Method 7471A (Mercury).

16.2. "Methods for the Chemical Analysis of Water and Wastes", EPA-600/4-79-020, U.S.EPA, August 1983, Method 245.5.

16.3. U.S.EPA Statement of Work for Inorganics Analysis, ILMO3.0.

16.4. QA-003, Quanterra QC Program.

16.5. QA-004, Rounding and Significant Figures.

16.6. QA-005, Method Detection Limits.

17. MISCELLANEOUS (TABLES, APPENDICES, ETC. . .)

17.1. Modifications/Interpretations from reference method.

17.1.1. Modifications from both 7471A and 245.5.

17.1.1.1. A potassium persulfate oxidation step has been included to facilitate the breakdown of organic mercurials which are not completely oxidized by potassium permanganate. Use of potassium persulfate in combination with the permanganate improves the recovery of mercury from organo-mercury compounds. The use of persulfate has been incorporated in several recent EPA mercury protocols.

17.1.1.2. The alternate run sequence presented in Section 11.2.11 is consistent with method requirements. An additional QC analysis (CRA) was added to accommodate the CLP protocol requirements.

17.1.2. Modifications from Method 7471A

17.1.2.1. Chapter 1 of SW846 specify the use of reagent water with a purity equivalent to ASTM Type II water. This SOP specifies the use of a Millipore DI system or equivalent to produce reagent water. This SOP requires that reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

17.1.2.2. Chapter 1 of SW-846 states that the method blank should not contain any analyte of interest at or above the MDL. This SOP states that the method blank must not contain any analyte of interest at or above the reporting limit.

17.1.2.3. Method 7471A does not state control criteria within the text of the method. The QC section of 7471A refers the analyst to Section 8.0 of Method 7000A, the generic atomic absorption method, which discusses flame and furnace methods. The ICV criteria stated in Method 7000A is ± 10%. This SOP requires ICV control limits of ± 20% based on the fact that the mercury ICV, unlike the ICV for the flame and furnace analytes, is digested and therefore is equivalent to a LCS. The CLP protocol 245.5 CLP-M recognizes this factor and requires control limits of ± 20%.

17.1.3. Modifications from 245.5

17.1.3.1. Method 245.5 Section 9.3 states concentrations should be reported as follows: Between 0.1 and 1 ug/g, to the nearest 0.01 ug; between 1 and 10 ug/g, to the nearest 0.1ug; above 10 ug/g, to the nearest ug. Quanterra reports all Hg results under this SOP to two significant figures.

17.2. Modifications from previous SOP

None.

17.3. Facility Specific SOPs

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

17.4. Documentation and Record Management

The following documentation comprises a complete CVAA raw data package:

- Raw data (direct instrument printout)
- Run log printout from instrument software where this option is available or manually generated run log. (A bench sheet may be substituted for the run log as long as it contains an accurate representation of the analytical sequence).
- Data review checklist - See Appendix B
- Standards Documentation (source, lot, date).
- Copy of digestion log.
- Non-conformance summary (if applicable).

Figure 1. Solid Sample Preparation for Mercury - Autoclave Procedure

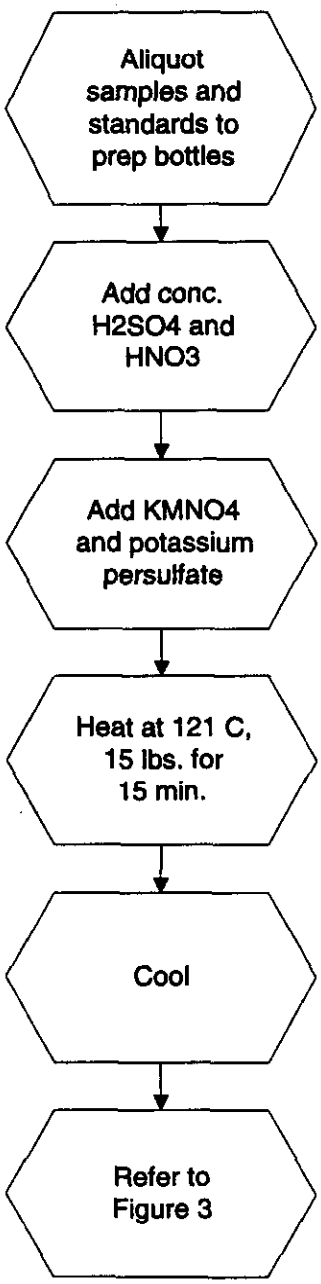


Figure 2. Solid Sample Preparation for Mercury - Water Bath Procedure

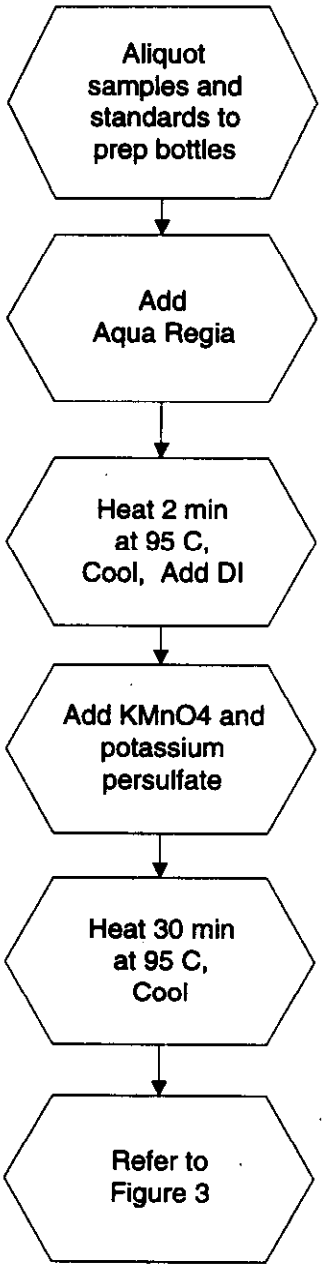
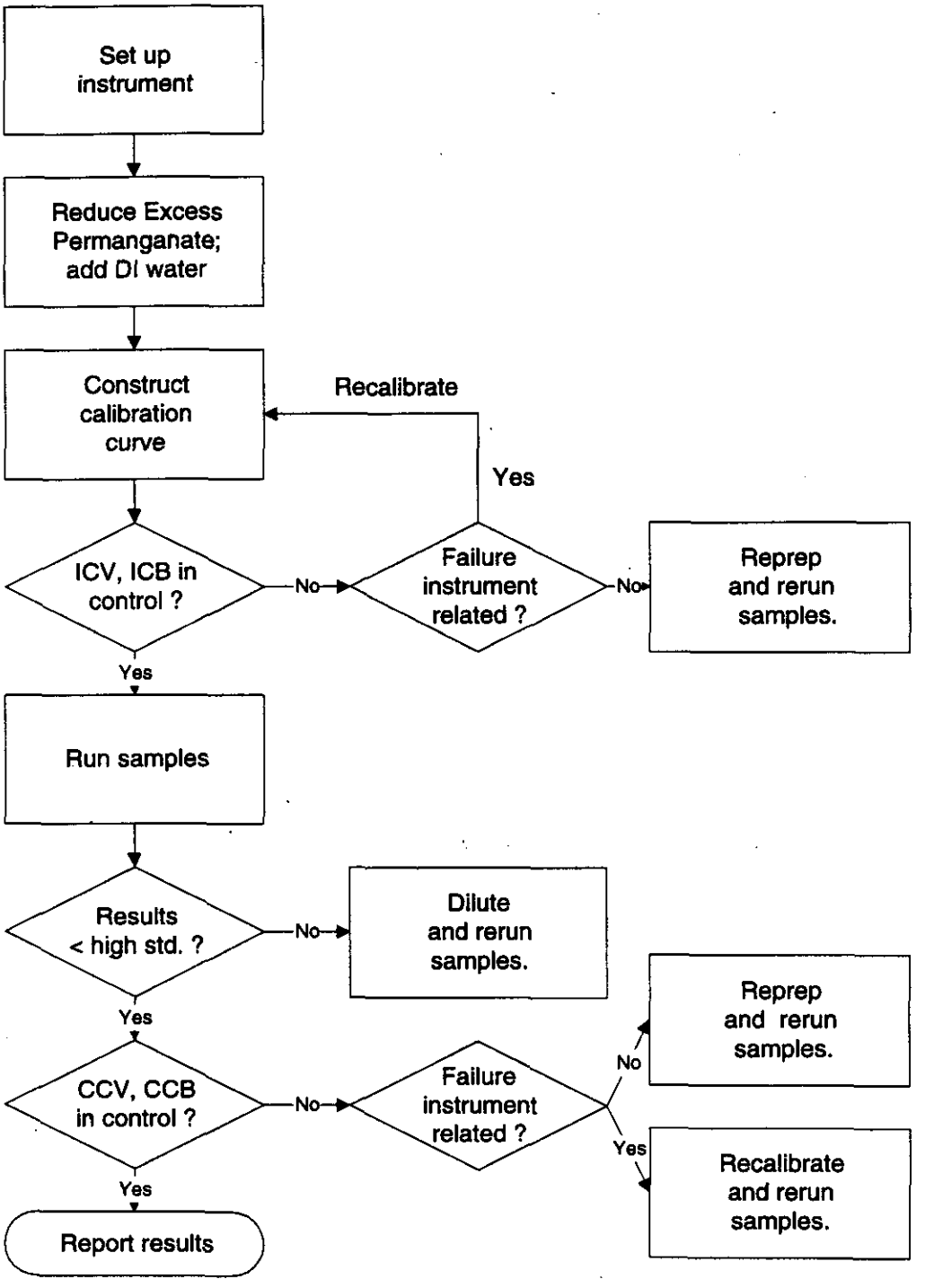


Figure 3. CVAA Mercury Analysis



APPENDIX A

TABLES

TABLE I. MERCURY REPORTING LIMITS, CALIBRATION STANDARD*, QC STANDARD AND SPIKING LEVELS

Soil RL (mg/kg)	0.1
Std 0 (mg/L)	0
Std 1 (mg/L)	0.0002
Std 2 (mg/L)	0.0005
Std 3 (mg/L)	0.001
Std 4 (mg/L)	0.002
Std 5 (mg/L)	0.005
Std 6 (mg/L) **	0.010
ICV (mg/L)	0.001 or 0.0025 ***
Our water LCS for soils currently is being spiked at 1.0 ppb. Our spiked at 5.0 ppb	0.0025 or 0.005 ***
MS (mg/L)	0.001

* **SOP specified calibration levels must be used unless prevented by the instrument configuration or client specific requirements. Deviations from specified calibration levels must be documented in the facility specific instrument operation SOP and must be approved by the facility technical manager and Quality Assurance Manager.**

** **Optional standard which may be used to extend the calibration range as allowed by the instrument configuration. If the instrument configuration prevents the use of 6 standards, the 2 ppb standard may be eliminated in favor of the 10 ppb standard.**

*** **Concentration level dependent on high calibration standard used. CCV must be 50% of the high standard concentration and the ICV must be 20-25% of the high standard concentration.**

TABLE II. Summary Of Quality Control Requirements

QC PARAMETER	FREQUENCY *	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
ICV	Beginning of every analytical run.	80 - 120 % recovery.	Terminate analysis; Correct the problem; Recalibrate or reprep batch (see Section 9.7).
ICB	Beginning of every analytical run, immediately following the ICV.	The result must be within +/- RL from zero.	Terminate analysis; Correct the problem; Recalibrate or reprep batch (see Section 9.7).
CCV	Every 10 samples and at the end of the run.	80 - 120 % recovery.	Terminate analysis; Correct the problem; Recalibrate and rerun all samples not bracketed by acceptable CCV or reprep batch (see Section 9.8).
CCB	Immediately following each CCV.	The result must be within +/- RL from zero.	Terminate analysis; Correct the problem; Recalibrate and rerun all samples not bracketed by acceptable CCB or reprep batch (see Section 9.8).
Method Blank	One per sample preparation batch of up to 20 samples.	The result must be less than or equal to the RL. Sample results greater than 20x the blank concentration are acceptable. Samples for which the contaminant is < RL do not require redigestion (See Section 9.4)	Redigest and reanalyze samples. Note exceptions under criteria section. See Section 9.4 for additional requirements.

*See Sections 11.2.10 and 11.2.11 for exact run sequence to be followed.

TABLE II. Summary of Quality Control Requirements (Continued)

QC PARAMETER	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Laboratory Control Sample (LCS)	One per sample preparation batch of up to 20 samples.	Aqueous LCS must be within 80 - 120% recovery or in-house control limits.	Terminate analysis; Correct the problem; Redigest and reanalyze all samples associated with the LCS (see Section 9.5).
Matrix Spike	One per sample preparation batch of up to 20 samples.	75 - 125 % recovery or in-house control limits. If the MS/MSD is out for an analyte, it must be in control in the LCS.	In the absence of client specific requirements, flag the data; no flag required if the sample level is > 4x the spike added. (see Section 9.6) For TCLP see Section 11.3.12
Matrix Spike Duplicate	See Matrix Spike	75 - 125 % recovery or in-house control limits; RPD ≤ 20%. (See MS)	See Corrective Action for Matrix Spike.

APPENDIX B
QUANTERRA Hg DATA REVIEW CHECKLIST

Quanterra Hg Data Review Checklist

Run/Project Information

Run Date: _____ **Analyst:** _____ **Instrument:** _____
Prep Batches Run: _____

Circle Methods used: **7470A / 245.1 :** CORP-MT-0005 Rev 1 **7471 / 245.5** : CORP-MT-0007 Rev 1
CLP - AQ : CORP-MT-0006 Rev 0 **CLP - SOL** : CORP-MT-0008 Rev 0

Review Items

A. Calibration/Instrument Run QC	Yes	No	N/A	2ndLevel
1. Instrument calibrated per manufacturer's instructions and at SOP specified levels ?				
2. ICV/CCV analyzed at appropriate frequency and within control limits?				
3. ICB/CCB analyzed at appropriate frequency and within +/- RL or +/- CRDL (CLP)?				
4. CRA run (CLP only)?				
B. Sample Results				
1. Were samples with concentrations > the high calibration standard diluted and reanalyzed?				
2. All reported results bracketed by in control QC ?				
3. Sample analyses done within holding time?				
C. Preparation/Matrix QC				
1. LCS done per prep batch and within QC limits ?				
2. Method blank done per prep batch and < RL or CRDL (CLP) ?				
3. MS run at required frequency and within limits ?				
4. MSD or DU run at required frequency and RPD within SOP limits?				
D. Other				
1. Are all nonconformances documented appropriately ?				
2. Current IDL/MDL data on file?				
3. Calculations and Transcriptions checked for error ?				
4. All client/ project specific requirements met?				
5. Date of analysis verified as correct ?				

Analyst: _____ **Date:** _____
Comments:

_____

_____

2nd Level Reviewer : _____ **Date:** _____

_____

_____

APPENDIX C

MSA GUIDANCE

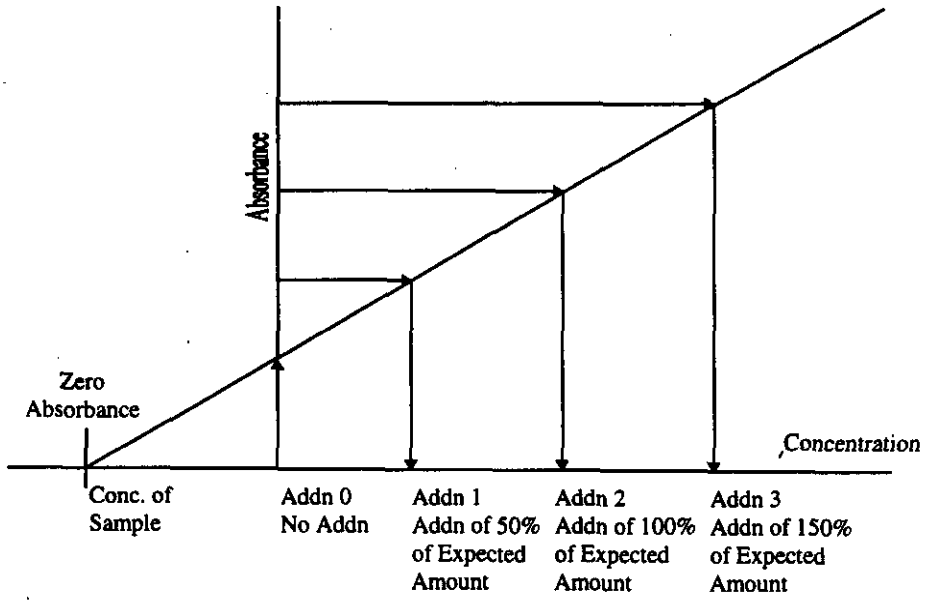
APPENDIX C. MSA GUIDANCE

Method of Standard Addition

Four equal volume aliquots of sample are measured and known amounts of standards are added to three aliquots. The fourth aliquot is the unknown and no standard is added to it. The concentration of standard added to the first aliquot should be 50% of the expected concentration. The concentration of standard added to the second aliquot should be 100% of the expected concentration and the concentration of standard added to the third aliquot should be 150% of the expected concentration. The volume of the unspiked and spiked aliquots should be the same (i.e., the volume of the spike added should be negligible in relation to the volume of sample).

To determine the concentration of analyte in the sample, the absorbance (or response) of each solution is determined and a linear regression performed. On the vertical axis the absorbance (or response) is plotted versus the concentrations of the standards on the horizontal axis using 0 as the concentration of the unspiked aliquot. An example plot is shown in Figure 1. When the resulting line is extrapolated back to zero absorbance, the point of interception of the horizontal axis is the concentration of the unknown. Calculate the correlation coefficient (r) and the x-intercept (where y=0) of the curve. The concentration in the digestate is equal to the negative x-intercept.

Figure 1



- For the method of standard additions to be correctly applied, the following limitations must be taken into consideration.
- The plot of the sample and standards must be linear over the concentration range of concern. For best results, the slope of the curve should be similar to that of a plot of the aqueous standard curve.
- The effect of the interference should not vary as the ratio of the standard added to the sample matrix changes.

APPENDIX D

TROUBLESHOOTING GUIDE

APPENDIX D. TROUBLESHOOTING GUIDE

Problem	Possible Cause
Poor or No Absorbance or Sensitivity Check failed	Incorrect wavelength Dirty windows Window loose Etched or dirty optics Wrong lamp Bad lamp Not enough or no sample introduced Empty sample cup Incorrectly made standards Gas leak EDL power supply set on "Continuous"
Erratic Readings	Source lamp not aligned properly Lamp not prewarmed Injection tip partially clogged Contaminated reagents Contaminated glassware Drying tube saturated Bad lamp Injection tip hitting outside of tube Injection tip coated or not set properly Leak in sample tubing Power fluctuations Air bubbles in tubing
EDL Won't Light	Lamp cable not plugged in Lamp power set at 0 Lamp is dead Power supply fuse is blown Short in cord
Standards reading twice or half normal absorbance or concentration	Incorrect standard used Incorrect dilution performed Dirty cell
Background Correction Light Blinking	Background screen or attenuator faulty

APPENDIX E

CONTAMINATION CONTROL GUIDELINES

APPENDIX E. CONTAMINATION CONTROL GUIDELINES

The following procedures are strongly recommended to prevent contamination:

All work areas used to prepare standards and spikes should be cleaned before and after each use.

All glassware should be washed with detergent and tap water and rinsed with 1:1 nitric acid followed by deionized water.

Proper laboratory housekeeping is essential in the reduction of contamination in the metals laboratory. All work areas must be kept scrupulously clean.

Powdered or Latex Gloves must not be used in the metals laboratory since the powder contains silica and zinc, as well as other metallic analytes. Only vinyl or nitrile gloves should be used in the metals laboratory.

Glassware should be periodically checked for cracks and etches and discarded if found. Etched glassware can cause cross contamination of any metallic analytes.

Autosampler trays should be covered to reduce the possibility of contamination. Trace levels of elements being analyzed in the samples can be easily contaminated by dust particles in the laboratory.

The following are helpful hints in the identification of the source of contaminants:

Reagents or standards can contain contaminants or be contaminated with the improper use of a pipette.

Improper cleaning of glassware can cause contamination.

Separate glassware if an unusually high sample is analyzed and soak with sulfuric acid prior to routine cleaning.

APPENDIX F

PREVENTIVE MAINTENANCE

APPENDIX F. PREVENTIVE MAINTENANCE

A maintenance log is used to record when maintenance is performed on instruments. When an instrument problem occurs indicate the date, time and instrument number, then identify the problem and corrective action in the maintenance log.

The following procedures are required to ensure that that the instrument is fully operational.

Cold Vapor Atomic Absorption (Leeman PS 200) (1)

Daily	Semi-annually	Annually
Check Optic Intensity	Check Hg lamp intensity. If intensity is below 500,000 clean cell and lenses	Change Hg lamp.
Check aperture.		Check liquid/gas separator.
Check argon flow.		
Check tubing.		
Check drain.		
Replace drying tube.		

Cold Vapor Atomic Absorption (PE 5000) (1)

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

Controlled Copy
Copy No._____

LABORATORY -SPECIFIC

QUANTERRA STANDARD OPERATING PROCEDURE

TITLE: GRAPHITE FURNACE ATOMIC ABSORPTION SPECTROSCOPY, SW-846 METHOD 7000A AND MCAWW 200 SERIES METHODS

(SUPERSEDES: CORP-MT-0003, REV. 1.0)

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C4-287

TABLE OF CONTENTS

1. SCOPE AND APPLICATION 3

2. SUMMARY OF METHOD 4

3. DEFINITIONS 4

4. INTERFERENCES 4

5. SAFETY 7

6. EQUIPMENT AND SUPPLIES 9

7. REAGENTS AND STANDARDS 9

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE 10

9. QUALITY CONTROL 10

10. CALIBRATION AND STANDARDIZATION 15

11. PROCEDURE 16

12. DATA ANALYSIS AND CALCULATIONS 20

13. METHOD PERFORMANCE 22

14. POLLUTION PREVENTION 22

15. WASTE MANAGEMENT 22

16. REFERENCES 22

17. MISCELLANEOUS (TABLES, APPENDICES, ETC. . .) 23

LIST OF APPENDICES:

APPENDIX A - TABLES 29

APPENDIX B - QUANTERRA GFAA DATA REVIEW CHECKLIST 36

APPENDIX C - MSA GUIDANCE 38

APPENDIX D - TROUBLESHOOTING GUIDE 41

APPENDIX E- CONTAMINATION CONTROL GUIDELINES 44

APPENDIX F - PREVENTATIVE MAINTENANCE 46

APPENDIX G - FOOTNOTES AND QUALIFIERS

APPENDIX H - FIRST AID PLAN/HYDROFLUORIC ACID

1. SCOPE AND APPLICATION

1.1. This procedure describes the analysis of certain metals by Graphite Furnace Atomic Absorption Spectroscopy (GFAA) using SW-846 protocol, Method 7000A and the MCAWW 200 series methods. Table I (Appendix A) lists the routine elements approved for analysis by Method 7000A and the 200 series methods. Table IA (Appendix A) lists non-routine elements that may be analyzed under Method 7000A and the 200 series methods, provided the method performance criteria presented in Section 13.0 are met.

1.2. GFAA analysis provides for the determination of metal concentrations in the parts per billion range. Detection limits, sensitivity and optimum concentration ranges of the metals will vary with the matrices and instrumentation used. The sensitivity and limited linear dynamic range of GFAA often implies the need to dilute a sample prior to analysis. The actual magnitude of the dilution can dramatically influence the quality of the analytical results. Therefore, sample types requiring large dilutions (>50:1) should be analyzed by another client approved test procedure that has a larger linear dynamic range or that is inherently less sensitive than GFAA.

1.3. Method 7000A is applicable to the determination of dissolved, suspended, total recoverable and total elements in ground water, aqueous samples, solids, sludges, wastes, sediments, air sampling media, biological tissue, wipes and TCLP, EP and other leachates/extracts. All matrices require digestion prior to analysis, with the exception of analyses for dissolved metals in filtered and acidified aqueous samples. Although digestion is not specifically required by the method, some clients and regulators do require digestion of dissolved samples and this must be clarified before project initiation.

1.4. Series methods are applicable to the determination of dissolved, suspended, total recoverable and total elements in ground water, aqueous samples, solids, sludges, wastes, and sediments. All matrices require digestion prior to analysis, with the exception of analyses for dissolved metals in filtered and acidified aqueous samples if the criteria in Section 11.2 are met. Although digestion is not specifically required by the method, some clients and regulators do require digestion of dissolved samples and this must be clarified before project initiation.

1.5. This method is not applicable to drinking water samples due to the wide array of state specific requirements which must be accommodated. Refer to facility specific SOPs for guidance on performing drinking water analyses.

2. SUMMARY OF METHOD

2.1. This method describes a technique for the determination of elements in solution. The basis of the method is the measurement of atomic absorption by an optical spectroscopic technique. A representative aliquot of a sample is placed in a graphite tube in the furnace, evaporated to dryness, charred and atomized, causing the atoms to be vaporized and dissociated within the graphite tube. A light beam from a hollow cathode lamp (HCL) or electrodeless discharge lamp (EDL) is directed through the graphite tube. The intensity of the transmitted radiation decreases in proportion to the amount of the ground state atoms in the vapor contained within the graphite tube. Because the wavelength of the light beam is characteristic of the metal being determined, the light energy absorbed by the sample in the tube is a measure of the concentration of that metal in the sample. Concentration of the analyte in the sample is determined by comparison of the sample absorbance to the calibration curve (absorbance vs. concentration).

2.2. Consult the appropriate SOP's for details on sample preparation methods.

3. DEFINITIONS

3.1. Dissolved Metals: Those elements which pass through a 0.45 um membrane. (Sample is acidified after filtration).

3.2. Suspended Metals: Those elements which are retained by a 0.45 um membrane.

3.3. Total Metals: The concentration determined on an unfiltered sample following vigorous digestion.

3.4. Total Recoverable Metals: The concentration determined on an unfiltered sample following treatment with hot, dilute mineral acid.

4. INTERFERENCES

Chemical and physical interferences are prevalent when analyzing samples using these methods.

4.1. The problem of oxide formation is greatly reduced with furnace procedures because atomization occurs in an inert atmosphere. The technique, however, is still subject to chemical interferences. The composition of the sample matrix can have a major effect on the analysis and must be taken into consideration in the analysis of each different matrix encountered. The effects of the sample matrix are evaluated by following the spike recovery check procedure described in Section 9.7.

4.2. Matrix modifiers are added to the samples prior to the determination of most analytes. The modifier prevents premature loss of the analytes as volatile salts and/or helps prevent signal suppression during atomization due to the formation of high temperature stable complexes.

4.3. Background interferences must be compensated for by the use of background correction. Background correction is especially important below 350 nm. Certain samples, when atomized, may absorb or scatter light from the lamp. This can be caused by the presence of gaseous molecular species, salt particles, or smoke in the sample beam. Zeeman background correction must be used for all analysis. Zeeman background correction is effective in overcoming composition or structured background interferences. It is particularly useful when analyzing for As in the presence of Al and when analyzing for Se in the presence of Fe.

4.4. Interference from a smoke-producing sample matrix can sometimes be reduced by extending the charring time at a higher temperature or utilizing an ashing cycle in the presence of air, or by diluting the sample. Care must be taken, however, to prevent the loss of the analyte.

4.5. Samples containing large amounts of organic materials should be oxidized by acid digestion before being placed in the furnace. In this way, broad band absorption will be minimized.

4.6. Carbide formation, resulting from the chemical environment of the furnace, has been observed in certain elements that form carbides at higher temperatures (e.g., Mo, V). When carbides form, the metal is released very slowly from the resulting metal carbide during the atomization cycle. For example, molybdenum may require 30 sec or more atomization time for the signal to return to the baseline level. Carbide formation is greatly reduced and sensitivity increased with the use of pyrolytically coated graphite and must be used for all analysis.

4.7. Based on anion interference studies in the graphite furnace, it is generally accepted that nitrate is the preferred anion to combine with metal cations. Therefore, nitric acid is preferable for any digestion or solubilization step. If another acid in addition to nitric acid is required, a minimum amount should be used. This applies particularly to hydrochloric acid and, to a lesser extent, to sulfuric and phosphoric acids.

4.8. Memory effects occur when the analyte is not totally volatilized during atomization. The analyte which remains in the furnace can produce false positive signals on subsequent samples. This condition depends on several factors: volatility of the element and its chemical form, whether pyrolytic graphite is used, the rate of atomization and furnace design. The analyst should establish the analyte

concentration which can be injected into the furnace and adequately removed in one complete set of furnace cycles. This concentration represents the maximum concentration of analyte within a sample which will not cause a memory effect on the subsequent samples. If during a sample analysis run this concentration is exceeded, the tube should be cleaned by performing blank tube burns , as needed, to assure the memory interference has been eliminated before continuing sample analysis.

4.9. Specific element interferences

- High lead concentrations cause spectral interferences for **antimony** on the 217.6-nm line. The interference is eliminated by the use of Zeeman background correction or the use of the secondary wavelength. Antimony also suffers from an interference produced by K_2SO_4.

- **Arsenic** and **selenium** analyses are subject to many interferences. Elemental arsenic and selenium and their compounds are volatile and are subject to losses during sample preparation. Verify that the analyte is not volatilized using spike samples. A matrix modifier must be added to minimize volatilization during drying and ashing. Caution must be employed in selecting temperatures and times for the dry and char steps. In addition to the normal interferences, arsenic and selenium can suffer from severe nonspecific absorption and light scattering caused by matrix components during atomization. Arsenic analysis is particularly susceptible to these problems because of its low analytical wavelength (193.7 nm). Zeeman background correction is used to avoid erroneous high results for arsenic from aluminum interferences and low bias for selenium from iron interferences.

- **Cadmium** can be affected by severe nonspecific absorption and light scattering caused by matrix during atomization. To minimize this, simultaneous background correction is required. A matrix modifier solution can reduce the effects of premature volatilization from excess chlorides.

- Due to interferences from nonspecific absorption and scattering, background correction should be used when analyzing for **chromium**. Low concentrations of calcium and/or phosphate may also cause interferences. Chromium is very susceptible to carbide formation and memory effects. . Nitrogen should not be used as the purge gas because of a possible CN band interference.

- For **lead**, matrix modification should be used to suppress sulfate interferences.

- The use of hydrochloric acid should be avoided for **silver** analysis since silver chloride is insoluble. Storage of silver standards in brown bottles may prevent silver nitrates from plating on the container walls.

- Hydrochloric acid or excessive chlorides will cause volatilization of **thallium** at low temperatures. The use of spike samples helps to verify that losses are not occurring.

5. SAFETY

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

5.2. Eye protection that satisfies ANSI Z87.1 (as per the Chemical Hygiene Plan), laboratory coat, and chemically resistant gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.Neoprene, natural rubber, nitrile and TRIonic® Cleanroom gloves provide varying degrees of protection against those chemicals listed. Refer to permeation/degradation charts for the actual data.

5.3. The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the Material Safety Data Sheets (MSDS) maintained in the laboratory. The following specific hazards are known:

5.3.1. The following materials are known to be **corrosive:**

hydrochloric acid, nitric acid, sulfuric acid, phosphoric and hydrofluoric acid.

5.3.2. The following materials are known to be **oxidizing agents**:

nitric acid and hydrogen peroxide.

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

5.5. The preparation of standards,reagents, and the processing of samples will be conducted in a fume hood with the sash closed as far as the operation will permit.

5.6. The use of hydrofluoric acid requires special safety precautions. Consult the facility EH&S manager and laboratory supervisor for guidance.

NOTE: Contact may not be painful at first. The fluoride ion rapidly penetrates the skin and may cause delayed effects: lasting ulcerations, bone degeneration, pulmonary edema, muscle paralysis, and cardiac arrest. Any suspected exposure to HF liquid/fumes must be immediately evaluated by appropriate medical staff (i.e. U.C. Davis Medical Group Emergency Room.) Review Appendix H for a detailed first aid plan before work with HF begins.

5.6.1. The CA Emergency Medical Services Authority recommends a magnesium sulfate solution (Epsom salt) or lime water (CaOH) as effective irrigating solutions. Magnesium-containing antacids (Maalox® or Mylanta®) can be applied topically.

5.6.2. Some metals react with HF to release flammable hydrogen gas.

5.6.3. Glass reacts with HF to produce toxic silicon tetrafluoride.

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

5.8. The Zeeman background correction technique utilizes a strong magnet. People with electronic cardiac pacemakers or similar devices that are affected by strong magnetic fields should not be near the instruments during operation.

5.9. Do not look directly into the beam of a HCL or EDL. The UV light that these lamps radiate is harmful to the eyes.

5.10. Do not look directly at the graphite tube when it is heated to incandescence to avoid possible injury to the eyes from the intense radiation.

5.11. The furnace heats to approximately 3000°C during atomization. Therefore, to avoid burns, be cautious not to touch the furnace housing until the furnace has cooled sufficiently.

5.12. Cylinders of compressed gas must be handled with caution, in accordance with local regulations. It is recommended that, wherever possible, cylinders be located outside the laboratory and the gas led to the instrument through approved lines.

5.13. The furnace must be properly vented with an exhaust hood directly over the furnace chamber to remove potentially harmful fumes generated when samples are heated.

6. EQUIPMENT AND SUPPLIES

6.1. Atomic Absorption Spectrophotometer equipped with graphite furnace, autosampler and background correction.

6.2. Hollow cathode (HCL) or electrodeless discharge lamp (EDL) with EDL power supply.

6.3. Argon gas supply, welding grade or equivalent.

6.4. Pyrolytically coated graphite tubes and platforms.

6.5. Coolflow or appropriate water cooling device.

6.6. Calibrated automatic pipettes or Class A glass volumetric pipettes.

6.7. Class A volumetric flasks.

6.8. Autosampler cups.

6.9. Disposable cups or tubes.

7. REAGENTS AND STANDARDS

7.1. Stock standards are purchased as custom Quanterra multi-element mixes or as single element solutions. All standards must be stored in FEP fluorocarbon or previously unused polyethylene or polypropylene bottles. Stock standard solutions must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the stock solutions may be used for up to one year and must be replaced sooner if verification from an independent source indicates a problem.

7.2. Working calibration and calibration verification solutions must be made daily. Standards must be prepared in a matrix of 2% HNO_3 per 100 mL for all analytes except antimony. Antimony standards must be made in a matrix of 2% HNO_3 and 5% HCl.

7.3. Refer to Tables III, IV, and V (Appendix A) for details regarding the working standard concentrations for calibration, calibration verification and spiking solutions.

7.4. Concentrated nitric acid (HNO_3), trace metal grade or better.

7.5. Concentrated hydrochloric acid (HCl), trace metal grade or better.

7.6. Hydrogen peroxide (H_2O_2), 30%.

7.7. Reagent water must be produced by a Millipore DI system or equivalent. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

7.8. Matrix modifiers as recommended by instrument manufacturers. The use of a palladium modifier is strongly recommended for the determination of all analytes. This will correct for general chemical interferences as well as allow for higher char and atomization temperatures without allowing the premature liberation of analyte.

8. SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1. Sample holding times for metals are six months from time of collection to the time of analysis.

8.2. Aqueous samples are preserved with nitric acid to a pH of <2 and may be stored in either plastic or glass. Refrigeration is not required. Preservation must be verified prior to analysis.

8.3. Soil samples do not require preservation but must be stored at 4°C ± 2° until the time of analysis.

9. QUALITY CONTROL

Table VI (Appendix A) provides a summary of quality control requirements including type, frequency, acceptance criteria and corrective action.

9.1. Initial Demonstration of Capability

Prior to the analysis of any analyte using 7000A or the 200 series methods, the following requirements must be met.

9.1.1. Instrument Detection Limit (IDL) -The IDL for each analyte must be determined for each analyte wavelength used on each instrument. The IDL must be determined annually. If the instrument is adjusted in any way that may affect the IDL, the IDL for that instrument must be redetermined. For example, the IDL must be redetermined after the instrument is moved or the internal optics cleaned. The IDL shall be determined by multiplying by 3, the standard deviation obtained from the analysis of a standard solution (each analyte in reagent water) at a concentration 3 - 5 times the previously determined IDL, with seven consecutive measurements. Each measurement

must be performed as though it were a separate analytical sample (i.e., each measurement must be followed by a rinse and/or any other procedure performed between the analysis of separate samples). Analytical spikes are not required for IDL determinations by 7000A and 200 series methods. IDLs determined by the CLP procedure can be used for this method. The result of the IDL determination must be below the Quanterra reporting limit.

9.1.2. Method Detection Limit (MDL) - An MDL must be determined for each analyte/matrix prior to the analysis of any samples. The MDL is determined using seven replicates of reagent water, spiked with all the analytes of interest, that have been carried through the entire analytical procedure. MDLs must be redetermined on an annual basis in accordance with 40 CFR Part 136 Appendix B requirements as detailed in Quanterra QA Policy QA-005. The spike level must be between the calculated MDL and 10X the MDL to be valid. The result of the MDL determination must be below the Quanterra reporting limit.

9.2. Method Blank (MB) - One method blank must be processed with each preparation batch. The method blank consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. The method blank should not contain any analyte of interest at or above the reporting limit (exception: common laboratory contaminants, see below) or at or above 5% of the measured concentration of that analyte in associated samples, whichever is higher (sample result must be a minimum of 20 times higher than the blank contamination level).

- If the analyte is a common laboratory contaminant (copper, iron, lead or zinc) the data may be reported with a qualifier if the concentration of the analyte in the method blank is less than two times the RL (see Appendix G) **Such action must be taken in consultation with the client and must be addressed in the project narrative.**

- Repreparation and reanalysis of all samples associated with an unacceptable method blank is required when reportable concentrations are determined in the samples (see exception noted above).

- If there is no analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. **Such action must be taken in consultation with the client and must be addressed in the project narrative.**

- If the above criteria are not met and reanalysis is not possible, then the sample data must be qualified. **This anomaly must be addressed in the project narrative and the client must be notified.**

- For dissolved metals samples which have not been digested, a continuing calibration blank (CCB) result is reported as the method blank. The CCB run immediately prior to the start of the dissolved sample analyses must be used for this purpose. No more than 20 samples can be associated with one CCB.

9.3. Laboratory Control Sample (LCS) - One aqueous LCS must be processed with each preparation batch. The LCS must contain all analytes of interest and must be carried through the entire analytical procedure. The LCS is used to monitor the accuracy of the analytical process. Aqueous LCS spike levels are provided in Table III (Appendix A). On-going monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines.

- If any analyte is outside established control limits the system is out of control and corrective action must occur. Until in-house control limits are established, a control limit of 80 - 120% recovery must be applied.

- In the event that an MS/MSD analysis is not possible, a Laboratory Control Sample Duplicate (LCSD) must be analyzed. The RPD of the LCS and LCSD must be compared to the matrix spike RPD limits.

- In the instance where the LCS recovery is greater than 120% and the sample results are < RL, the data may be reported with qualifiers. **Such action must be taken in consultation with the client and must be addressed in the case narrative.**

- Corrective action will be repreparation and reanalysis of the batch unless the client agrees that other corrective action is acceptable.

- For dissolved metals samples which have not been digested, a continuing calibration verification (CCV) result is reported as the LCS. The CCV run immediately prior to the start of the dissolved sample analyses must be used for this purpose. No more than 20 samples can be associated with one CCV.

9.4. Matrix Spike/Matrix Spike Duplicate (MS/MSD) - One MS/MSD pair must be processed for each preparation batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Some client specific data quality objectives (DQO's) may require the use of sample duplicates in place of or in

addition to MS/MSD's. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, these results may have immediate bearing only on the specific sample spiked. Samples identified as field blanks cannot be used for MS/MSD analysis. Spiking levels are provided in Table III (Appendix A).

- If any analyte recovery or RPD falls outside the acceptance range, the recovery of that analyte must be in control for the LCS and the MS/MSD must be reanalyzed once to confirm the out of control event. Until in-house control limits are established, a control limit of 80 - 120 % recovery and 20% RPD must be applied to the MS/MSD. If the LCS recovery is within limits, then the laboratory operation is in control and the results may be accepted. If the recovery of the LCS is outside limits, corrective action must be taken. Corrective action will include repreparation and reanalysis of the batch. MS/MSD results which fall outside the control limits must be addressed in the narrative.

- If the native analyte concentration in the MS/MSD exceeds 4 times the spike level for that analyte, the recovery data are reported as NC (i.e., not calculated). If the reporting software does not have the ability to report NC then the actual recovery must be reported and narrated as follows: "Results outside of limits do not necessarily reflect poor method performance in the matrix due to high analyte concentrations in the sample relative to the spike level."

- If an MS/MSD is not possible due to limited sample volume, then a laboratory control sample duplicate (LCSD) should be analyzed. The RPD of the LCS and LCSD must be compared to the matrix spike RPD limits.

- For dissolved metals samples which have not been digested, a MS/MSD must be performed per batch of up to 20 samples by spiking two aliquots of the sample at the levels specified in Table III (Appendix A).

9.5. Initial Calibration Verification (ICV/ICB) - Calibration accuracy is verified by analyzing a second source standard (ICV). The ICV result must fall within 10% of the true value for that solution. An ICB is analyzed immediately following the ICV to monitor low level accuracy and system cleanliness. The ICB result must fall within +/- the reporting limit (RL) from zero. If either the ICV or ICB fail to meet criteria, the analysis should be terminated, the problem corrected and the instrument recalibrated. (See Section 11.14 for required run sequence).

9.6. Continuing Calibration Verification (CCV/CCB) - Calibration accuracy is monitored throughout the analytical run through the analysis of a known standard after every 10

samples. The CCV must be a mid-range standard at a concentration other than that of the ICV. For analyses that require 200 series methods the CCV result must fall within 10% of the true value. For Method 7000A, the CCV result must fall within 20% of the true value for that solution. A CCB is analyzed immediately following each CCV. (See Section 11.14 and 11.15 for required run sequence.) The CCB result must fall within +/- RL from zero. Each CCV and CCB analyzed must reflect the conditions of analysis of all associated samples. Sample results may only be reported when bracketed by valid ICV/CCV and ICB/CCB pairs. If a mid-run CCV or CCB fails, the analysis must be terminated, the problem corrected, the instrument recalibrated, the calibration verified and the affected samples reanalyzed.

9.7. Spike Recovery Check (AS) - In order to verify the presence or absence of matrix interferences, a post digest analytical spike is analyzed for each sample, including the method blank. A post digest spike is not required on the LCS or matrix spike sample. The analytical spike of a sample must be run immediately after that sample. See Table III (Appendix A) for spiking levels. The percent recovery of the spike will then determine how the sample will be quantitated and reported as follows:

- If the spike recovery is less than 40%, the sample must be diluted and rerun with another spike (recommended dilution 5-10X) and flagged with the appropriate qualifier (see Appendix G). If the spike recovery still falls less than 40% the client should be contacted and an alternate method of analysis recommended (i.e., Trace or ICP).

- If the spike recovery is greater than or equal to 40% and less than 85%, report the data with the following flag: "Post digestion spike recovery fell between 40-85% due to matrix interference."

- If the spike recovery is greater than or equal to 85% and less than or equal to 115%, report the data unflagged.

- If the spike recovery is greater than 115% and less than 150% and the sample result is less than the Quanterra reporting limit, then report the data unflagged.

- If the spike recovery is greater than 115% and less than 150% and the sample result is greater than the Quanterra reporting limit, then report the data with the following flag "l": "Post digestion spike recovery fell between 115-150% due to matrix interference."

- If the spike recovery is greater than 150% and the sample result is less than the Quanterra reporting limit, then report the data unflagged.

- If the spike recovery is greater than 150% and the sample result is greater than the Quanterra reporting limit then dilute the sample and rerun with another spike. Until the spike recovery falls less than 150%, this dilution step must be repeated.

- If the method blank (MB) analytical spike recovery is less than 85% or greater than 115%, the spiking solution must be verified by respiking and rerunning the MB once. If the MB analytical spike is still out of control, correct the problem and reanalyze all analytical samples associated with that blank.

9.8. Method of Standard Addition (MSA) -This technique involves adding known amounts of standard to one or more aliquots of the processed sample solution. This technique compensates for a sample interferent that may enhance or depress the analyte signal, thus producing a different slope from that of the calibration standards. It will not correct for additive interferences which cause a baseline shift. By definition, the recovery check procedure defined in Section 9.7 is a single point MSA. Sample results are not biased corrected based on the single point MSA result unless this is specifically requested by the client. Refer to Section 11.16 for additional information on when full 4 point MSA is required as well as Appendix C for specific MSA requirements.

9.9. Quality Assurance Summaries - Certain clients may require specific project or program QC which may supersede the SOP requirements. Quality Assurance Summaries (QAS) should be developed to address these requirements.

10. CALIBRATION AND STANDARDIZATION

10.1. Set up the instrument with the operating parameters recommended by the manufacturer. Allow the instrument to become thermally stable before beginning calibration (approximately 30 minutes of warm-up is required). Refer to the facility specific instrument SOP and GFAA instrument manual for detailed setup and operation protocols.

10.2. Calibrate the instrument according to instrument manufacturer's instructions, using a minimum of three standards and a blank. See Table V (Appendix A) for calibration standard details. One standard must be at the Quanterra reporting limit as defined in Tables I and IA (Appendix A). Calibration standards must be prepared fresh daily.

10.3. Calibration must be performed daily (every 24 hours) and each time the instrument is set up for an element. The instrument calibration date and time must be included in the raw data.

10.4. Refer to Section 9.0 for calibration verification procedures, acceptance criteria and corrective actions.

11. PROCEDURE

11.1. Because of differences between various makes and models of GFAA instrumentation, no detailed operating instructions can be provided. Refer to the facility specific instrument operating SOP and the GFAA instrument manual for detailed setup and operation protocols.

- Table II (Appendix A) provides recommended GFAA analytical guidelines for wavelength, char and atomization temperatures, and tube type.

- All labs are required to detail the furnace conditions/programs utilized for each analyte/instrument within the facility specific instrument operation SOP.

11.2. For 200 series analyses, dissolved samples must be digested unless it can be documented that the sample meets all of the following criteria:

A. COD is < 20 ppm.
B. Visibly transparent with a turbidity measurement of 1 NTU or less.
C. Colorless with no perceptible odor.
D. Is of one liquid phase and free of particulate or suspended matter following acidification.

11.3. The use of an autosampler for all runs is required. The use of an autosampler for the addition of matrix modifier and analytical post digestion spikes is strongly recommended if the instrument in use is capable of the automated additions.

11.4. To reduce the number of reruns due to dilutions it is recommended that whenever possible the samples be run by ICP first to "screen" the samples prior to GFAA analysis. The information obtained from the ICP run will enable the analyst to either eliminate the GFAA analysis completely (i.e., if sufficiently high report ICP result with client approval) or dilute the samples appropriately. Screening also may prevent potential contamination of the analytical system as well as other samples during the instrument run.

11.5. To facilitate the early identification of QC failures and samples requiring rerun it is strongly recommended that sample data be reviewed periodically throughout the run.

11.6. To facilitate the data review and reporting processes it is strongly recommended that all necessary dilutions and post digestion spikes be performed before closing out the instrument run.

11.7. Daily, prior to the analysis of samples, a sensitivity check must be performed and recorded. The sensitivity of the instrument may be checked by calculating the

characteristic mass. If the daily results do not compare within +/- 30% of the reference sensitivity the instrument may need adjustment. Refer to Appendix D for assistance if the criterion is not met.

11.8. At a minimum, single burn analyses will be performed. When double burns are performed, the CV or RSD must be less than or equal to 20% if the sample results are greater than the reporting limit.

11.9. All measurements must fall within the defined calibration range to be valid. Dilute and reanalyze all samples for analytes that exceed the highest calibration standard. Acid strength must be maintained in the dilution of samples through the use of acidified dilution water (2% HNO_3).

11.10. If the sample results are negative and the absolute value of the negative result is greater than the reporting limit, the sample must be diluted and reanalyzed.

11.11. Samples requiring large dilutions due to the presence of high concentrations of analyte may be reported using alternate techniques (i.e., ICP, flame) with the approval of the client. Whenever possible, samples should be processed by ICP, prior to GFAA, to facilitate in determining appropriate dilution factors or eliminate the GFAA analysis altogether.

11.12. Baseline correction or cleanout burns are acceptable as long as it is performed after every sample or after the CCV and CCB; resloping is acceptable as long as it is immediately preceded and followed by a compliant CCV and CCB. Cleanout burns must be counted as injections when determining instrument QC frequency.

11.13. Graphite tubes may be changed during an analytical run, providing the last samples analyzed on the tube are a compliant CCV and CCB, and the first samples analyzed on the new tube are a compliant CCV and CCB.

11.14. The following analytical sequence must be used with 7000A and 200 series methods:

Instrument Calibration (Blank and three standards)
ICV
ICB
Maximum 10 samples
CCV
CCB
Maximum 10 samples
CCV
CCB

Repeat sequence of 10 samples between CCV/CCB pairs as required to complete run
CCV
CCB

Refer to Quality Control Section 9.0 and Table VI (Appendix A) for quality control criteria to apply to Methods 7000A and the 200 series methods.

Note: Samples include the method blank, LCS, MS, MSD, duplicate, field samples and sample dilutions. Spike recovery check (AS) injections are not considered as independent samples for the purpose of determining instrument QC frequency.

11.15. The following run sequence is consistent with 7000A, CLP and 200 series, and may be used as an alternate to the sequence in 11.14. This run sequence is recommended if multiple method requirements must be accommodated in one analytical run:

Instrument Calibration (Blank and three standards)
ICV
ICB
CRA**
CCV
CCB
5* samples with AS (maximum 20 burns)
CCV
CCB
5* samples with AS (maximum 20 burns)
CCV
CCB
Repeat sequence of 5* samples between CCV/CCB pairs as required to complete run.
CCV
CCB

Refer to the appropriate CLP SOP (CORP-MT-0004) for quality control requirements for QC samples.

* Note- Only 5 samples can be analyzed between CCV/CCB because double burn analyses are performed as required by CLP protocols.

**Note- Refer to the CLP SOP for information on the CRA.

11.16. For TCLP samples, full four point MSA will be required if all of the following conditions are met:

1) recovery of the analyte in the matrix spike is not at least 50%,

2) the concentration of the analyte does not exceed the regulatory level, and,

3) the concentration of the analyte is within 20% of the regulatory level.

The reporting and regulatory limits for TCLP analyses as well as matrix spike levels are detailed in Table IV (Appendix A). Appendix C provides guidance on performing MSA analyses.

11.17. Guidelines are provided in the appendices on procedures to minimize contamination of samples and standards, preventive maintenance and troubleshooting.

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

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

12. DATA ANALYSIS AND CALCULATIONS

12.1. ICV percent recoveries are calculated according to the equation:

$$\%R = 100\left(\frac{Found(ICV)}{True(ICV)}\right)$$

12.2. CCV percent recoveries are calculated according to the equation:

$$\%R = 100\left(\frac{Found(CCV)}{True(CCV)}\right)$$

12.3. Analytical spike and matrix spike recoveries are calculated according to the following equation:

$$\% R = 100\left(\frac{SSR - SR}{SA}\right)$$

Where:
SSR = Spike Sample Result
SR = Sample Result
SA = Spike Added

NOTE: When the sample concentration is less than the IDL, use SR = 0 for purposes of calculating analytical spike recovery.

12.4. The relative percent difference (RPD) of matrix spike/matrix spike duplicates or sample duplicates are calculated according to the following equations:

$$RPD = 100\left[\frac{|MSD - MS|}{\left(\frac{MSD + MS}{2}\right)}\right]$$

Where:
MS = determined spiked sample concentration
MSD = determined matrix spike duplicate concentration

$$RPD = 100\left[\frac{|DU1 - DU2|}{\left(\frac{DU1 + DU2}{2}\right)}\right]$$

Where:
DU1 = Sample result
DU2 = Sample duplicate result

12.5. The final concentration for a digested aqueous sample is calculated as follows:

$$mg/L = \frac{C \times V1 \times D}{V2}$$

Where:

C = Concentration (mg/L) from instrument readout

D = Instrument dilution factor
V1 = Final volume in liters after sample preparation
V2 = Initial volume of sample digested in liters

12.6. The final concentration determined in digested solid samples when reported on a dry weight basis is calculated as follows:

$$mg\ /\ Kg, dry\ weight = \frac{C \times V \times D}{W \times S}$$

Where:
C = Concentration (mg/L) from instrument readout
D = Instrument dilution factor
V = Final volume in liters after sample preparation
W = Weight in Kg of wet sample digested
S = Percent solids/100

Note: A Percent Solids determination must be performed on a separate aliquot when dry weight concentrations are to be reported. If the results are to be reported on a wet weight basis, the "S" factor should be omitted from the above equation.

12.7. The LCS percent recovery is calculated according to the following equation:

$$\%R = 100\left(\frac{Found(LCS)}{True(LCS)}\right)$$

12.8. Appropriate factors must be applied to sample values if dilutions are performed.

12.9. Sample results should be reported with up to three significant figures in accordance with the Quanterra significant figure policy.

13. METHOD PERFORMANCE

13.1. Each laboratory must have initial demonstration of performance data on file for each analyte of interest as described in Section 9.0.

13.2. Refer to Tables I & IA in Appendix A for the list of routine as well as non-routine analytes that may be analyzed using this SOP.

13.3. Method performance is determined by the analysis of matrix spike and matrix spike duplicate samples as well as method blanks and laboratory control samples. The matrix spike recovery should fall within +/- 20 % and the matrix spike duplicates

should compare within 20% RPD. Method blanks must meet the criteria specified in Section 9.2. The laboratory control samples should recover within 20% of the true value until in house control limits are established.

13.4. Training Qualification:

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

14. POLLUTION PREVENTION

14.1. Standards and/or solutions are not approved for disposal to the sinks.

15. WASTE MANAGEMENT

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

15.2. Standards should be purchased and prepared in volumes consistent with laboratory use to minimize the volume of expired standards to be disposed.

15.3. All waste must be disposed of according to the facility hazardous waste management procedures, Attachment C of the Quanterra Inc., Chemical Hygiene Plan, Section WS002, Table 1.

15.4. Samples and other solutions containing high concentrations of toxic materials must be disposed of according to the facility hazardous waste management procedures, Attachment C of the Quanterra, Inc., Chemical Hygiene Plan, Section WS003, Disposal of Samples After Analysis.

16. REFERENCES

16.1. Test Methods for Evaluating Solid Waste , Physical/Chemical Methods, SW-846, 3rd Edition, Final Update I, July 1992. Methods 7000A, 7060A, 7740, 7421, 7841, 7041, 7131A, 7191, 7211 and 7761.

16.2. Methods for the Chemical Analysis of Water and Waste, 1983.

16.3. CFR Part 136, Table IB, 7-1-92.

16.4. CORP-MT-0004, Graphite Furnace Atomic Absorption Spectroscopy Method, 2XX.X - CLP-M, SOW ILMO3.0.

16.5. QA-003, Quanterra QC Program.

16.6. QA-004, Rounding and Significant Figures.

16.7. QA-005, Method Detection Limits.

16.8. LS-CAL-2002, STANDARDS AND QC SOLUTIONS PREPARATION-METALS.

16.9. SAC-QA-0004, MAINTENANCE AND CALIBRATION CHECK OF FIXED AND ADJUSTABLE VOLUME AUTOPIPETTORS.

17. MISCELLANEOUS (TABLES, APPENDICES, ETC. . .)

17.1. Modifications/Interpretations from reference method.

17.1.1. Modifications from both 7000A and 200 series methods.

17.1.1.1. The 200 series methods and Chapter 1 of SW846 specify the use of reagent water with a purity equivalent to ASTM Type II water. This SOP specifies the use of a Millipore DI system or equivalent to produce reagent water. This SOP requires that reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

17.1.1.2. The alternate run sequence presented in Section 11.15 is consistent with method requirements. Additional QC analyses were added to accommodate the CLP protocol requirements.

17.1.2. Modifications from Method 7000A.

17.1.2.1. Chapter 1 of SW-846 states that the method blank should not contain any analyte of interest at or above the MDL. This SOP states that the method blank must not contain any analyte of interest at or above the reporting limit. Common lab contaminants are allowed up to two times the reporting limit in the blank following consultation with the client.

17.1.2.2. The SW846 methods contain contradictory statements regarding the frequency of CCV analysis. Section 7.3.6 of Method 7000A

states a check standard should be run after approximately every 10 sample injections. Section 8.3 (Quality Control) states a check sample must be run every 10 samples. Since Quanterra defines an analytical sample as a sample with its corresponding interference test and the SW846 document contains discrepancies in the frequency requirements for CCV analysis, this SOP requires that a CCV be analyzed every 10 analytical samples or 20 burns, whichever is more frequent.

17.1.2.3. Section 1.2 of Method 7000A states "To ensure valid data with furnace techniques, the analyst must examine each matrix for interference effects and, if detected, treat them accordingly, using <u>either</u> successive dilution, matrix modification, or method of standard addition." Quanterra utilizes matrix modifiers for all GFAA analyses and has also elected to run all samples using single point MSA rather than spike one "representative" sample per matrix type. The serial dilution test is presented in Method 7000A as a means to determine if interferences are present. Since the Quanterra SOP proceeds directly to single point MSA on all samples the use of the serial dilution test is not incorporated in this SOP. Sample results are not biased corrected based on the single point MSA result unless this is specifically requested by the client.

17.1.2.4. The SW846 methods contain contradictory statements regarding the acid matrix for calibration standards. Section 5.7 of Method 7000A states that the calibration standards should be prepared using the same type of acid or combination of acids and at the same concentration as will result in the samples following processing. Section 5.0 in each of the 7000 series methods states differing specific concentrations of acids in each method (i.e., Method 7421 states a 0.5% HNO_3 matrix). Since the addition of nitric acid to samples acts as a modifier (i.e., improves the recovery of the analyte) and the digestion procedures used incorporate higher nitric acid concentrations than specified in the individual methods, this SOP requires a 2% HNO_3 matrix for all standards.

17.1.3. Modifications from 200 series methods.

17.1.3.1. Method 200.0 section 9.3.7 states that a check standard should be run after approximately every 10 sample injections. Method 200.0 section 10.2.2 states that the curve must be verified every 20 samples. Since Quanterra defines an analytical sample as a sample

with its corresponding interference test and Method 200.0 contains discrepancies in the frequency requirements for CCV analysis, this SOP requires that a CCV be analyzed every 10 analytical samples or 20 burns whichever is more frequent.

17.1.3.2. The 200 series methods contain contradictory statements regarding the number of calibration standards to be used for calibration. Section 8.3 of the introductory method states to "Prepare a blank and at least four calibration standards using the same type of acid or combination of acids and at the same concentration as will result in the samples following processing. Section 10.2.1 of the same document states that a calibration curve is composed of a minimum of a reagent blank and three standards. This SOP requires the use of a blank and three standards.

17.1.3.3. Each analyte specific 200 series method specifies matrix modifiers. EPA recognizes that there have been many improvements in matrix modifier technology since these methods were originally written. In order to minimize interferences due to sample matrix, Quanterra has adopted the use of a palladium matrix modifier and/or currently recommended manufacturer modifiers.

17.2. Modifications from previous SOP

Facility specific flags were identified for method blanks, and MSA reporting.

17.3. Facility Specific SOPs

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

17.4. Documentation and Record Management

The following documentation comprises a complete GFAA raw data package:

- Raw data (direct instrument printout signed by analyst)
- Relevant sample preparation benchsheets.

C4-312

C4-313

- Run log printout from instrument software where this option is available or manually generated run log. (A bench sheet may be substituted for the run log as long as it contains an accurate representation of the analytical sequence and sufficient date and time information to verify that the QC time criteria were met).

  Note: The Ward CVT benchsheet option is recommended as an efficient way to generate a benchsheet and run log.

- Data review checklist - See Appendix B

- Standards Documentation (including prep date, source and lot number).

17.5. Non-conformance summary (if applicable).

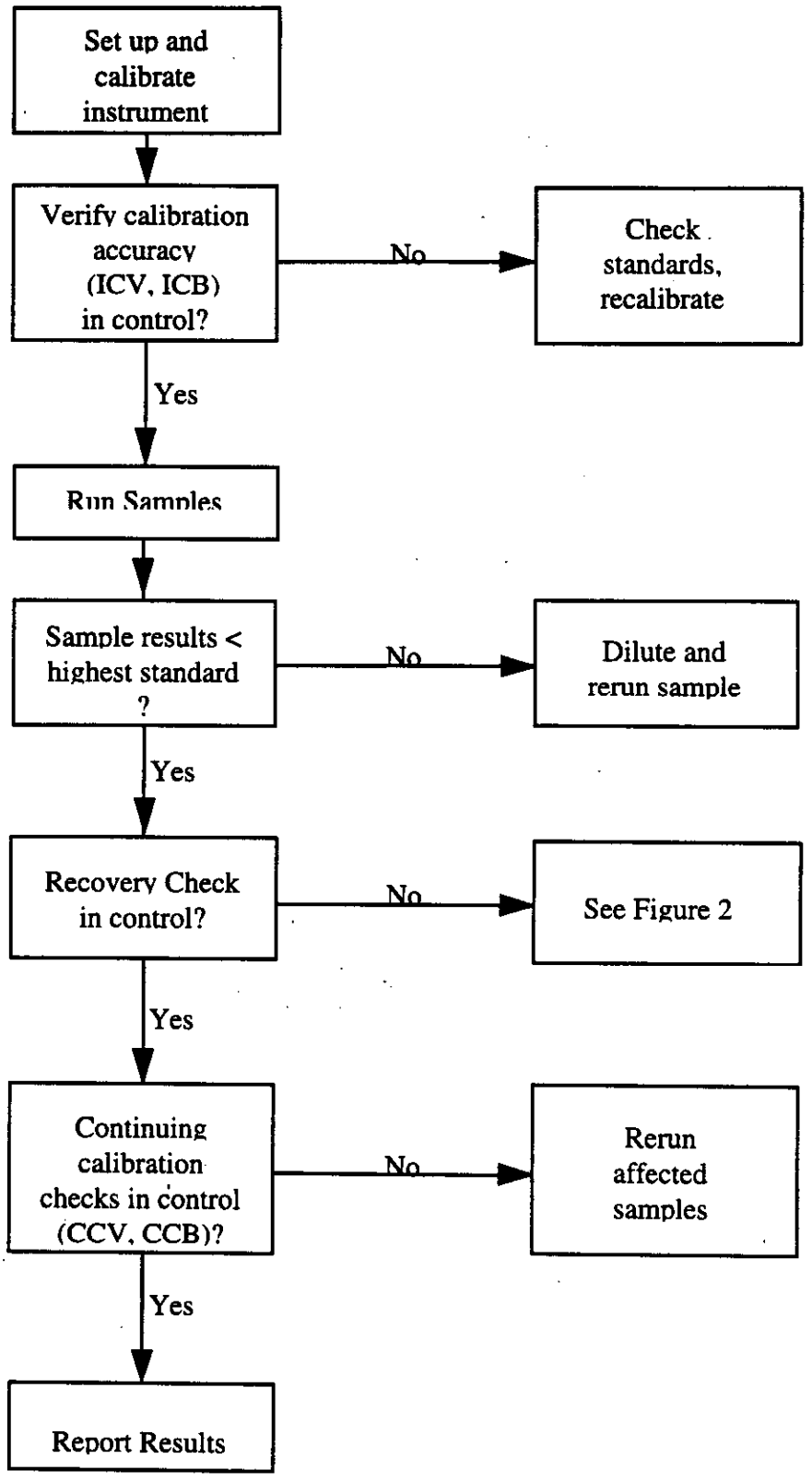


Figure 1. GFAA Analysis Flow Diagram

C4-315

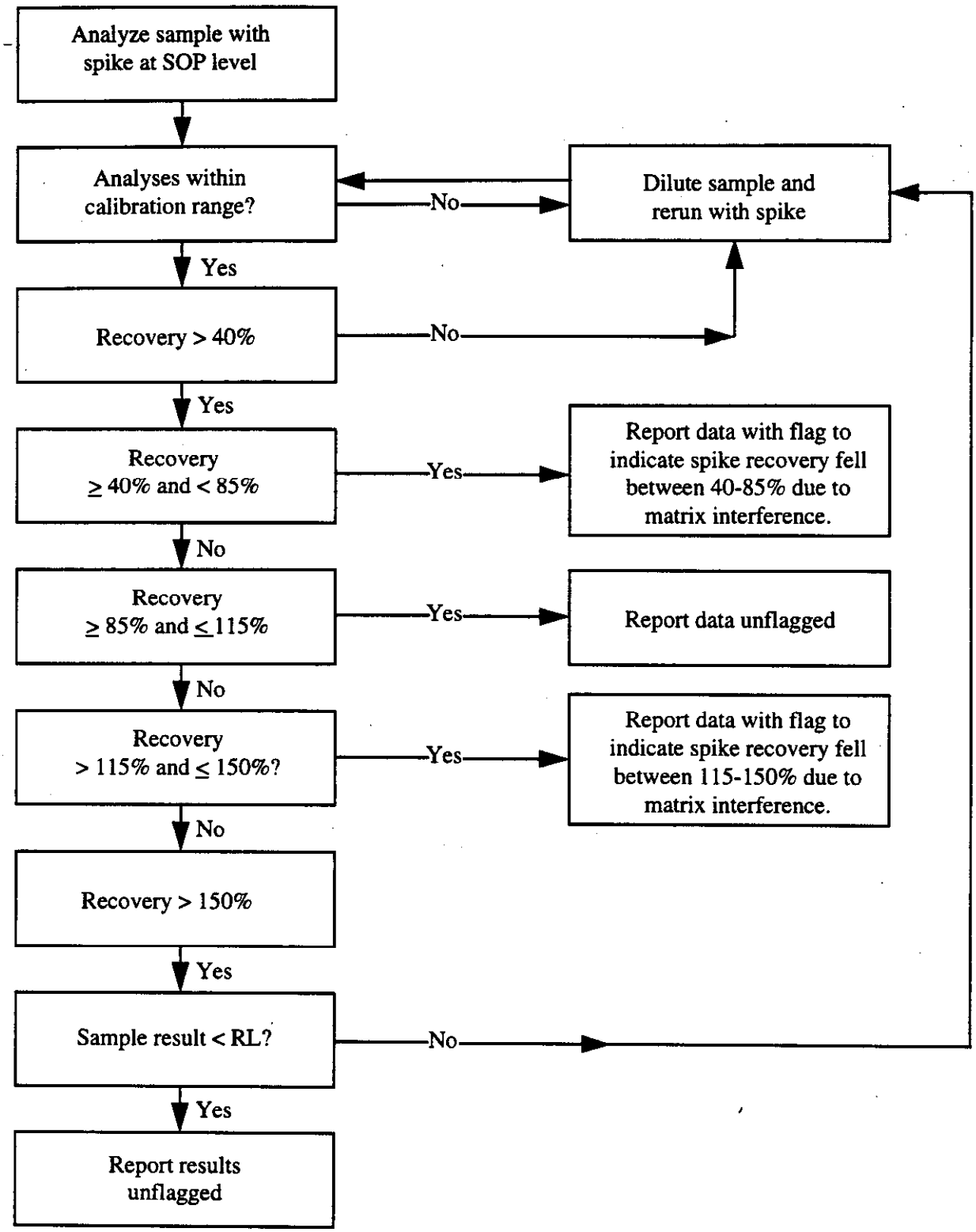


Figure 2. Recovery Check Protocol

C4-316

APPENDIX A

TABLES

TABLE I. 7000 and 200 Series Routine Analyte List

ELEMENT	Symbol	CAS #	SW-846 Method	MCAWW Method	Aqueous RL (ug/L)	Solid RL (mg/Kg)
Arsenic	As	7440-38-2	7060A	206.2	10	1.0
Selenium	Se	7782-49-2	7740	270.2	5	0.5
Lead	Pb	7439-92-1	7421	239.2	3	0.3
Thallium	Tl	7440-28-0	7841	279.2	10	1.0
Antimony	Sb	7440-36-0	7041	204.2	10	1.0
Cadmium	Cd	7440-43-9	7131A	213.2	1	0.1
Chromium	Cr	7440-47-3	7191	218.2	5	0.5
Copper	Cu	7440-50-8	7211	220.2	5	0.5
Silver	Ag	7440-22-4	7761	272.2	1	0.1
Nickel	Ni	7440-02-0	NA	249.2	5	0.5

TABLE IA. Non-routine GFAA Analytes *

ELEMENT	Symbol	CAS #	SW846 Method	MCAWW Method
Barium	Ba	7440-39-3	7081	208.2
Beryllium	Be	7440-41-7	7091	210.2
Cobalt	Co	7440-48-4	7201	219.2
Iron	Fe	7439-89-6	7381	236.2
Manganese	Mn	7439-96-5	7461	243.2
Molybdenum	Mo	7439-98-7	7481	246.2
Vanadium	V	7440-62-2	7911	286.2
Zinc **	Zn	7440-66-6	7951	289.2

Note: Elements are not available at all Quanterra Laboratories.

* Additional elements may be possible by this method. ICP is recommended for all elements listed in Table IA.

**Not recommended by this procedure due to background contamination levels.

NA - Not applicable.

TABLE II. Recommended GFAA Analytical Guidelines

Element	λ (nm)	Char Temp°C	Atom Temp°C	Tube
Arsenic	193.7	1200-1400	2000-2800	Py/Pl
Selenium	196.0	900-1400	1900-2700	Py/Pl
Lead	283.3	700-1100	1500-2300	Py/Pl
Thallium	276.8	400-1100	1400-2600	Py/Pl
Antimony	217.6	400-1400	1900-2800	Py/Pl
Cadmium	228.8	500-1200	1400-2300	Py/Pl
Chromium	357.9	1100-1650	2300-2800	Py/Pl
Copper	324.8	800-1300	1900-2500	Py/Pl
Silver	328.1	500-1000	1600-2600	Py/Pl
Nickel	232.0	900-1300	2100-2500	Py/Pl

*Py/Pl refers to a pyrolytically coated platform tube.

TABLE III . Matrix Spike, Analytical Spike and Laboratory Control Sample Levels (ug/L)

ELEMENT	LCS/ Matrix Spike	Analytical Spike
Arsenic	40	20
Selenium	40	20*
Lead	40	20
Thallium	40	20
Antimony	40	20
Cadmium	4	4
Chromium	10	10
Copper	20	10
Silver	5	5
Nickel	20	20

*If running in combined protocol mode (Section 11.15), a spike level of 10ppb is used to accommodate CLP criteria.

TABLE IV. TCLP Reporting Limits, Regulatory Limits and Matrix Spike Levels

ELEMENT	RL (ug/L)	Regulatory Limit (ug/L)	Spike Level (ug/L)
Arsenic	500	5000	5000
Barium	10000	100000	50000
Cadmium	100	1000	1000
Chromium	500	5000	5000
Lead	500	5000	5000
Selenium	250	1000	1000
Silver	500	5000	1000

TABLE V. Calibration and Calibration Verification Standards

ELEMENT	Std 1	Std 2	Std 3	ICV	CCV
Arsenic	10	40	80	30	40
Selenium	5	40	80	30	40
Lead	3	40	80	30	40
Thallium	10	30	60	20	30
Antimony	10	25	50	20	25
Cadmium	1	2.5	5	2	2.5
Chromium	5	15	25	10	15
Copper	5	20	40	10	20
Silver	1	5	10	4	5
Nickel	5	50	100	40	50

***SOP specified calibration levels must be used unless prevented by the instrument configuration or client specific requirements. Deviations from specified calibration levels must be documented in the facility specific instrument operation SOP and must be approved by the facility technical manager and Quality Assurance Manager.**

TABLE VI. Summary Of Quality Control Requirements

QC PARAMETER	FREQUENCY *	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
ICV	Beginning of every analytical run.	90 - 110 % recovery	Terminate analysis; Correct the problem; Recalibrate.
ICB	Beginning of every analytical run, immediately following the ICV.	The result must be within +/- RL from zero.	Terminate analysis; Correct the problem; Recalibrate.
CCV	Every 10 samples and at the end of the run.	Method 7000A: 80 - 120 % recovery. 200 series: 90 -110 % recovery.	Terminate analysis; Correct the problem; Recalibrate and rerun all samples not bracketed by acceptable CCV.
CCB	Immediately following each CCV.	The result must be within +/- RL from zero.	Terminate analysis; Correct the problem; Recalibrate and rerun all samples not bracketed by acceptable CCB.
Method Blank	One per sample preparation batch of up to 20 samples.	The result must be less than or equal to the RL. Common lab contaminants may be accepted up to 2x the RL after consultation with the client (See Section 9.2) Sample results greater than 20x the blank concentration are acceptable. Samples for which the contaminant is < RL do not require redigestion (See Section 9.2).	Redigest and reanalyze samples. Note exceptions under criteria section. See Section 9.2 for additional requirements.

*See sections 11.14 and 11.15 for exact run sequence to be followed.

TABLE VI. Summary of Quality Control Requirements (Continued)

QC PARAMETER	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Laboratory Control Sample (LCS)	One per sample preparation batch of up to 20 samples.	Aqueous LCS must be within 80 - 120% recovery or in-house control limits. Samples for which the contaminant is < RL and the LCS results are > 120% may not require redigestion or reanalysis (see Section 9.3)	Terminate analysis; Correct the problem; Redigest and reanalyze all samples associated with the LCS.
Matrix Spike	One per sample preparation batch of up to 20 samples.	80 - 120 % recovery or in-house control limits. If the MS/MSD is out for an analyte, it must be in control in the LCS.	In the absence of client specific requirements, flag the data; no flag required if the sample level is > 4x the spike added. For TCLP see Section 11.16
Matrix Spike Duplicate	See Matrix Spike	80 - 120 % recovery or in-house control limits; RPD ≤ 20%. (See MS)	See Corrective Action for Matrix Spike.
Analytical Spike	One per each sample analyzed including method blank.	85 - 115 % recovery.	See Section 9.7 (Spike Recovery Check).

APPENDIX B
QUANTERRA GFAA DATA REVIEW CHECKLIST

Quanterra GFAA Data Review Checklist

Run/Project Information

Run Date: _____ **Analyst:**_____ **Instrument:**_____
Prep Batches Run:_____

Circle Methods used: **7000A / 2XX.X : CORP-MT-0003 Rev 1**
CLP : CORP-MT-0004 Rev 1

Review Items

A. Calibration/Instrument Run QC	Yes	No	N/A	2nd Level
1. Instrument calibrated per manufacturer's instructions and at SOP specified levels ?				
2. ICV/CCV analyzed at appropriate frequency and within control limits?				
3. ICB/CCB analyzed at appropriate frequency and within +/- RL or +/- CRDL (CLP)?				
4. CRA run (CLP only)?				
B. Sample Results				
1. Were samples with concentrations > the high calibration standard diluted and reanalyzed?				
2. RSD of replicate burns < 20 % when sample results > RL ?				
3. All reported results bracketed by in control QC ?				
4. Sample analyses done within holding time?				
C. Preparation/Matrix QC				
1. LCS done per prep batch and within QC limits ?				
2. Method blank done per prep batch and < RL or CRDL (CLP) ?				
3. MS run at required frequency and within limits ?				
4. MSD or DU run at required frequency and RPD within SOP limits?				
5. Analytical spike within QC limits or appropriate action taken ?				
D. Other				
1. Are all nonconformances documented appropriately ?				
2. Current IDL/MDL data on file?				
3. Calculations and Transcriptions checked for error ?				
4. All client/ project specific requirements met?				
5. Date/time of analysis verified as correct ?				

Analyst: _____ **Date:**_____
Comments:

_____

_____

2nd Level Reviewer : _____ **Date:**_____

_____

_____

APPENDIX C

MSA GUIDANCE

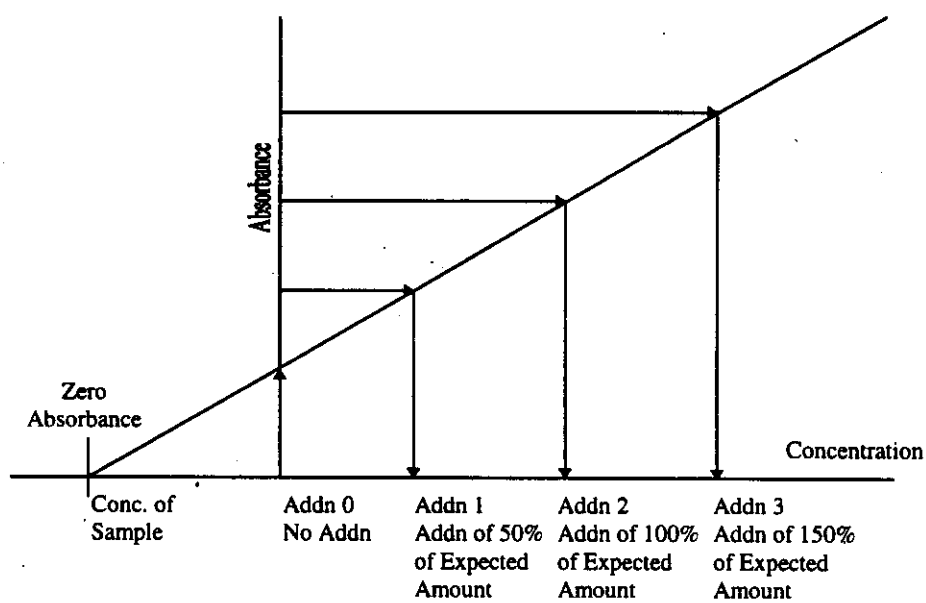
APPENDIX C. MSA GUIDANCE

Method of Standard Addition

Four equal volume aliquots of sample are measured and known amounts of standards are added to three aliquots. The fourth aliquot is the unknown and no standard is added to it. The concentration of standard added to the first aliquot should be 50% of the expected concentration. The concentration of standard added to the second aliquot should be 100% of the expected concentration and the concentration of standard added to the third aliquot should be 150% of the expected concentration. The volume of the unspiked and spiked standard should be the same.

In order to determine the concentration of analyte in the sample, the analytical value of each solution is determined and a plot or linear regression performed. On the vertical axis the analytical value is plotted versus the concentrations of the standards on the horizontal axis. An example plot is shown in Figure 1. When the resulting line is extrapolated back to zero absorbance, the point of interception of the horizontal axis is the concentration of the unknown.

Figure 1



- For the method of standard additions to be correctly applied, the following limitations must be taken into consideration.
- The plot of the sample and standards must be linear over the concentration range of concern. For best results, the slope of the curve should be similar to that of a plot of the aqueous standard curve.
- The effect of the interference should not vary as the ratio of the standard added to the sample matrix changes.

APPENDIX D

TROUBLESHOOTING GUIDE

APPENDIX D. TROUBLESHOOTING GUIDE

Problem	Possible Cause
Poor or No Absorbance or Sensitivity Check failed (See Section 11.4)	Read time too short Incorrect wavelength Low atomization temperature Char temperature too high Dry temperature too high No read step in program Dirty windows Window loose Etched or dirty optics Wrong lamp Bad lamp Bad graphite tube Bad contact rings Not enough or no sample injected Empty sample cup No modifier in sample Incorrectly made standards Poor injection into tube or on platform Flush solution empty Gas flow during read step Gas leak EDL power supply set on "Continuous"
Tube Impedance Error	Bad tube Bad contact rings Sample matrix Door not tight Argon supply out Cooling system malfunction

Problem	Possible Cause
Erratic Readings or High % RSD	Source lamp not aligned properly Lamp not prewarmed Injection tip partially clogged Rinse bottle empty Water cooling not on Bad lamp Injection tip hitting outside of tube Injection tip coated or not set properly Salt buildup on tip Leak in sample capillary Ruby valve clogged Wrong or no modifier Incomplete dry step Dry temperature too high Bad tube Bad contact rings Unstable power supply Air bubbles in syringe or cylinders(Varian)
EDL Won't Light	Lamp cable not plugged in Lamp power set at 0 Lamp is dead Power supply fuse is blown Short in cord Lamp driver faulty (EDL2)
Sample Not Drying or Crackling	Incorrect dry temperature Tube is placed incorrectly Tube is bad Platform not seated properly Furnace door not tight Sample matrix Bad contact rings
Standards reading twice normal absorbance or concentration	Incorrect standard used Incorrect dilution perform
Background Correction Light Blinking	Background screen or attenuator faulty D2 lamp and element lamp energy don't match Analyte lamp energy too low (bad lamp) Background screen or attenuator faulty
Preparation blank spike recovery outside recovery limits	Incorrect concentration of nitric acid HCl added to sample in prep lab Injection tip worn or clogged Sample pump worn Contaminated cup or spike solution

APPENDIX E

CONTAMINATION CONTROL GUIDELINES

APPENDIX E. CONTAMINATION CONTROL GUIDELINES

The following procedures are strongly recommended to prevent contamination:

All work areas used to prepare standards and spikes should be cleaned before and after each use.

All glassware should be washed with detergent and tap water and rinsed with 1:1 nitric acid followed by deionized water.

Proper laboratory housekeeping is essential in the reduction of contamination in the metals laboratory. All work areas must be kept scrupulously clean.

Powdered or Latex Gloves must not be used in the metals laboratory since the powder contains silica and zinc, as well as other metallic analytes. Only vinyl or nitrile gloves should be used in the metals laboratory.

Glassware should be periodically checked for cracks and etches and discarded if found. Etched glassware can cause cross contamination of any metallic analytes.

Autosampler trays should be covered to reduce the possibility of contamination. Trace levels of elements being analyzed in the samples can be easily contaminated by dust particles in the laboratory.

The following are helpful hints in the identification of the source of contaminants:

Yellow pipet tips and volumetric caps can sometimes contain cadmium.

Some sample cups have been found to contain lead.

The markings on glass beakers have been found to contain lead. If acid baths are in use for glassware cleaning, they should be periodically checked for contaminants since contaminant concentrations will increase over time.

The buildup of matrix modifiers in the furnace housing can cause cross contamination. For example: If nickel nitrate is used as a matrix modifier, the furnace housing should be thoroughly cleaned prior to the analysis of nickel.

New glassware, especially beakers, can be a source of silica and boron.

Reagents or standards can contain contaminants or be contaminated with the improper use of a pipette.

Improper cleaning of glassware can cause contamination.

Pyrolytic tubes can contain contaminants. If suspected, the tube should be cleaned by operating the furnace at full power as required to eliminate the contamination.

Latex gloves contain over 500 ppb of zinc.

APPENDIX F

PREVENTIVE MAINTENANCE

APPENDIX F. PREVENTIVE MAINTENANCE

A maintenance log is used to record when maintenance is performed on instruments. When an instrument problem occurs indicate the date, time and instrument number, then identify the problem and corrective action in the maintenance log.

The following procedures are required to ensure that that the instrument is fully operational.

Daily
- Clean contact rings and windows
- Check tube and platform, replace if corroded, flaking or sensitivity check fails
- Check gas lines and supply level
- Check rinse solution and fill if needed
- Check drain lines and waste containers, empty if needed
- Clean outside of capillary tip
- Adjust autosampler arm as needed
- Debubble syringe (Varian instruments only)
- Clean fume extraction tip, replace fume extraction filter and water trap (PE4100ZL)

Weekly
- Trim capillary tip as needed
- Check water level in coolflow
- Wipe exterior of instrument

Monthly
- Clean filters and radiators

Quarterly
- Change contact rings (if needed)
- Clean instrument water system

Annually
- Notify manufacturer service engineer to clean optics

To obtain the maximum graphite tube life the following guidelines should be followed.

1. Contact rings should be cleaned when replacing the graphite tube.
2. After insertion, every new tube should be thermally conditioned. It is recommended that for the first heating, a ramp time between 60 and 100 seconds and a temperature range from ambient temperature to 2650°C be applied. After a few seconds at 2650°C and a cool down of 20 seconds, the process should be repeated three times with a ramp time 10 seconds.
3. It is very important to long tube life to always use a ramp time for thermal pretreatment. No higher temperature increase than 50°C per second is recommended.

4. For optimal performance, no higher atomization temperatures should be used than 2650°C for pyrolytically coated tubes and 2700°C for uncoated tubes. For refractory elements, application of higher atomization temperature can initially increase the signal, but will drastically reduce the tube lifetime.

5. When using peak area, atomization time has to be sufficiently long for the signal to come back to the baseline. High atomization and heatout temperatures should be applied as long as required, and not a second longer.

6. To avoid a negative effect of acids on tube lifetime, always keep a check on acid concentrations, use a slow ramp for thermal pretreatment and use pyrolytically coated tubes as specified in Table II.

7. If the temperature of the GFAA cooling water is too low and the flowrate is too high, condensation from atmospheric humidity can occur around the contact rings. A cooling water flowrate of 2 L/min +/-0.5 L/min is specified by Perkin Elmer. The use of a coolflow is strongly recommended since it always provides the same water flow and temperature.

8. Nitrogen gas results in a lower peak sensitivity for several elements. It can also cause the generation of cyanogen above 2300°C, making it potentially dangerous in a poorly ventilated room. Perkin Elmer recommends the use of argon.

APPENDIX G

Footnotes and Qualifiers

The following are footnotes that are to be applied to data by the reviewer during data reduction and LIMS entry. Use these qualifiers in the absence of any other client-specific requirements.

Qualifier/Flag	Definition	Conditions for use and how applied
B	This analyte is also detected in the blank at a reportable level.	The flag is applied to each applicable analyte in each sample when: • analytes are detected above the reporting limit in both the blank and the samples or • laboratory contaminants are detected in the blank at a concentration less than 2X the reporting limit. See section 9.2 for specific acceptance procedures.
p	Reporting limit was raised when the sample was diluted to verify a suspected matrix interference.	Apply to the sample if: • the AS recovery is <40%, • the sample is diluted, and reanalyzed with another analytical spike. See section 9.7 of this SOP.
l	The post-digestion spike recovery fell between 115%-150% due to matrix interference.	Apply to the sample if: • the AS recovery is >115% and • the AS recovery is <150% and • the sample result is greater than the reporting limit.

APPENDIX H

FIRST AID PLAN/HYDROFLUORIC ACID

SANTA ANA - SPECIFIC

QUANTERRA STANDARD OPERATING PROCEDURE

TITLE: TOXICITY CHARACTERISTIC LEACHING PROCEDURE AND SYNTHETIC PRECIPITATION LEACHING PROCEDURE

(SUPERSEDES: NONE)

Prepared by: _____

Reviewed by: _____
Technical Specialist

Approved by: _____
Quality Assurance Manager

Approved by: _____
Environmental Health and Safety Coordinator

Approved by: _____
Laboratory Manager

Approved by: _____
Corporate Technology and/or Corporate QA

Proprietary Information Statement:

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C4-340

Table of Contents

1. SCOPE AND APPLICATION 3
2. SUMMARY OF METHOD 4
3. DEFINITIONS 5
4. INTERFERENCES 5
5. SAFETY 6
6. EQUIPMENT AND SUPPLIES 7
7. REAGENTS AND STANDARDS 8
8. SAMPLE COLLECTION, PRESERVATION AND STORAGE 10
TABLE 1 - HOLDING TIMES (DAYS) 12
9. QUALITY CONTROL 12
10. CALIBRATION AND STANDARDIZATION 13
11. PROCEDURE 14
11.1. GENERAL COMMENTS 14
11.2. PRELIMINARY EVALUATIONS 14
TABLE 2 - MINIMUM REQUIRED LEACHATE VOLUMES 14
11.3. PROCEDURE: NON-VOLATILE CONSTITUENTS: SEMI-VOLATILES, PESTICIDES, HERBICIDES, METALS (BOTTLE LEACH) 22
11.4. PROCEDURE: VOLATILE CONSTITUENTS (ZHE) 24
12. DATA ANALYSIS AND CALCULATIONS 30
13. METHOD PERFORMANCE 32
14. POLLUTION PREVENTION 32
15. WASTE MANAGEMENT 32
16. REFERENCES 33
17. MISCELLANEOUS (METHOD INTERPRETATIONS, RECORD MANAGEMENT, ETC...) 34

LIST OF APPENDICES

APPENDIX A - TABLES 36
APPENDIX B - FIGURES 38
APPENDIX C - WORKSHEETS 42
APPENDIX D - FLOW CHARTS 49

C4-341

1. SCOPE AND APPLICATION

1.1. This SOP describes the application of the Toxicity Characteristic Leaching Procedure (TCLP), SW846 Method 1311. The Toxicity Characteristic (TC) of a waste material is established by determining the levels of 8 metals and 31 organic chemicals in the aqueous leachate of a waste. The TC is one of four criteria in 40 CFR Part 261 to determine whether a solid waste is classified as a hazardous waste. The other three are corrosivity, reactivity and ignitability. The TC Rule utilizes the TCLP method to generate the leachate under controlled conditions which were designed to simulate leaching through a landfill. EPA's "worst case" waste disposal model assumes mismanaged wastes will be exposed to leaching by the acidic fluids generated in municipal landfills. The EPA's model also assumes the acid/base characteristics of the waste will be dominated by the landfill fluids. The TCLP procedure directs the testing laboratory to use a more acidic leaching fluid if the sample is an alkaline waste, again in keeping with the model's assumption that the acid fluids will dominate leaching chemistry over time.

1.2. The specific list of TC analytes and regulatory limits may be found in Appendix A.

Note: The list in Appendix A does not include the December 1994 EPA rule for Universal Treatment Standards for Land Disposal Restrictions. Those requirements include 216 specific metallic and organic compounds and, in some cases, lower detection limit requirements (see 40 CFR 268.40). TCLP leachates are part of the new Universal Treatment Standards, but the conventional analytical methods will not necessarily meet the new regulatory limits. Consult with the client and with Quanterra Technical Specialists before establishing the instrumental methods for these regulations.

1.3. This SOP also describes the application of the Synthetic Precipitation Leaching Procedure (SPLP) which was designed to simulate the leaching that would occur if a waste was disposed in a landfill and exposed only to percolating rain water. The procedure is based on SW846 Method 1312. The list of analytes for SPLP may extend beyond the toxicity characteristic compounds shown in Appendix A. With the exception of the use of a modified extraction fluid, the SPLP and TCLP protocols are essentially equivalent. Where slight differences may exist between the SPLP and TCLP they are distinguished within this SOP.

1.4. The procedure is applicable to liquid, solid, and multiphase wastes.

1.5. The results obtained are highly dependent on the pH of the extracting solution, the length of time that the sample is exposed to the extracting solution, the temperature

during extraction, and the particle size/surface area of the sample. These parameters must be carefully controlled.

1.6. The reporting limits are based on the individual samples as well as the individual analysis techniques. However, the sample is determined to be hazardous if it contains any analyte at levels greater than or equal to the regulatory limits.

1.7. If a total analysis of the waste demonstrates that individual analytes are not present in the waste, or that they are present but at such low concentrations that the appropriate regulatory levels could not possibly be exceeded, the procedure need not be run. If the total analysis results indicate that TCLP is not required, the decision to cease TCLP analysis should be remanded to the client.

1.8. If an analysis of any one of the liquid fractions of the procedure leachate indicates that a regulated compound is present at such a high concentration that, even after accounting for dilution from the other fractions of the leachate, the concentration would be equal to or above the regulatory level for that compound, then the waste is hazardous and it may not be necessary to analyze the remaining fractions of the leachate. However, the remaining analyses should not be terminated without the approval of the client.

1.9. Volatile organic analysis of the leachate obtained using a bottle extraction, normally used for extractable organics and metals, can be used to demonstrate that a waste is hazardous, but only the ZHE option can be used to demonstrate that the concentration of volatile organic compounds is below regulatory limits.

2. SUMMARY OF METHOD

2.1. For liquid wastes that contain less than 0.5% dry solid material, the waste, after filtration through 0.6 to 0.8 µm glass fiber filter, is defined as the TCLP leachate.

2.2. For wastes containing greater than or equal to 0.5% solids, the liquid, if any, is separated from the solids and stored for later analysis. The particle size of the remaining solid phase is reduced, if necessary. The solid phase is extracted with an amount of extraction fluid equal to 20 times the weight of the solid phase. For TCLP, the extraction fluid employed for extraction of non-volatile analytes is a function of the alkalinity of the solid phase of the waste. For SPLP, the extraction fluid employed is a function of the region of the country where the sample site is located if the sample is a soil. If the sample is a waste or wastewater the extraction fluid employed is a pH 4.2 solution. Two leachates may be generated: a) one for analysis of non-volatile constituents (semi-volatile organics, pesticides, herbicides and metals and/or b) one from a Zero Headspace Extractor (ZHE) for analysis of volatile organic constituents.

Following extraction, the liquid leachate is separated from the solid phase by filtration through a 0.6 to 0.8 µm fiber filter.

2.3. If compatible (i.e., multiple phases will not form on combination), the initial liquid phase of the waste is added to the liquid leachate and these are prepared and analyzed together. If incompatible, the liquids are analyzed separately and the results are mathematically combined to yield a volume-weighted average concentration.

3. DEFINITIONS

3.1. "Leachate" is used to refer to the TCLP solution generated from this procedure.

3.2. "Percent Wet Solids" is that fraction of a waste sample (as a percentage of the total sample) from which no liquid may be forced out by an applied pressure.

4. INTERFERENCES

4.1. Oily wastes may present unusual filtration and drying problems. As recommended by EPA (see Figure 3), oily wastes will be assumed to be 100% liquid and analysis for total concentrations of contaminants will be performed. This applies specifically to samples containing viscous non-aqueous liquids that would be difficult to filter.

4.2. Wastes containing free organic liquids (i.e., those with separable non-aqueous liquid phases) will be assumed to be 100% liquid and totals analysis will be performed to determine if the oil exceeds TCLP limits.

4.3. Solvents, reagents, glassware and other sample processing hardware may yield artifacts and/or interferences to sample analysis. All these materials must be demonstrated to be free from interferences under the conditions of the analysis by analyzing method blanks as described in the Section 9.0 and the individual determinative SOPs.

4.4. Glassware and equipment contamination may result in analyte degradation. Soap residue on glassware and equipment may contribute to this. All glassware and equipment should be rinsed very carefully to avoid this problem.

4.5. Phthalates may be eliminated by proper glassware cleanup and by avoiding plastics. Only glass, Teflon or Type 316 stainless steel tumblers may be used for leachates to be analyzed for organics. Plastic tumblers may be used for leachates to be analyzed for the metals.

4.6. Overexposure of the sample to the environment will result in the loss of volatile components.

4.7. Potential interferences that may be encountered during analysis are discussed in the individual analytical methods.

5. SAFETY

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

5.2. Eye protection that satisfies ANSI Z87.1 (as per the Chemical Hygiene Plan), laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.3. The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the Material Safety Data Sheets (MSDS) maintained in the laboratory. The following specific hazards are known:

5.3.1. Chemicals that have been classified as **carcinogens**, or **potential carcinogens**, under OSHA include:

Methylene chloride

5.3.2. Chemicals known to be **flammable** are:

Methanol

5.3.3. The following materials are known to be **corrosive**:

Hydrochloric acid, nitric acid, sulfuric acid, acetic acid, sodium hydroxide

5.3.4. The following materials are known to be **oxidizing agents**:

Nitric Acid.

5.4. Gas pressurized equipment is employed in this procedure. Be sure all valves and gauges are operating properly and that none of the equipment, especially tubing, is

over-pressurized. CAUTION: Do not open equipment that has been pressurized until it has returned to ambient pressure.

5.5. A rotary agitation apparatus is used in this procedure. Certain samples may break the glass jars used in the procedure. For these samples, extra caution, including plastic or polyethylene overwraps of the glass jar, may be necessary.

5.6. Secure tumbler and extraction apparatus before starting rotary agitation apparatus.

5.7. During sample rotation, pressure may build up inside the bottle. Periodic venting of the bottle will relieve pressure.

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

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

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

5.11. Due to the potential for ignition and/or flammability, do not attempt to dry non-aqueous liquid samples in an oven.

6. EQUIPMENT AND SUPPLIES

6.1. Extraction vessels

6.1.1. For volatile analytes - zero-headspace extraction (ZHE) vessel, gas-pressure actuated, Millipore YT3009OHW or equivalent (see Figure 2).

6.1.2. For metals - either borosilicate glass jars (1/2 - 1 gallons, with Teflon lid inserts) or 1 L HDPE (Nalgene or equivalent) bottles may be used.

6.1.3. For non-volatile organics - only borosilicate glass may be used.

6.2. Vacuum filtration apparatus, capable of 0 - 50 psi and stainless steel pressure filtration apparatus (142 mm diameter), capable of 0 - 50 psi.

6.3. Borosilicate glass fiber filters, 0.6 - 0.8 µm (Whatman GF/F 14.2 cm, 0.7 µm or equivalent). When analyzing for metals, wash the filters with 1 N nitric acid and de-ionized water prior to use. Glass fiber filters are fragile and should be handled with care.

6.4. Rotary agitation apparatus, multiple-vessel, Associated Design and Manufacturing Company 3740-6 or equivalent (see Figure 1). The apparatus must be capable of rotating the extraction vessel in an end-over-end fashion at 30 ± 2 rpm.

6.5. ZHE Extract Collection Devices are used to collect the initial liquid phase and the final extract of the waste from the ZHE device, either of the following may be used:

6.5.1. Gas-tight syringes, 100 mL capacity, Hamilton 0158330 or equivalent, or

6.5.2. Tedlar bags

6.6. Top loading balance, capable of 0 - 4000 ± 0.01g (all measurements are to be within ± 0.1 grams).

6.7. pH meter and probe capable of reading to the nearest 0.01 unit, and with automatic temperature compensation.

6.8. pH probes.

6.9. Magnetic stirrer/hotplate and stirring bars.

6.10. VOA vials, 20 mL, with caps and septa.

6.11. Glass jars, 1/2 - 1 gallon, with Teflon lid-inserts.

6.12. Nalgene plastic bottles, 1 liter.

6.13. Miscellaneous laboratory glassware and equipment.

7. REAGENTS AND STANDARDS

7.1. Reagent water for non-volatile constituents must be produced by a Millipore DI system or equivalent. For volatile constituents, water must be passed through an activated carbon filter bed (Milli-Q or tap water passed through activated carbon). Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

7.2. Hydrochloric acid, 1 N: Carefully add 83 mL concentrated reagent grade HCl to 800 mL reagent water, cool and dilute to 1 liter with reagent water. Cap and shake to mix well.

7.3. Nitric acid, 1 N: Carefully add 64 mL concentrated reagent grade HNO_3 to 800 mL of reagent water, cool and dilute to 1 liter with reagent water. Cap and shake to mix well.

7.4. Sodium hydroxide, 1 N: Carefully add 40 g reagent grade NaOH pellets to 800 mL reagent water, stir until the pellets are completely dissolved, cool and dilute to 1 liter with reagent water.

CAUTION: Heat is generated during this process.

7.5. Acetic acid, glacial: concentrated, reagent grade liquid (HOAc).

7.6. pH calibration solutions: buffered to a pH of 4, 7, and 10. Commercially available. Fresh buffer solution must be used each day of analysis.

7.7. TCLP Leaching Fluids

7.7.1. General Comments

7.7.1.1. The pH of both solutions listed below should be monitored daily *and/or prior to use* and the pH probes are to be calibrated prior to use.

7.7.1.2. The leaching fluids MUST be prepared correctly. If the desired pH range is not achieved and maintained, the TCLP may yield erroneous results due to improper leaching. If the pH is not within the specifications, the fluid must be discarded and fresh extraction fluid prepared.

7.7.1.3. Additional volumes of extraction fluids listed above may be prepared by multiplying the amounts of acetic acid and NaOH by the number of liters of extraction fluid required.

7.7.2. TCLP Fluid #1: Carefully add 5.7 mL glacial acetic acid and 64.3 mL of 1 N NaOH to 500 mL reagent water in a 1 liter volumetric flask. Dilute to a final volume of 1 L with reagent water, cap and shake to mix well. When correctly prepared, the pH of this solution is 4.93 ± 0.05.

7.7.3. TCLP Fluid #2: Carefully add 5.7 mL glacial acetic acid to 500 mL reagent water in a 1 liter volumetric flask. Dilute to a final volume of 1 L with reagent water, cap and shake to mix well. When correctly prepared, the pH of this solution is 2.88 ± 0.05.

7.8. Nitric acid, 50% solution: Slowly and carefully add 500 mL concentrated HNO_3 to 500 mL reagent water. Cap and shake to mix well.

7.9. Sulfuric acid / nitric acid (60/40 weight percent mixture) H_2SO_4/HNO_3. Cautiously mix 60 g of concentrated sulfuric acid with 40 g of concentrated nitric acid.

7.10. SPLP Leaching fluids

7.10.1. SPLP solutions are unbuffered and exact pH may not be attained. The pH of TCLP and SPLP fluids should be checked prior to use. If not within specifications, the fluid should be discarded and fresh fluid prepared.

7.10.2. SPLP fluid #1: Add 60/40 weight percent mixture of sulfuric and nitric acids to reagent water until the pH is 4.20 ± 0.05 This fluid is used for soils from a site that is east of the Mississippi River and for wastes and wastewaters.

7.10.3. SPLP fluid #2: Add 60/40 weight percent mixture of sulfuric and nitric acids to reagent water until the pH is 5.00 ± 0.05. This fluid is used for soils from a site that is west of the Mississippi River.

7.10.4. SPLP fluid #3: This fluid is reagent water and is used for leaching of volatiles. Additionally, any cyanide-containing waste or soil is leached with fluid #3 because leaching of cyanide containing samples under acidic conditions may result in the formation of hydrogen cyanide gas.

7.11. Methanol and methylene chloride - used to aid in cleaning oil contaminated equipment.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Samples being analyzed for non-volatile organic compounds should be collected and stored in glass containers with Teflon lid liners. Chemical preservatives shall NOT be added UNTIL AFTER leachate generation.

8.2. Samples being analyzed for metals only can be collected in either glass or polyethylene containers.

8.3. When the waste is to be evaluated for volatile analytes, care should be taken to minimize the loss of volatiles. Samples shall be collected and stored in a manner intended to prevent the loss of volatile analytes (e.g., samples should be collected in Teflon lined septum capped vials with minimal headspace and stored at 4 ± 2 °C). Samples should be opened only immediately prior to extraction.

8.4. Samples should be refrigerated to 4 ± 2 °C unless refrigeration results in irreversible physical changes to the waste. If precipitation occurs, the entire sample (including precipitate) should be extracted.

8.5. The minimum TCLP sample collection size is determined by the physical state or states of the waste and the analytes of concern. The amount of waste required varies with the percent solids. The lower the percent solids, the more waste will be required for preliminary and final testing. For aqueous samples containing between 0.5 and 10% solids, several kilograms of sample are required to complete the analyses. The general minimal requirements when the samples are 100% solids include: 1 - 32 oz jar for semi-volatile organic analysis and metals, and 1 - 4 oz jar for volatile organic analysis. Low density sample materials, such as rags or vegetation, will require larger volumes of sample. For liquid samples (less than *0.5%* solids), minimum requirements are 2 - 32 oz jars for semi-volatile organic analysis and metals, and 2 - 8 oz jars for volatile organic analysis. If volatile organic analysis is the only requested parameter, 2 separate jars are required. If matrix spike or duplicate control samples are requested, additional sample volume is required. If sufficient sample volumes were not received, analyses cannot be started and the client should be notified as soon as possible.

8.6. TCLP leachates should be prepared for analysis and analyzed as soon as possible following extraction. Leachates or portions of leachates for metallic analyte determinations must be acidified with nitric acid to a pH less than 2, unless precipitation occurs. If precipitation occurs upon addition of nitric acid to a small aliquot of the leachate, then the remaining portion of the leachate shall not be acidified and the leachate shall be analyzed as soon as possible. All other leachates should be stored under refrigeration (4 ± 2 °C) until analyzed. ZHE leachates must be stored in VOA vials filled to eliminate all headspace.

8.7. Samples are subject to appropriate treatment within the following time periods:

Table 1 - Holding Times (days)

Parameter	Collection to TCLP Leach	TCLP Leach to Preparation	Preparation to Analysis	Total Elapsed Time
Volatiles:	14	N/A	14	28
Semi-volatiles:	14	7	40	61
Mercury:	28	N/A	28	56
Other Metals:	180	N/A	180	360

NOTE: The initial holding time is measured from date of collection to date extraction started, and subsequent holding times are measured from the date extraction starts. If sample holding times are exceeded, the values obtained will be considered minimal concentrations. Exceeding holding times is not acceptable in establishing that a waste does not exceed the regulatory level. Exceeding the holding time will not invalidate characterization if the waste exceeds the regulatory limit.

9. QUALITY CONTROL

9.1. Quality Control Batch (QC Batch) - QA-003 defines a QC Batch as a set of up to 20 field samples of similar matrix that behave similarly and are processed using the same procedures, reagents and standards within the same time period. The same lot of reagents must be used within a batch. A minimum of one TCLP extraction blank (Method Blank), one Laboratory Control Sample (LCS), one Matrix Spike (MS), and one Matrix Spike Duplicate (MSD) will be prepared with each TCLP leachate batch.

9.2. Batching Samples - Groups of samples with visibly different bulk matrices (e.g., petroleum sludge and soil samples) must be batched separately for QC testing purposes.

9.3. TCLP Extraction Blanks - A minimum of one blank (using the same extraction fluid as used for the samples) must be prepared and analyzed for every batch of samples extracted in a particular vessel type. The blanks are generated in the same way as the samples (i.e:, blanks will be tumbled and filtered with the samples). Extraction vessels will be uniquely numbered. Each time a new batch is set up the blank should be rotated sequentially to the next vessel to ensure all vessels are periodically checked. Consult the Quanterra QC Program and the individual analysis SOPs for blank acceptance criteria.

9.4. Laboratory Control Sample (LCS) - A LCS is required with each batch of 20 or fewer samples. The LCS shall be generated after a batch of TCLP leachates have been generated (i.e., at the time of the preparative digestion or extraction) by spiking an

aliquot of the appropriate extraction fluid used for that batch. Consult the individual analysis SOPs for additional LCS guidance (i.e., spike amounts, spike levels, recovery criteria, etc.).

9.5. Matrix Spike (MS/MSD) - Matrix spikes are used to monitor the performance of the analytical methods on the matrix and to assess the presence of interferences. A MS/MSD pair are required with each batch of 20 or fewer samples.

9.5.1. Matrix spikes are to be added after filtration of the TCLP leachate. Spikes are not to be added prior to the TCLP leaching. For metals, matrix spikes are to be added before preservation with nitric acid.

9.5.2. The use of internal calibration or alternate methods may be needed when the recovery of the matrix spike is below the expected performance (see Section 9.6.2).

9.5.3. Consult the individual analysis SOPs for additional guidance on spike compounds and levels.

9.6. Corrective Actions

9.6.1. Consult the Quanterra QC Program and individual analysis SOPs for corrective action for blanks and LCS

9.6.2. Method of Standard Additions (MSA) shall be used for metals if all of the following conditions are met:

- Recovery of the analyte in matrix spike is not at least 50%,
- the concentration of the analyte does not exceed the regulatory level, and
- the concentration of the analyte measured in the sample is within 20% of the appropriate regulatory level.

If the matrix spike recovery is 5% or less due to dilution or matrix interference, contact the project manager and client for guidance. The client should also be contacted prior to initiation of any MSA steps. Refer to the individual analysis SOPs for details on how to perform MSA analysis.

10. CALIBRATION AND STANDARDIZATION

10.1. Refer to appropriate analysis SOPs.

11. PROCEDURE

11.1. GENERAL COMMENTS

11.1.1. One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented on a Nonconformance Memo kept in the project file and described in the final report. The variation must be approved by a project manager, Technical Specialist and QA Manager. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

11.2. PRELIMINARY SAMPLE EVALUATIONS (Refer to Flow Chart #1, Appendix D)

11.2.1. Preliminary TCLP evaluations (percent solids, particle size, selection of extraction fluid, and fluid/leachate compatibility) are required to be done using a minimum of a 100 gram aliquot of waste. This aliquot may also undergo the actual TCLP or SPLP extraction for Non-volatiles ONLY IF it has NOT been oven dried. If the solid portion is oven dried, a separate aliquot must be used for the actual leaching procedure.

11.2.2. Consult the holding times for the appropriate tests (Section 8.7) and prioritize extractions such that holding times are not exceeded.

11.2.3. Determine the total volume of TCLP leachate (solid phase leachate + liquid filtrate) that needs to be generated for analysis according to the following:

Table 2. Minimum Required Leachate Volume

Analysis	Required Volume (mL)
Volatiles	3 x 20
Semi-volatiles	400
Pesticides	200
Herbicides	200
Metals	300

11.2.3.1. SPLP - similar volumes are *required like TCLP* volatiles and metals.

11.2.3.2. For TCLP and SPLP samples used for matrix spike and matrix spike duplicate analysis, three times the listed volumes are required.

11.2.4. **Sample Description** (enter data on Worksheet 1)

11.2.4.1. Record the number of phases observed in the sample. It is common that when more than one container of multi-phasic materials is received from the field, each container will show different amounts of each phase.

11.2.4.1.1. If the sample has multiple phases and is received in more than 1 bottle then the contents of each bottle should be combined in a single larger container prior to processing the sample further. If this is not possible, then the alternate procedure described in the following section should be used.

11.2.4.1.2. Properly record the relative amounts of each phase by measuring the depth of the layers in each container after the contents have been allowed to settle. Determine the combined volume of each phase for all containers. Then mark the phase composition on a single container, mix thoroughly to obtain a representative subsample, and accurately measure the phase composition according to the following procedure. The two sets of values (combined volumes per phase and phase composition for one container) are used to determine the correct volume/mass adjustments on the final result. This procedure is not appropriate if testing will be done for volatile organic compounds.

11.2.4.2. Solid - record the visible presence of a solid material heavier than water. If the sample contains more than one solid phase (e.g., wood and sediment mixed with water), *document comment on benchsheet.*

11.2.4.3. Liquid - record the number of liquid phases observed in the sample according to apparent density. It may be impossible to distinguish apparent density if only one liquid phase is observed and there is no indication on the COC form. If this is the case,

record it as aqueous material and let the subsequent analytical record show if the liquid is organic.

11.2.5. **Percent Solid Phase** (enter data on Worksheet No. 1)

11.2.5.1. Percent Solids and ZHE Extractions - The ZHE filtration apparatus cannot accurately determine percent solids less than 5%. If an extraction is to be performed solely for volatile organic compounds and the percent solids concentration is apparently greater than 5%, proceed to Section 11.4 (Procedure: ZHE Extraction Procedure, Volatile Constituents). Otherwise, continue with the steps in this section. The aliquot of sample used here cannot be used again for the ZHE extraction.

11.2.5.2. Determine Type of Filtration Apparatus Needed

11.2.5.2.1. If the waste will obviously yield no free liquid when subjected to pressure filtration (i.e., it is 100% solid), then proceed to Section 11.2.6 (Particle-size Reduction).

11.2.5.2.2. If the sample is mostly a non-viscous liquid (water or non-viscous organic liquid) of low solids content (<10%) or a highly granular, liquid containing waste vacuum filtration may be used.

11.2.5.2.3. If the sample is viscous (sludge or has high solids content), use pressure filtration.

11.2.5.3. Weight of subsample and filtrate for percent solids measurement

11.2.5.3.1. Assemble the filtration apparatus (use blunt forceps to handle the 0.6 to 0.8 µm filter membrane).

11.2.5.3.2. Homogenize the waste, transfer a minimum of a 100 g subsample to the weighing vessel. Measure and record the gross weight.

11.2.5.3.3. Measure and record the tare weight of the filtration vessel.

11.2.5.3.4. Transfer the sample to the filtration device attempting to spread the waste sample evenly over the surface of the filter. Measure and record the tare weight of the empty weighing vessel and any residual sample.

11.2.5.3.5. Calculate and record the net weight of sample used for testing.

11.2.5.4. Filtration for percent solids

11.2.5.4.1. Slowly apply gentle pressure or vacuum of 10 psi to the filtration apparatus. Allow the sample to filter until no SIGNIFICANT additional liquid has passed through the filter during a 2 minute period.

11.2.5.4.2. Repeat previous step by increasing the pressure in 10 psi increments until a maximum of 50 psi is reached. Stop the filtration when no additional filtrate is generated within a 2 minute period.

Note: Some samples will contain liquid material that does not filter (e.g., oil). Do not attempt to filter the sample again by exchanging filters. Viscous oils or any wastes which does not pass through the filter is classified as a solid.

11.2.5.4.3. Remove the filtrate collection vessel, weigh and record the gross weight.

11.2.5.4.4. Calculate and record the net weight of filtrate This result will be used in the percent solids calculation.

11.2.5.4.5. Pour the filtrate into a graduated cylinder. Measure and record the volume of the aqueous phase. Measure and record the volume of any organic phase. If more than one organic phase is observed, enter "see below" and provide a description at the bottom of *benchsheet.* These results will be used in the final sample calculations.

11.2.5.4.6. Retain the filtrate for use in Section 11.2.8 (Determination of Filtrate/Extraction Fluid

Compatibility), and for possible recombination with the filtrate obtained in Section 11.3.

11.2.5.5. Percent of Wet Solids

11.2.5.5.1. Calculate the total weight of wet solids using Equation: $E = D\text{-}B$.

11.2.5.5.2. Calculate the weight percent of wet solids using Equation: $\% E = \frac{E}{C} x\ 100$.

11.2.5.5.3. If the percent wet solids result is ≥ 0.5% and < 5.0%, and it is noticed that a small amount of the aqueous filtrate is entrained in the wetting of the filter, proceed to Section 11.2.5.7 to complete the percent solids measurement on a dry-weight basis.

Note: If obviously oily (non-aqueous) material is entrained on the filter, do not dry the filter; proceed to Section 11.2.6 (Particle-Size Reduction).

11.2.5.5.4. If the percent wet solids result is greater than 5.0%, proceed to Section 11.2.6 (Particle-Size Reduction) and mark "≥0.5%" on *bench sheet.*

11.2.5.5.5. If the percent wet solids result is less than 0.5%, discard the solid phase. No leaching will be necessary; the filtrate is equivalent to the final leachate.

11.2.5.6. Weight percent of dry solids (skip this step for oily samples).

Note: These steps are required only if it is noticed that a small amount of the filtrate is entrained in wetting of the filter <u>and</u> the percent wet solids content is ≥ 0.5% and < 5.0 %.

11.2.5.6.1. Remove the filter with the wet solids from the filtration apparatus.

11.2.5.6.2. Dry the filter and solid phase at 100 ± 20 ° C.

11.2.5.6.3. Remove the filter from the oven and allow to cool in a desiccator.

11.2.5.6.4. Weigh and record the gross dry weight.

11.2.5.6.5. Repeat the drying step. Weigh and record the second gross dry weight. If the two weightings do not agree within 1%, perform additional drying and weighing until successive weighings agree within 1%.

11.2.5.6.6. Calculate the weight percent of dry solids using:

$$\textit{Weight Percent Dry Solids} = \frac{\textit{Gross Dry Weight}}{\textit{Weight of Sample}} \times 100$$

11.2.5.6.7. If the dry solids result is ≥ 0.5% and the sample will be extracted for non-volatile constituents, proceed to Section 11.2.6 (Particle Size Reduction) using a fresh wet portion of waste.

11.2.5.6.8. If the percent solids result is less than 0.5%, discard the solid phase. No leaching will be necessary; the filtrate is the TCLP leachate. Proceed to Section 11.2.8 (Determination of Filtrate/Leachate Compatibility) to determine whether or not the material is a non-aqueous, immiscible liquid.

11.2.6. **Particle-size Reduction for Fluid Selection** (enter data on Worksheet 2)

11.2.6.1. The subsample used for fluid selection must consist of particles less than 1 mm in diameter (versus the less than 1 cm requirement for the material used for the actual extraction). The method requires a smaller particle size to partially compensate for the shorter duration of contact time with the leachate solution as compared to the full extraction. Inappropriate use of coarser materials could result in the selection of the wrong fluid type.

11.2.6.2. Surface area exclusion - size reduction is not required if the sample surface area is greater than or equal to 3.1 cm^2 per gram.

11.2.6.3. If the sample contains particles greater than 1 mm in diameter, crush, cut, or grind the solids to the required size.

11.2.6.4. Consult your supervisor or manager when dealing with unusual sample matrices (e.g., wood, cloth, metal, brick).

11.2.7. **Determination of Appropriate Extraction Fluid** (Worksheet 2)

11.2.7.1. If the solid content is greater than or equal to 0.5%, and if the sample is being analyzed for metals or nonvolatile organic compounds, the type of leaching solution must be determined.

11.2.7.2. Follow times, temperature, and particle size specified in this section as closely as possible. If reaction time between the acid solution and solid waste is too short or too long, the procedure may produce false pH readings.

11.2.7.3. For SPLP, refer to Section 7.10 for fluid selection. Matrix type must be specified by the client. Check special instructions or see the project manager, then put a check mark by the fluid type selected.

11.2.7.4. The TCLP leaching fluid for all volatiles is Fluid #1.

11.2.7.5. For TCLP leach fluid determination for non-volatile analytes, continue with the following steps.

11.2.7.6. Calibrate the pH meter with fresh buffer solution in accordance with the pH SOP.

11.2.7.7. Weigh out a 5.0 ± 0.1 g subsample (less than 1 mm particle size) of the solid phase into a 250-mL beaker. If 5.0 grams not used, enter the actual weight on line C2.

11.2.7.8. Add 96.5 ± 1.0 mL of reagent water, cover with a watchglass, and stir for 5 minutes on a stirrer. If a different volume used, enter the actual volume.

11.2.7.9. Measure and record the sample pH.

Note: To avoid damaging the pH probe when organic liquid is present, use narrow range pH indicator paper.

11.2.7.10. If the pH is less than or equal to 5.0, use Fluid #1 and proceed to Section 11.2.8 (Fluid Compatibility).

11.2.7.11. If the fluid pH is greater than 5.0, add 3.5 mL 1 N HCl, cover with a watchglass. Slurry the sample briefly then heat at 50 °C for 10 minutes.

Note: The heating cycle is a critical step. If the solid waste does not remain in contact with the acidic solution under specified time and temperature conditions, an erroneous pH may be measured.

11.2.7.12. Cool to room temperature.

11.2.7.13. Measure and record the pH immediately after the sample has reached room temperature.

11.2.7.13.1. If the pH is less than or equal to 5.0, use Fluid #1.

11.2.7.13.2. If the pH is greater than 5.0, use Fluid #2.

11.2.8. **Determination of Filtrate/Extraction Fluid Compatibility** (skip this step for SPLP extractions)

11.2.8.1. Place 5 mL of the appropriate leaching fluid (determined in the previous step) into a 20-25 mL vial.

Note: Use fluid type # 1 if simply testing the filtrate for a sample with less than 0.5% solids.

11.2.8.2. Add 5 mL of the initial filtrate, cap and shake.

11.2.8.3. If the phases are miscible, the initial filtrate and solid phase leachate will be physically recombined upon completion of the leachate generation.

11.2.8.4. If the phases are NOT miscible, document on bench sheet. The initial filtrate and the solid phase leachate will be prepared and analyzed separately and the results mathematically combined (see Section 12.1.4).

11.2.9. For samples requiring analysis for semi-volatile organics, pesticides, herbicides or metals proceed to Section 11.3.

11.2.10. For samples requiring analysis for volatile organics (ZHE), proceed to Section 11.4.

11.3. **BOTTLE EXTRACTION PROCEDURE: NON-VOLATILE CONSTITUENTS: SEMI-VOLATILES, PESTICIDES, HERBICIDES, METALS** (Refer to Flow Chart #2, Appendix D)

11.3.1. All masses should be recorded to the nearest 0.1 g.

11.3.2. The aliquot used in the Preliminary Evaluation MAY be used for this procedure ONLY if it was not oven dried. If the sample is 100% solid or if the preliminary aliquot was not oven dried proceed directly to Section 11.3.7 (Particle Size Reduction). If the Preliminary Evaluation aliquot was oven dried then, using a fresh aliquot of sample, continue as described in Sections 11.3.3 through 11.3.6.

11.3.3. Examine the sample and determine the type of filtration to employ per Section 11.2.5.2.

11.3.4. Repeat the steps outlined in Sections 11.2.5.3 through 11.2.5.5.3.

11.3.5. Determine and record the volume (mass) of the initial filtrate. Cover with aluminum foil and retain for use as defined in Section 11.3.18.

11.3.6. Determine and record the "solid" phase mass by subtracting the mass of the liquid filtrate from the mass of the subsample.

11.3.7. Evaluate the solid portion of the waste for particle size. If it contains particles greater than 1 cm in size, prepare the solid portion of the waste for leaching by crushing, cutting, or grinding such that all particles are less than 1 cm in size (i.e., capable of passing through a 9.5 mm, 0.375 inch, standard sieve). Size reduction is not required if the sample surface area is greater than or equal to 3.1 cm^2 per gram. If particle size reduction was required, record this on Worksheet 3, line E1.

11.3.7.1. Consult your supervisor or manager when dealing with unusual sample matrices (e.g., wood, cloth, metal, brick). Scissors or shears may be used to cut cloth, plastic or sheet metal. Saws may be used for wood or solid metal. Bricks, rocks, or other solids amenable to grinding should be subcontracted out for particle size reduction (Contact PA or PM). Note that size reduction to

fine powder is not appropriate, and could invalidate results. If necessary, consult client for guidance.

11.3.8. Determine the minimum total volume of solid phase leachate that needs to be generated. Refer to Section 11.2.3.

11.3.9. Divide the total volume of solid phase leachate required by 20 to determine the mass of solid phase required for leaching. Round this mass UP to the nearest 5 g.

11.3.10. Weigh the required mass of solid phase into an appropriate bottle (plastic for metals only, glass for all others) and **slowly** add 20 times its mass of appropriate leaching fluid as determined under Section 11.2.7 (e.g., 20 g of sample would require 400 g of leaching fluid). Record the weight of the sample aliquoted for the extraction and the amount of extraction fluid added.

11.3.11. Ensure any effervescence has stopped before capping the bottle tightly. Secure in a rotary agitator and rotate end-over-end at 28-32 rpm for 16-20 hours. The temperature of the room should be 23 ± 2°C. The room temperature and time should be checked at both the start and end of the extraction and recorded.

NOTE: As agitation continues, pressure may build up within the bottle for some types of wastes. To relieve excessive pressure, the bottle may be removed and opened periodically in a properly vented hood to relieve any built-up pressure.

11.3.12. Remove the bottle and filter the sample using vacuum or pressure filtration by filtering through a new glass fiber filter as discussed in Sections 11.2.5.5.1 - 11.2.5.5.2. For final filtration of the TCLP leachate, the glass fiber filter may be changed, if necessary, to facilitate filtration. Filters must be acid washed if metals are to be determined (see Section 6.3). The entire sample need not be filtered; however, sufficient volume should be generated to support the required analyses. Record the date and time the filtration is completed.

11.3.13. If the waste contained no initial filtrate, this solution from 11.3.12 is defined as the TCLP leachate.

11.3.14. If the waste did yield an initial filtrate, consult the worksheet for initial filtrate/leachate compatibility. If they are compatible, they are to be

combined in the correct proportions (see Section 12.1.4) and mixed well. This combined solution is defined as the TCLP leachate.

11.3.15. If the individual phases are NOT compatible, they are to be prepared and analyzed separately and the results combined mathematically. See Section 12.1.5.

11.3.16. Measure and record the pH of the TCLP leachate. (Do not attempt to measure the pH of oily samples as the probe may be rendered inoperable.)

11.3.17. Prepare subsamples for metals for MS/MSD quality control testing using the appropriate TCLP spiking solution (do not spike for organics). Refer to the appropriate determinative SOPs for further guidance on the spike components, levels and action criteria.

11.3.18. Immediately preserve the leachate as follows:

Metals	pH < 2 w/50% HNO_3 for non-oils (do not acidify oils)
All others	Refrigerate to 4 ± 2 °C

Note: Refer to Section 8.6 if precipitation occurs upon preservation.

11.3.19. Label each sample with the appropriate information and submit to the appropriate analytical groups for prep and analysis with copies of the TCLP preparation worksheets.

11.4. **ZHE EXTRACTION PROCEDURE: VOLATILE CONSTITUENTS** (Refer to Flow Chart #3, Appendix D)

11.4.1. Use the ZHE device to obtain a TCLP leachate for analysis of volatile compounds only. Leachate resulting from the use of the ZHE shall NOT be used to evaluate the mobility of non-volatile analytes (e.g., metals, pesticides, etc...).

11.4.2. Due to some shortcomings of the method, losses of volatile compounds may occur. Extra care should be observed during the ZHE procedure to ensure that such losses are minimized. Charge the ZHE with sample only once and do not open the device until the final extract has been collected. Do not allow the waste, the initial liquid phase or the extract to be exposed to the atmosphere any longer than necessary.

11.4.3. If the TCLP extraction is for volatile components only, refer to Section 11.2.5.1 before proceeding.

11.4.4. All masses should be recorded to the nearest 0.1 g.

11.4.5. Assemble the ZHE apparatus. Test for leakage by closing all valves except the gas inlet/outlet valve and pressurizing to 50 psi. Allow to stand for 15 minutes and check the pressure on the built-in gauge to make sure it is not leaking. If the pressure is NOT 50 psi, consult your supervisor.

11.4.6. Adjust the ZHE piston in the ZHE body to the appropriate height (slightly moisten the O-rings with leaching fluid if necessary).

11.4.7. Consult the worksheet and examine the sample. If the sample appears to be different from the preliminary information found on the worksheet, consult your supervisor.

11.4.8. If the preliminary evaluations indicated the need for particle size reduction, homogenize the waste, weigh out a sufficient size subsample and prepare for leaching by crushing, cutting, or grinding such that all particles are less than 1 cm in size as measured with a ruler (Do NOT sieve the sample). Size reduction is not required if the sample surface area is greater than or equal to 3.1 cm^2 per gram.

Note: To minimize loss of volatiles, samples for volatiles that require particle size reduction should be kept in sample storage (at 4 °C) until immediately before size reduction. Aggressive reduction which would generate heat should be avoided and exposure of the waste to the atmosphere should be avoided to the extent possible. Size reduction to a fine powder is not appropriate. Also see Section 11.3.11.

11.4.8.1. Consult your supervisor or manager when dealing with unusual sample matrices (e.g., wood, cloth, metal, brick). Scissors or shears may be used to cut cloth, plastic or sheet metal. Saws may be used for wood or solid metal. Bricks, rocks, or other solids amenable to grinding should be subcontracted out for particle size reduction (Contact PA or PM).

11.4.9. Place the ZHE apparatus on the balance and tare the balance.

11.4.10.

11.4.11. Determine the appropriate size subsample to weigh using the percent solids information from Section 11.2.5 and record the weight.

11.4.11.1. For wastes that are 100% solids, a 25 g sample is used.

11.4.11.2. For wastes containing < 0.5% solids the liquid portion of the waste, after filtration, is defined as the TCLP leachate. Filter enough of the sample to support all of the volatile analyses required.

11.4.11.3. For wastes containing ≥ 0.5% and < 5.0% solids, a 500 g subsample of waste is recommended.

11.4.11.4. If the sample has ≥ 5.0% solids, the appropriate sample size should be determined using the equation in Section 12.1.2.

Note: For wastes containing greater than 0.5% wet or dry solids (Section 11.2.5), the "solids" value from the ZHE filtration process may be used to determine the volume of fluid to load into the ZHE. This approach is recommended since the solids value from Section 11.2.5 may differ from the filtration solids due to sample variability or differences in the filtration apparatus.

11.4.12. Homogenize and transfer an appropriate size subsample of the waste into the ZHE and record the mass.

11.4.13. Carefully place the glass fiber filter between the support screens and secure to the ZHE. Tighten all the fittings.

11.4.14. Place the ZHE in a vertical position; open both the gas AND liquid inlet/outlet valves. Attach a gas line to the gas inlet/outlet valve.

11.4.15. If the waste is 100% solid, slowly increase the pressure to a maximum of 50 psi to force out as much headspace as possible and proceed to Section 11.4.18.

11.4.16. If the waste is < 100% solids, carefully apply gentle pressure of 10 psi (or more, if necessary) to force all headspace slowly out of the ZHE. At the FIRST appearance of liquid from the liquid inlet/outlet valve, quickly close the valve and discontinue gas pressure.

11.4.17. Assemble a syringe and place the plunger in all the way. Adjust the tension on the plunger to provide slight drag. Attach the pre-weighed syringe or Tedlar bag to the liquid inlet/outlet valve and open the valve. Record the tare weight of the collection device.

11.4.18. Carefully apply gas pressure of no more than 10 psi to force out the liquid phase. Allow the sample to filter until no SIGNIFICANT additional filtrate has passed in a 2 minute period.

Note: If the capacity of the syringe is reached, close the liquid inlet/outlet valve, discontinue gas pressure, remove the syringe and return to Section 11.4.15.

11.4.19. Repeat previous step increasing the pressure in 10 PSI increments until 50 psi is reached and no significant liquid has passed in a 2 minute period. Remove the collection device and record the total weight of the collection device with filtrate on Worksheet 4, Line 3a. Close the valve and discontinue gas pressure. Transfer the filtrate to VOA vials and label appropriately. Calculate the weight of filtrate collected.

Notes: If the original waste contained less than 0.5% solids (Section 11.2.5), this filtrate is defined as the TCLP leachate and you may proceed to Section 11.4.28. Otherwise, save the vials by storing at 4 C under minimal headspace conditions, for recombination as in Section 11.4.27.

The material remaining in the ZHE is defined to be the "solid" phase. Calculate the weight of the solid phase using Equation 4.

11.4.20. Based on the information from Sections 11.2.5 and 11.4.11 and using the formula in 12.1.3, determine the weight of fluid to load into the ZHE on the "solid" phase. The ZHE device has approximately a 500-mL capacity. Based on the need to add an amount of extraction fluid equal to 20 times the mass of the "solid" phase, the ZHE can therefore accommodate a maximum of 25 grams of "solid".

Note: The TCLP ZHE prep uses only TCLP fluid #1; the SPLP ZHE prep uses only SPLP fluid #1*(#3 at Santa Ana).*

11.4.21. Load the fluid transfer reservoir with an excess of Fluid #1 and preflush the transfer line to eliminate air pockets. Be sure the required volume remains.

11.4.22. Attach the transfer line to the liquid inlet/outlet valve and open the valve. Carefully pump the required volume into the ZHE and close the valve. Disconnect the transfer line.

11.4.23. Check the ZHE to make sure all the valves are closed and manually rotate the ZHE (end-over-end) 2 or 3 times. Reposition the ZHE in the vertical position.

11.4.24. Pressurize the ZHE to 5-10 psi. Allow to stand for 10 minutes, and then recheck the pressure. If the ZHE appears to be leaking, follow the corrective action protocols recommended by the manufacturer and repeat the analysis.

11.4.25. Slowly open the liquid inlet/outlet valve to bleed out any headspace that may have been introduced during the introduction of the Fluid. Upon the first sign of liquid from the valve, close the valve.

11.4.26. Repressurize the ZHE to 5-10 psi and place in the rotary agitator. Rotate at 28-32 rpm for 16-20 hours. Room temperature should be 23 ± 2 °C. The room temperature and time should be checked at both the start and end of the extraction and recorded.

11.4.27. Confirm that the pressure of 5-10 psi was maintained throughout the leaching. If it was NOT maintained, return to Section 11.4.1 and repeat the leachate with a new aliquot of sample.

11.4.28. Attach a syringe or Tedlar bag and open the liquid inlet/outlet valve to collect the aqueous leachate and proceed as outlined in 11.4.19 - 11.4.20. Record the volume/mass of the leachate and any oil. Record the date and time the filtration is completed.

Notes: If the waste contained an initial liquid phase, the liquid may be filtered directly into the same collection device holding the initial liquid phase of the waste.

A separate filtrate collection container must be used if combination would create multiple phases or there is not enough volume left within the filtrate collection container.

11.4.29. If the waste contained an initial filtrate (Section 11.4.18) that is miscible with the solid phase leachate (as determined in Section 11.2.8), the solid phase leachate and the initial filtrate are directly recombined in the correct

proportions (see Section 12.1.4). If the individual phases are NOT compatible, they are to be collected, prepped and analyzed separately.

Note: Chill the filtrate and receiving vessels before recombining.

11.4.30. Following collection, store the TCLP leachate in 3 20-mL VOA vials with minimal headspace at 4 ± 2 °C and prepare for analysis as soon as possible using the appropriate organic extraction procedure (see Section 16.3).

11.4.31. If the individual phases are analyzed separately, combine the results mathematically by using the recombination calculation in Section 12.1.5.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Calculations

12.1.1. Calculation of Percent Wet Solids:

$$Percent\ Wet\ Solids = 100\left(\frac{Mass,"solid"\ phase}{Mass, initial\ subsample}\right)$$

12.1.2. Calculation of weight of waste to charge to ZHE:

$$Weight\ of\ waste\ to\ charge\ to\ ZHE = 100\left(\frac{25}{\%\ wet\ solids}\right)$$

12.1.3. Calculation of weight of extraction fluid to use:

$$Weight\ of\ Extraction\ fluid = \frac{20\ x\ \%\ wet\ solids\ x\ weight\ of\ waste\ to\ be\ extracted}{100}$$

12.1.4. Calculation of volume of initial filtrate phase to recombine with solid phase leachate:

$$\text{Volume of filtrate for recombination} = \left(\frac{Weight\ of\ solids\ leached}{Total\ weight\ of\ solids}\right)\left(\frac{Leachate\ recovered}{Fluid\ added}\right)\left(Volume\ of\ initial\ aqueous\ filtrate\right)$$

12.1.5. Mathematical recombination of analytical results:

$$Final\ Analyte\ Concentration = \frac{(V_1 \times C_1) + (V_2 \times C_2)}{V_1 + V_2}$$

V_1 = total volume of the initial filtrate phase (L).
C_1 = analyte concentration in initial filtrate phase (mg/L).
V_2 = volume of the theoretical solid phase leachate (L).
C_2 = analyte concentration in solid phase leachate (mg/L).,

12.2. REPORTING REQUIREMENTS

12.2.1. Follow these reporting conventions for multi-phase samples:

12.2.1.1. If both phases have positive results, use the values from each phase to calculate the recombined result. Use the reporting limit for each phase to calculate the recombined reporting limit.

12.2.1.2. If both phases are "ND," not detected, the recombined result is "ND," and the reporting limit is calculated from the reporting limit for each phase.

12.2.1.3. If one phase is "ND" and the other phase has a positive result, use the reporting limit for the "ND" phase and the positive value for the other phase to calculate the combined result. The combined reporting limit is based on the reporting limit for both phases. If the combined result is less than the combined reporting limit, then supply a footnote to indicate that "a positive result was detected below the calculated detection limit."

12.2.2. Units - regardless of the nature of the sample, all TCLP and SPLP results are reported in units of mg/L.

12.2.3. For limits and significant figures, consult the appropriate analytical methods (Section 16.3).

12.2.4. Anomalies - all anomalies observed during the leach procedure must be noted on the worksheet or an anomaly form. Some examples of such anomalies are:

12.2.4.1. Sample was monolithic - subsample was obtained by crushing, cutting, grinding, sawing, etc.

12.2.4.2. Insufficient sample - less than the required 100 g minimum was available.

12.2.4.3. Multiple phases - "X" phases were present.

12.2.4.4. Sample was oil - single phase.

12.2.4.5. Sample contained liquid which did not filter under test conditions.

12.3. REVIEW REQUIREMENTS

12.3.1. Review all applicable holding times. If a holding time was exceeded, confirm that a holding time violation form was properly documented and routed.

12.3.2. If Total analysis results are available, those results may be compared with the TCLP analysis results according to the following:

$$Total \geq 20 \times TCLP$$

NOTE: Assumes the sample is 100% Solids.

12.3.3. Total constituent analysis results can be used to demonstrate the TCLP protocol is unnecessary. In performing a TCLP analysis, there is a 20:1 dilution of the original sample with the leaching solution. Thus, if the "total constituent" result is less than 20 times the TC level, it is impossible for the leachate to "fail" and the TCLP does not need to be performed. For example, the TC level for lead is 5.0 mg/L (ppm). Therefore, if a sample of lead-contaminated soil contains less than 100 ppm total lead, a TCLP test need not be run for lead.

13. METHOD PERFORMANCE

13.1. Refer to individual analysis SOPs.

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

14. POLLUTION PREVENTION

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

15. WASTE MANAGEMENT

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

16. REFERENCES

16.1. Method 1311, Toxicity Characteristic Leaching Procedure, Revision 0, July 1992, SW-846 Final Update I.

16.2. Method 1312, Synthetic Precipitation Leaching Procedure, Revision 0, November 1992, SW-846 Proposed Update II.

16.3. Related Documents

16.3.1. Toxicity Characteristic: Corrections to Final Rule. Method 1311, Federal Register, Vol. 55, No. 126, Friday, June 29, 1990.

16.3.2. Toxicity Characteristic: Final Rule. Method 1311, Federal Register, Vol. 55, No. 61, Thursday, March 29, 1990.

16.3.3. Technical Background Document and Response To Comments, Method 1311, Toxicity Characteristic Leaching Procedure, USEPA/OSW, April, 1989.

16.3.4. QA-003, Quanterra QC Program

16.3.5. CORP-IP-0003: Acid Digestion of Aqueous Samples by SW846 and MCAWW 200 Series Methods.

16.3.6. CORP-MT-0001: Inductively Coupled Plasma-Atomic Emission Spectroscopy, Spectrometric Method for Trace Element Analysis, Method 6010A and Method 200.7.

16.3.7. CORP-MT-0003: Graphite Furnace Atomic Absorption Spectroscopy, SW846 Method 7000A and MCAWW 200 Series Methods.

16.3.8. CORP-MT-0005: Preparation and Analysis of Mercury in Aqueous Samples by Cold Vapor Atomic Absorption, SW-846 7470A and MCAWW 245.1.

16.3.9. CORP-IP-0003: Acid Digestion of Aqueous Samples by SW846 and MCAWW 200 Series Methods.

16.3.10. CORP-MS-0002: Determination of Volatile Organics by GC/MS based on Methods 8240B and 8260A.

16.3.11. CORP-MS-0001 : GC/MS Analysis Based on Method 8270B, SW846.

16.3.12. CORP-GC-0001: Gas Chromatographic Analysis Based on Methods 8000A, 8010B, 8020A, 8080A and 8150B, SW846.

16.3.13. CORP-OP-0001: Extraction and Cleanup of Organic Compounds from Waters and Soils, Based on SW846 3500 Series, 3600 Series, 8150 and 600 Series Methods.

17. MISCELLANEOUS

17.1. Modifications/Interpretations from Reference Methods

17.1.1. Section 8: Preliminary Evaluations. Section 7.1 of the source method states that the sample aliquot used for the preliminary evaluation "...may not actually undergo TCLP extraction." Section 7.1.5 of the source method indicates that the portion used for the preliminary evaluation may be used for either the ZHE or non-volatile extraction if the sample was 100% solid. Section 7.1.5 further indicates that if the sample was subjected to filtration (i.e., < 100% solid) that this aliquot may be used for the non-volatile extraction procedure only as long as sufficient sample is available (minimum 100 g). Samples which have been subjected to the oven drying step may not be used for TCLP extraction because solid phase degradation may result upon heating.

17.1.2. Section 11.2.5.6.3: Percent Solids Determination. Section 7.1.2 of the source method indicates that "if the percent wet solids is ≥ 0.5% and it is noticed that a small amount of the filtrate is entrained in wetting of the filter" that the filter should be oven dried to determine percent dry solids ". Drying of oil or organic matrices can both be hazardous and inappropriate. Additionally, it may be impossible to achieve a constant weight when performing this step. Due to safety concerns, if obviously oily or heavy organic matrices are entrained on the filter, the filter is not oven dried.

17.1.3. Section 11.2.8: Preliminary Determination of Filtrate/Extraction Fluid Compatibility. Section 7.2.13 of the source method provides no guidance as to how to make this determination. As a result, the procedure herein was developed and incorporated into the Preliminary Determinations section.

17.1.4. Section 9.2: TCLP Extraction Blanks. Section 8.1 of the source method states that a minimum of one blank for every 20 extractions "...that have been conducted in an extraction vessel." Quanterra has interpreted this to mean one blank per twenty samples leached per TYPE of leaching vessel (i.e., Bottle or ZHE) per leach fluid used.

17.1.5. Section 11.2.7.9: Determination of Appropriate Extraction Fluid. Method 1311 does not address the appropriate approach to take if the pH equals 5.0. This SOP requires that Fluid #1 must be used if the pH is less than or equal to 5.0.

17.1.6. Section 9.4: QA/QC - Matrix Spikes. Section 8.2 of the source method states "A matrix spike shall be performed for each waste type..." and "A minimum of one matrix spike must be analyzed for each analytical batch." Further, Section 8.2.3 of the source method also states "The purpose of the matrix spike is to monitor the performance of the analytical methods used, and to determine whether matrix interferences exist." The standard Quanterra QAPP is designed to address the performance monitoring of analytical methodology through the LCS program. A minimum of one MS and MSD will be prepared for each TCLP leachate batch. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, the MS/MSD results have immediate bearing only on the specific sample spiked and not all samples in the batch.

17.1.7. Section 8.2.2 of the source method states that "In most cases, matrix spikes should be added at a concentration equivalent to the corresponding regulatory level." The method also states "If the analyte concentration is less than one half the regulatory level, the spike concentration may be as low as one half of the analyte concentration but may not be less than five times the method detection limit". For several analytes, spiking at the regulatory level is inappropriate to the range of analysis afforded by the determinative methods. Due to the wide range in these levels, Quanterra spikes at the levels specified in the determinative SOPs.

17.2. Modifications from Previous SOP

None

17.3. Documentation and Record Management

The following documentation comprises a complete TCLP preparation raw data package:

- Completed worksheets (Appendix C)
- Non-conformance summary (if applicable).
- Anomaly documentation (if applicable).

APPENDIX A

TABLES

Table 3 - Toxicity Characteristic Analytes and Regulatory Levels (Final Rule)

Contaminant	mg/L
Arsenic	5.0
Barium	100.0
Benzene	0.5
Cadmium	1.0
Carbon tetrachloride	0.5
Chlordane	0.03
Chlorobenzene	100.0
Chloroform	6.0
Chromium	5.0
Total Cresols (used if isomers not resolved)	200.0
2,4-D	10.0
1,4-Dichlorobenzene	7.5
1,2-Dichloroethane	0.5
2,4-Dinitrotoluene	0.13
1,1-Dichloroethylene	0.7
Endrin	0.02
Heptachlor (& epoxide)	0.008
Hexachlorobenzene	0.13
Hexachlorobutadiene	0.5
Hexachloroethane	3.0
Lead	5.0
Lindane	0.4
Mercury	0.2
Methoxychlor	10.0
Methyl ethyl ketone	200.0
Nitrobenzene	2.0
Pentachlorophenol	100.0
Pyridine	5.0
Selenium	1.0
Silver	5.0
Tetrachloroethylene	0.7
Toxaphene	0.5
Trichloroethylene	0.5
2,4,5-Trichlorophenol	400.0
2,4,6-Trichlorophenol	2.0
2,4,5-TP (Silvex)	1.0
Vinyl chloride	0.2

APPENDIX B

FIGURES

Figure 1 & 2 - Rotary Agitation Apparatus and Zero Headspace Extraction Vessel (ZHE)

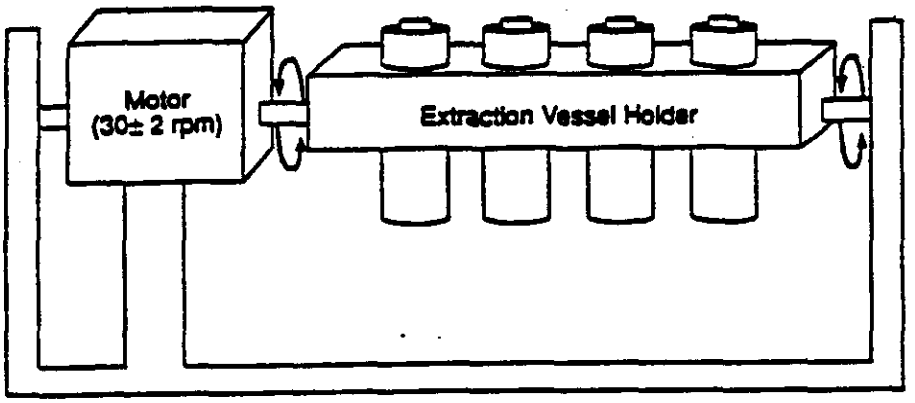

Figure 1. Rotary Agitation Apparatus

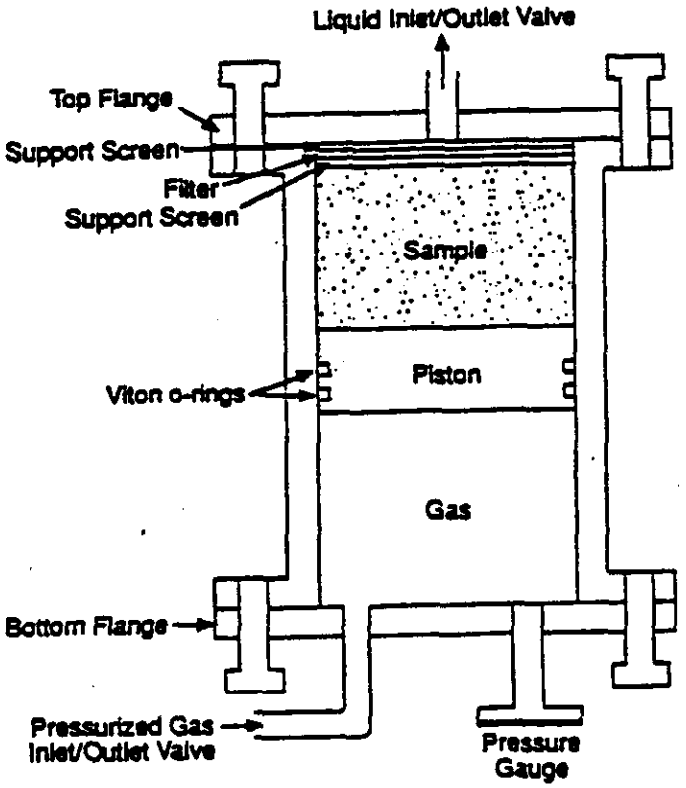

Figure 3 - US Environmental Protection Agency Memorandum #35, Page 1

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
WASHINGTON, D.C. 20460

OFFICE OF
SOLID WASTE AND EMERGENCY RESPONSE

MEMORANDUM # 35

DATE: June 12, 1992

SUBJECT: Notes on RCRA Methods and QA Activities

From: Gail Hansen, Chief Gail Hansen
Methods Section (OS-331)

This memo addresses the following topics:

- 1992 Symposium on Waste Testing and Quality Assurance
- SW-846 Update
  - Final Rule for January 23, 1989 Proposed Rule
  - Notice, Proposed Rulemaking for the Second Update to the Third Edition
- Chlorofluorocarbon 113 (CFC-113) Solvent Replacement Update
- Environmental Monitoring Methods Index (EMMI)
- Sampling Work Group Formation
- MICE Update
- Oily Waste Analysis
- Electronic SW-846 Availability.

1

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C4-380

Figure 3 - US Environmental Protection Agency Memorandum #35, Page 10

Oily Waste Analysis

One of the most frequently asked questions on the MICE Service concerns the application of the TCLP, Method 1311, to oily wastes. Many callers request technical guidance on the extraction of oily wastes due to the difficulty in the filtration on these types of waste. In many cases, an oily waste does not filter completely due to premature clogging of the glass fiber filter. This can result in the retention of standing liquid on the glass fiber filter. Material that do not pass through the glass fiber filter at the conclusion of the filtration step is defined by the method as the solid phase of the waste. The solid phase is then subjected to the leaching procedure of the TCLP. For oily wastes, clogging of the glass fiber filter can result in an overestimation of the amount of solid material available for leaching.

To solve this problem, the Agency recommends a conservative approach, one that probably will overestimate the amount of leaching. Rather than performing the TCLP extraction on the unfiltered portion of the oily waste, assume the waste is 100% liquid (e.g., will pass through the glass fiber filter) and perform a totals analysis on the oily waste to determine if the oil exceeds the appropriate regulatory level.

Filterable waste oil generated during the TCLP must be analyzed for a variety of organic and inorganic analytes. The OSW recognizes the difficulty in achieving acceptable performance for the analysis of waste oil using methods currently provided in SW-846. As a result, the Agency will provide several new methods for the preparation and analysis of oil samples to the Organic Methods Workgroup in July. In addition, a microwave assisted digestion procedure should improve the analysis of metals and will be proposed as part of the Second Update of the Third Edition of SW-846. Brief descriptions of these techniques are provided below, for additional information on the organic procedures contact Barry Lesnik at (202) 260-7459. For additional information on microwave digestion contact Ollie Fordham (202) 260-4778.

The use of purge-and-trap (Method 5030) for volatiles in oil generally results in severe contamination of analytical instrumentation. Traps, transfer lines and chromatography columns may become contaminated with oil. This leads to elevated baselines, hydrocarbon background in subsequent analyses, and cross-contamination. Headspace (Method 3810) is currently allowed only as a screening procedure in SW-846. The Agency is evaluating the use of headspace in conjunction with isotope dilution mass spectrometry for the quantitative analysis of volatiles in oil. Headspace reduces interference problems encountered with purge-and-trap. However, headspace quantitation can be questionable because the distribution of analytes is not

C4-381

APPENDIX C

WORKSHEETS

TCLP/SPLP Worksheet 1 - Sample Description

Laboratory Sample No.					
Field Sample No.					
A. Sample Description					
Number of phases					
1.					
2.					
a. lighter than					
b. water					
c. heavier than water					
B. Percent Solid Phase					
1. weight of filter (g)					
2. weight of subsample					
a. gross weight (g)					
b. tare weight (g)					
c. net weight (g)					
3. weight of filtrate					
a. gross weight (g)					
b. tare weight (g)					
c. net weight (g)					
4. total weight wet solids[0]					
5. weight percent wet solids[1] (%)					
6. weight percent dry solids[2] (%)					
a. gross dry weight (g)					
b. final gross dry weight[2] (g)					
c. percent dry solids (%)					
7. volume of initial aqueous filtrate (mL)					
8. volume of initial organic filtrate (mL)					

[0] Total Weight of wet solids $= (2c - 3c)$

[1] Weight percent wet solids $= 100\left(\frac{\text{Total weight wet solids, B4}}{\text{Weight of subsample, B2c}}\right)$

[2] Weight percent dry solids $= 100\left(\frac{(\text{Gross dry weight, B6b}) - (\text{weight of filter, B1})}{\text{Weight of subsample, B2c}}\right)$

Comments: _____

_____

Analyst: _____ Date: _____

C4-383

TCLP/SPLP Worksheet 2 - Selection of Extraction Fluid

TCLP

Laboratory Sample No.					
Field Sample No.					
C. Extraction Fluid Determination—does not apply to determination of volatile organic components.					
1. particle size reduction? yes/no (<1 mm)					
2. sample weight, ✓ if 5.0 ± 0.1 g					
3. volume of water, ✓ if 96.5 ± 1.0 mL					
4. initial pH (after 5 min. mixing time)					
5. if pH >5.0, ✓ if 3.5 mL 1N HCL					
6. ✓ if heated and held at 50^{0}C for ten minutes					
7. secondary pH (at room temperature)					
D. Selection of Extraction Fluid					
1. ✓ if pH from § C (4) or § C (7) is <5.0, use extraction fluid No. 1.					
2. ✓ if pH from § C (7) IS >5.0, use extraction fluid No. 2					
3. ✓ if filtrate and fluid are miscible					

SPLP

Laboratory Sample No.					
Field Sample No.					
D. Selection of Extraction Fluid (✓ one)					
Fluid 1: Soils—East of the Mississippi River; Wastes; or Wastewaters.					
Fluid 2: Soils—West of Mississippi River					
Fluid 3: If VOCs or Cyanide containing wastes.					
1. ✓ if filtrate and fluid are miscible					

Comments: _____

_____

Analyst: _____ Date: _____

TCLP/SPLP Worksheet 3 - Extraction for Metals, Semi-Volatile Organic Components and Pesticides/Herbicides

Laboratory Sample No.					
Field Sample No.					
E. Determination of Samples Size for Leach Testing—the method requires a minimum 100 gram sample size for extraction					
1. particle size reduction? yes/no (<9.5 mm)					
2. weight of wet solids after filtration (g)					
F. Determination of Amount of Extraction Fluid—the selection of the correct extraction fluid is found in Section D., Worksheet No. 2					
1. Fluid weight = 20 x solids weight (g)					
G. Record of Extraction Test—the extraction period is specified as 18 ± 2					
1. room temperature initial (°C) final (°C)					
2. vessel number					
3. extraction start time					
4. extraction stop time					
5. date and time filtration complete					
6. pH of leachate					
7. volume of leachate (for multiphase samples only) (mL)					
8. volume of initial aqueous filtrate for recombination[3] (mL)					
9. combined initial aqueous + leachate (mL)					

[3] Volume of filtrate used for recombination $= \left(\frac{\text{Weight of solids leached, E2}}{\text{Total weight of solids, B4}}\right)\left(\frac{\text{Leachate recovered, G6}}{\text{Fluid added, F1}}\right)\left(\text{Volume of initial aqueous filtrate, B7}\right)$

Comments: _____

_____

Analyst: _____ Date: _____

C4-385

TCLP/SPLP Worksheet 4 - Zero Headspace Extraction (ZHE)

Laboratory Sample No.					
Field Sample No.					
H. Determination of Sample Size for Leach Testing—maximum 25 grams					
1. particle size reduction? yes/no (<9.5 mm)					
2. weight of wet solid (g)					
I. Determination of Amount of Extraction Fluid No. 1					
1. weight of material added to ZHE (g)					
2. volume of filtrate in syringe (mL)					
3. weight of filtrate in syringe					
a. gross weight (g)					
b. tare weight (g)					
c. net weight (g)					
4. wet solids in ZHE[4] (g)					
5. weight of fluid to add[5] (g)					
J. Record of ZHE Extraction Text—the extraction period is as 18 ± 2 hours.					
1. room temperature initial (°C) final (°C)					
2. ZHE vessel number					
3. extraction start date & time					
4. extraction stop date & time					
5. ✓ if still under positive pressure					
6. filtration completed date & time					
7. volume of filtrate recovered after leaching (mL)					
a. volume of oil recovered after leaching (mL)					
8. volume of initial aqueous filtrate for recombination[6] (mL)					
9. combined initial aqueous + leachate (mL)					

[4] Solids remaining in ZHE $= (\textit{Material added}, I1) - (\textit{Weight of filtrate in syringe}, I3)$

[5] Weight of fluid to add $= 20(\textit{Wet solids in ZHE}, I4)$

[6] Volume of filtrate used for recombination $= \left(\frac{\textit{Weight of solids leached}, I4}{\textit{Total weight of solids}, B4}\right)\left(\frac{\textit{Leachate recovered}, J7}{\textit{Fluid added}, I5}\right)(\textit{Volume initial aqueous filtrate}, B7)$

Comments: _____

Analyst: _____ Date: _____

TCLP/SPLP Worksheet 5 - Organic Results

C4-386

TCLP/SPLP ZHE Worksheet No. 5
Organic Results

Sample Number: _____

Organics Preparation		Volume	Units
Combined initial Aqueous/Leachate from § J9	V_A		mL
Oil phase Volume for Calculations[7]	V_O		mL

Analyte		Oil Phase			Combined Initial Aqueous/Leachate			Final Results	
Compound	Std. Reporting Limit (mg/L)	Dilution Factor	Result C_O	Reporting Limit	Dilution Factor	Results C_A	Reporting Limit	Result C_{Final}	Reporting Limit

$$C_{Final} = \frac{(C_A \times V_A) + (C_O \times V_O)}{V_A + V_O}$$

[7] Volume of oil phase for calculation $= \left(\frac{\text{Wet solids leached, J4}}{\text{Total solid, B4}}\right)\left(\frac{\text{Leachate recovered, J7}}{\text{Fluid added, I5}}\right)(\text{Initial volume of oil, B8}) + (\text{Oil recovered from leaching, J7a})$

Comments: _____

_____

Analyst: _____ Date: _____

C4-387

TCLP/SPLP Worksheet 6 - Metals Results

Metals Results

Sample Number : _____

Metals Preparation		Volume	Units
Combined Initial Aqueous/Leachate from §G8	VA		mL
Oil phase volume for calculations[8]	VO		mL

Analyte		Oil Phase			Combined Initial Aqueous/Leachate			Final Results	
Element	Std. Reporting Limit (mg/L)	Dilution Factor	Result Co	Reporting Limit	Dilution Factor	Results C_A	Reporting Limit	Result C_{Final}	Reporting Limit
Arsenic	0.5								
Barium	10								
Cadmium	0.1								
Chromium	0.5								
Lead	0.5								
Mercury	0.0002								
Selenium	0.25								
Silver	0.5								

$$C_{Final} = \frac{(C_A \times V_A) + (C_O \times V_O)}{V_A + V_O}$$

[8] Volume of oil phase for calculation $= \left(\frac{\text{Wet solids leached, E2}}{\text{Total solid, B4}}\right)\left(\frac{\text{Leachate recovered, G7}}{\text{Fluid added, F1}}\right)(\text{Initial volume of oil, B8}) + (\text{Oil recovered from leaching, G6a})$

Notes: If sample contains no solids, volume of oil phase (V_0) is the volume of the initial organic filtrate

In cases where analytes are detected in only one phase, a footnote indicating a result reported below the reporting limit should be included.

Comments: _____

_____

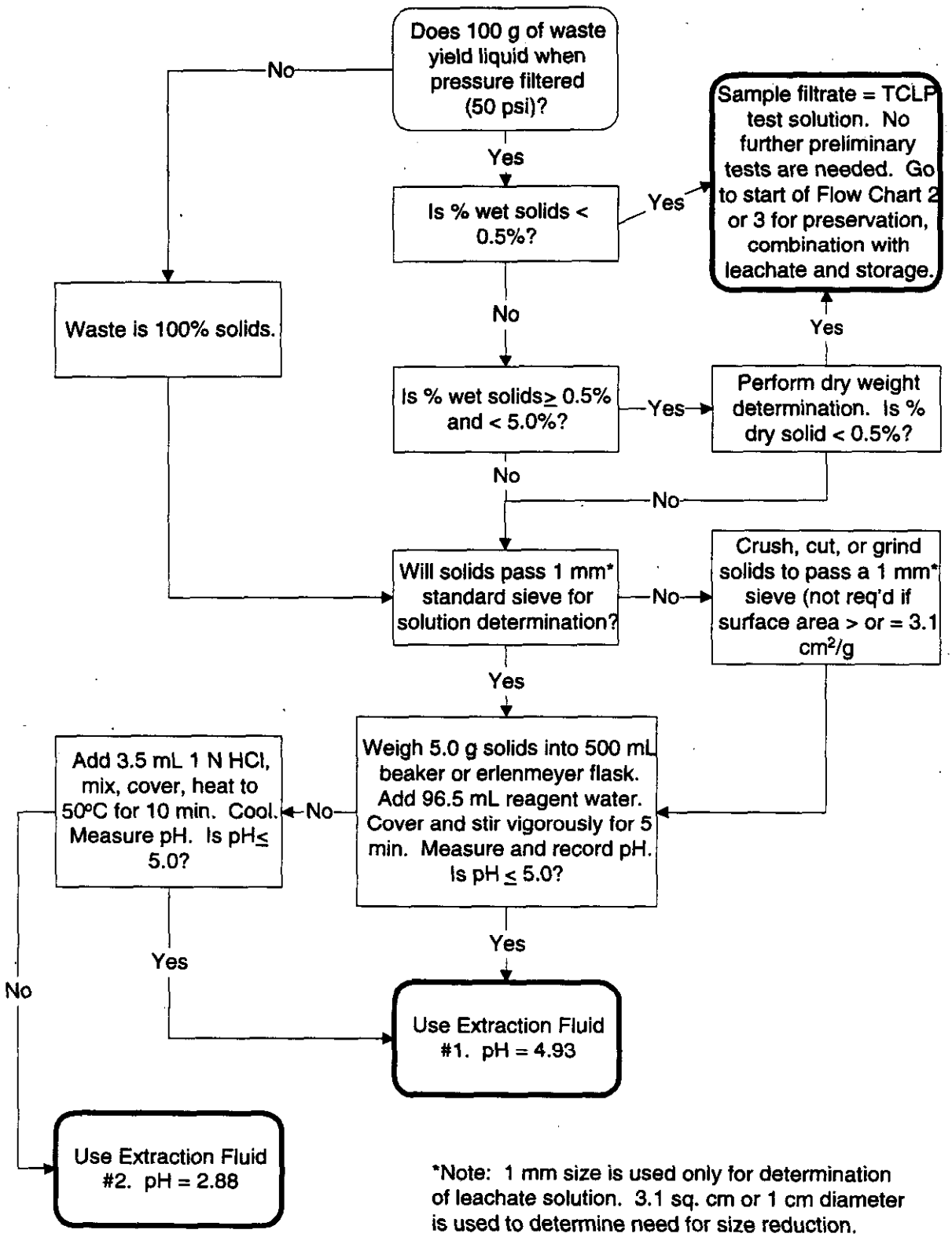
Analyst: _____ Date: _____

C4-388

APPENDIX D

FLOW CHARTS

Flow Chart 1. Preliminary Sample Evaluation (Section 11.2)

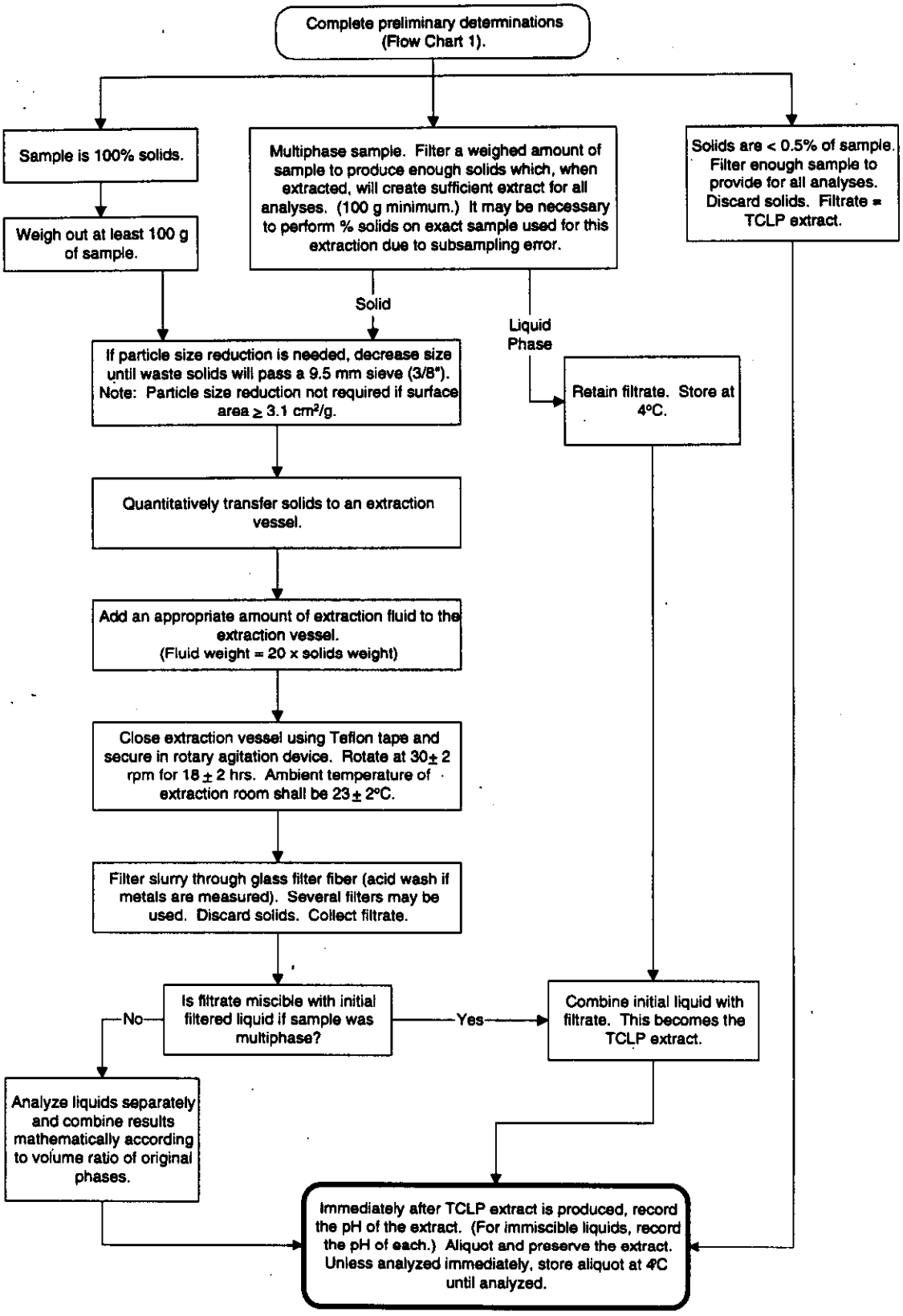


*Note: 1 mm size is used only for determination of leachate solution. 3.1 sq. cm or 1 cm diameter is used to determine need for size reduction.

C4-390

APPENDIX D - FLOW CHARTS

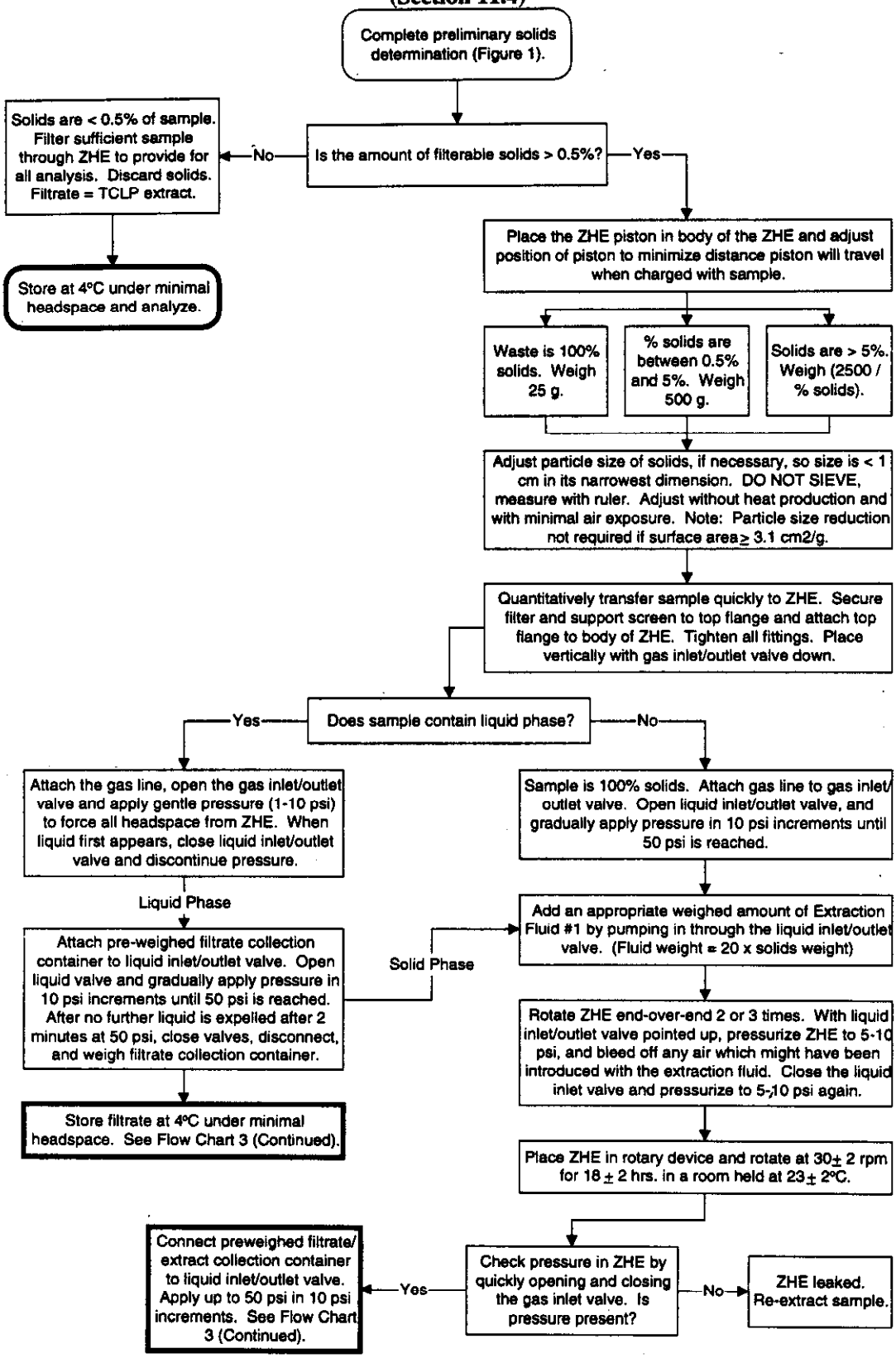
Flow Chart 2. Bottle Extraction, Non-Volatile Constituents (Section 11.3)



B4-391

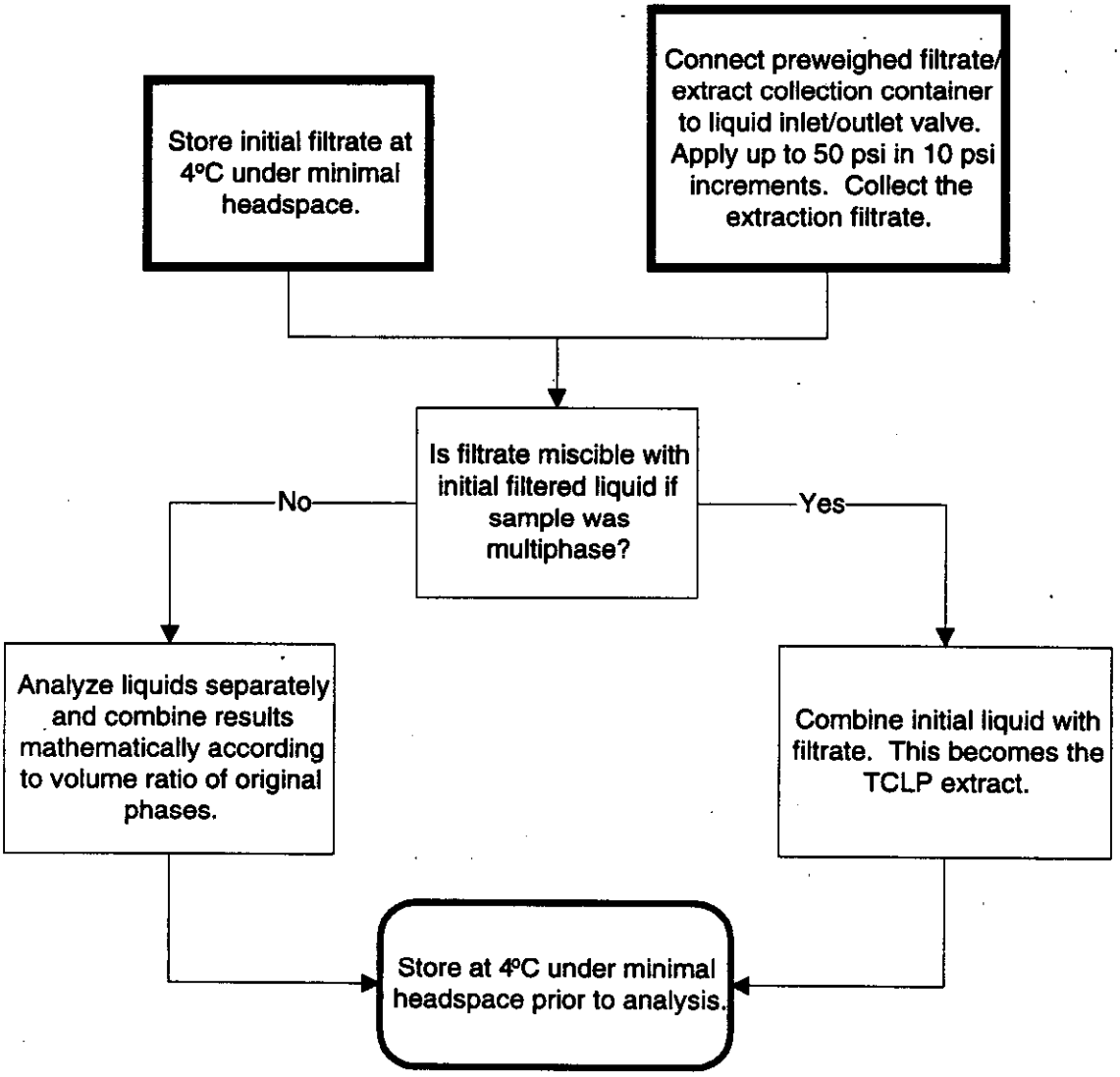
APPENDIX D - FLOW CHARTS

Flow Chart 3. ZHE Extraction, Volatile Constituents (Section 11.4)



C4-392

Flow Chart 3. ZHE Extraction (Continued)



QUANTERRA STANDARD OPERATING PROCEDURE

TITLE: DETERMINATION OF SOLIDS IN WATERS AND WASTES

(SUPERSEDES: CORP-WC-0002, REVISION 1)

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C4-394

TABLE OF CONTENTS

1. SCOPE AND APPLICATION 3

2. SUMMARY OF METHOD 3

3. DEFINITIONS 4

4. INTERFERENCES 5

5. SAFETY 6

6. EQUIPMENT AND SUPPLIES 7

7. REAGENTS AND STANDARDS 8

8. SAMPLE COLLECTION, PRESERVATION, AND STORAGE 9

9. QUALITY CONTROL 10

10. CALIBRATION AND STANDARDIZATION 12

11. PROCEDURE 13

12. DATA ANALYSIS AND CALCULATIONS 25

13. METHOD PERFORMANCE 27

14. POLLUTION PREVENTION 27

15. WASTE MANAGEMENT 27

16. REFERENCES 28

17. MISCELLANEOUS (TABLES, APPENDICES, ETC.) 28

FIGURE 1: DECISION TREE FOR FAILED EC/TDS RATIO 30

TABLE 1: SUMMARY OF QUALITY CONTROL REQUIREMENTS FOR SOLIDS DETERMINATIONS 32

1. SCOPE AND APPLICATION

1.1. This SOP is applicable to the determination of total solids, total suspended solids, total dissolved solids, volatile solids, volatile dissolved solids, and volatile suspended solids using gravimetric techniques. This SOP is based on the residue methods contained in *Methods for Chemical Analysis of Waters and Wastes* (MCAWW) and *Standard Methods for the Examination of Water and Wastewater* (SM).

1.2. This SOP is applicable to drinking, surface, and saline waters and domestic and industrial wastes.

1.3. The Total Solids protocol is based on MCAWW Method 160.3 and Standard Method 2540B.

1.4. The Total Dissolved Solids protocol is based on MCAWW Method 160.1 and Standard Method 2540C.

1.5. The Total Suspended Solids protocol is based on MCAWW Method 160.2 and Standard Method 2540D.

1.6. The Volatile Solids protocol is based on MCAWW Method 160.4 and Standards Method 2540E.

1.7. The Volatile Dissolved Solids protocol is based on MCAWW Method 160.4 and Standards Method 2540E.

1.8. The Volatile Suspended Solids protocol is based on MCAWW Method 160.4 and Standards Method 2540E.

1.9. The methods cover a practical range of 10 mg/L to 20,000 mg/L (TSS: 4 mg/L - 20,000 mg/L). As a practical matter, the final residue weight should be limited to about 200 mg.

2. SUMMARY OF METHOD

2.1. Total Solids (TS): A well-mixed aliquot of the sample is quantitatively transferred to a preweighed evaporating dish and evaporated to dryness at 103-105 °C. The increase in weight over that of the empty dish represents the total solids.

2.2. Total Dissolved Solids (TDS): A well-mixed sample is filtered through a glass fiber filter. The filtrate is quantitatively transferred into a preweighed evaporating dish and is evaporated to dryness and then dried to constant weight at 180 °C. The increase in weight over that of the empty dish represents the total dissolved solids. The filter from this procedure may also be used for TSS/VSS determination.

2.3. Total Suspended Solids (TSS): A well-mixed sample is filtered through a pre-weighed glass fiber filter. The residue on the filter is dried to constant weight at 103-105 °C. The increase in weight over that of the pre-weighed filter represents the TSS content. The filtrate from this procedure may be used for TDS determination. The filter from this procedure may also be used for VSS analysis.

2.4. Volatile Solids (VS): The residue obtained from the determination of total solids is ignited at 550 °C in a muffle furnace. The loss of weight on ignition is reported as mg/L volatile solids.

2.5. Volatile Dissolved Solids (VDS): The residue obtained from the determination of total dissolved solids is ignited at 550 °C in a muffle furnace. The loss of weight on ignition is reported as mg/L volatile dissolved solids.

2.6. Volatile Suspended Solids (VSS): A well-mixed sample is filtered through a glass fiber filter to separate the suspended material. The filter is dried and weighed, then ignited at 550 °C and reweighed. Volatile suspended solids is determined from the weight loss after ignition. The filter from the analysis of TSS may be used for the determination of VSS.

3. DEFINITIONS

3.1. Total Solids (TS): The term applied to the residue left in the vessel after evaporation of a sample and its subsequent drying in an oven at 180 °C. Total solids includes "total suspended solids," the portion of solids retained by a filter, and "total dissolved solids," the portion that passes through the filter.

3.2. Total Dissolved Solids (TDS): Those solids capable of passing through a glass fiber filter and dried to constant weight at 180 °C. TDS is also referred to as filterable residue.

3.3. Total Suspended Solids (TSS): Those solids which are retained by a glass fiber filter and dried to constant weight at 103-105 °C. TSS is also referred to as non-filterable residue.

3.4. Volatile Solids (VS): The portion of total solids which is lost on ignition at 550 °C.

3.5. Volatile Dissolved Solids (VDS): The portion of total dissolved solids which is lost on ignition at 550 °C.

3.6. Volatile Suspended Solids (VSS): The portion of suspended solids which is lost on ignition at 550 °C.

3.7. Aliquot: A representative portion of a sample.

3.8. Reagent Water: Deionized water which is free of the analyte(s) of interest.

4. INTERFERENCES

4.1. Method interferences may be caused by contaminants, reagents, glassware, and other sample processing hardware. All these materials must be routinely demonstrated to be free from interferences under the conditions of analysis by running method blanks.

4.2. Non-homogeneous samples may give erroneous results. Samples should be mixed as thoroughly as possible before removing an aliquot for analysis.

4.3. Non-representative particulates such as leaves, sticks, fish, and lumps of fecal matter should be excluded from the sample if it is determined that their inclusion is not desired in the final result. The presence/removal of these artifacts should be noted on the benchsheet.

4.4. Samples containing large amounts of solids may filter slowly. Prolonged filtration times resulting from filter clogging may produce high TSS results due to increased colloidal materials captured on the clogged filter.

4.5. Oil and grease in the samples will cause unreliable results due to difficulty in drying to constant weight. Floating oil and grease, if present, should be included in the sample and dispersed by a blender device before aliquoting.

4.6. Filtration apparatus, filter material, pre-washing, post-washing, and drying temperatures are specified because these variables have been shown to affect the results.

4.7. The temperature at which the residue is dried has an important bearing on the results because weight losses due to volatilization of organic matter, mechanically

occluded water, water of crystallization, and gases from heat-induced chemical decomposition, as well as weight gains due to oxidation, depend on temperature and time of heating.

4.8. Each sample requires close attention to desiccation after drying. Minimize opening the desiccator because moist air enters. Some samples may be stronger desiccants than those used in the desiccator and may take on water.

4.9. Highly mineralized waters containing significant concentrations of calcium, magnesium, chloride, and/or sulfate may be hygroscopic and will require prolonged drying, desiccation and rapid weighing.

4.10. Samples containing high concentrations of bicarbonate may require careful and possibly prolonged drying to ensure that all the bicarbonate is converted to carbonate.

4.11. Too much residue in the drying vessel will crust over, entrapping water that will not be driven off during drying. Total residue should be limited to about 200 mg.

4.12. Some samples may have fine suspended solids which will pass through the glass fiber filter causing high TDS results.

4.13. Aluminum pans should not be used for TS or TDS analyses. Components in some samples may react to form aluminum compounds, causing unreliable results.

4.14. For samples high in dissolved solids, thoroughly wash the filter to ensure removal of dissolved material prior to TSS determination.

4.15. The volatile solids tests are subject to many errors due to the loss of water of crystallization, loss of volatile organic matter prior to combustion, incomplete oxidation of certain complex organics and decomposition of mineral salts during combustion. The results should not be considered an accurate measure of organic carbon in the sample.

5. SAFETY

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

5.2. Eye protection that satisfies ANSI Z87.1 (as per the Chemical Hygiene Plan), laboratory coat, and chemically resistant gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have

been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.3. The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the Material Safety Data Sheets (MSDS) maintained in the laboratory.

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

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

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

5.7. If possible, place the oven or muffle furnace under a canopy hood or vent in to a fume hood. Do not stand directly in front of an oven or muffle furnace as the door is being opened. Stand behind the door to avoid breathing fumes that may be present.

5.8. Caution should be used when handling apparatus treated in the oven. Items will be hot. Heat resistant gloves and tongs must be used when placing material into or removing material from the muffle furnace. At a minimum, tongs must be used when placing material into or removing material from the oven.

6. EQUIPMENT AND SUPPLIES

6.1. Analytical balance capable of weighing to 0.0001 g.

6.2. Vacuum filtration apparatus.

6.3. Vacuum pump equipped with moisture trap.

6.4. Glass fiber filter disks, 47 mm, without organic binder (Gelman Type A/E) or equivalent.

Note: Commercially available pre-weighed filters may be used for TSS and VSS analyses.

6.5. Evaporating dishes, 100 mL, made of either porcelain, Vycor or platinum. These must be thoroughly cleaned, rinsed with deionized water, and baked at 180 ± 2 °C for at least one hour before use. Store in a desiccator.

Note: Glass beakers may also be used for TS or TDS determinations. Glass beakers may not be used for procedures requiring use of a muffle furnace.

6.6. Desiccators providing sufficient space for storage of samples in process separate from filters and evaporating dishes.

6.7. Desiccant containing a color indicator of moisture concentration or an instrumental indicator.

6.8. Drying ovens set at 103-105 °C and 180 ± 2 °C. Separate ovens should be maintained at appropriate temperatures if possible.

6.9. Muffle furnace (550 °C ± 50 °C).

6.10. Thermometers, NIST traceable.

6.11. Conductivity meter and associated apparatus.

6.12. Graduated cylinders, "to contain," assorted sizes.

6.13. Volumetric flasks, Class A, assorted sizes.

6.14. Glass weighing dishes large enough to hold a 47 mm filter.

6.15. Forceps for handling filters.

6.16. Crucible tongs.

6.17. Zetex gloves or other gloves capable of providing protection at 550 °C.

7. REAGENTS AND STANDARDS

7.1. Reagent water must be produced by a Millipore DI system or equivalent (see also Section 10.3).

7.2. LCS solution (ERA Mineral Standard for TSS and 500 mg/L NaCl for TDS and TS): Place 500 mg sodium chloride into a 1000 mL volumetric flask and dilute to volume with deionized water. Mix well. Prepare fresh every three months.

7.3. Spike solutions for Matrix Spikes for TDS analysis is 500 ppm TDS (5 g NaCl to diluted to a final volume of 1000 mL).

Note: Commercially available reference materials may also be used.

8. SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1. Samples should be collected in either plastic or glass bottles.

8.2. Samples must be stored at 4 °C ± 2 °C to minimize microbiological decomposition of solids. Bring samples to room temperature before analysis.

8.3. To achieve the reporting limits listed in Section 13, the recommended minimum volumes are as follows:

Parameter	Volume (mL)
TS	100
TDS	100
TSS	100
VS	100
VDS	100
VSS	250

8.4. Holding times for solids determinations are as follows:

Parameter	Holding Time (days)	Parameter	Holding Time (days)
TS	7	VS	7
TDS	7	VDS	7
TSS	7	VSS	7

9. QUALITY CONTROL

The Quanterra QC Program document (QA-003) provides further details of the QC and corrective action guidelines presented in this SOP. Refer to this document if additional guidance is required.

Table I (Section 17.7) provides a summary of quality control requirements including type, frequency, acceptance criteria, and corrective action.

9.1. Initial Demonstration of Capability

Prior to analysis of any samples using this SOP, the following requirements must be met:

9.1.1. Method Detection Limit (MDL): An MDL must be determined prior to analysis of any samples. The MDL is determined using seven replicates of reagent water, spiked with a reference material that has been carried through the entire analytical procedure. MDLs must be redetermined on an annual basis in accordance with 40 CFR Part 136 Appendix B requirements as detailed in Quanterra QA Policy QA-005. The result of the MDL determination must be below the Quanterra standard reporting limit.

9.1.2. Initial Demonstration Study: This requires the analysis of four QC check samples. The QC check sample is a well-characterized, laboratory-generated sample used to monitor method performance, which should contain the analyte(s) of interest. The results of the initial demonstration study must be acceptable before analysis of samples under this SOP may begin.

9.1.2.1. Four aliquots of the check sample (LCS) are prepared and analyzed using the procedures detailed in this SOP.

9.1.2.2. Calculations and acceptance criteria for the LCS are given in Section 9.4.

9.2. Batch Definition: Batch definitions are provided in the Quanterra Quality Control Program (QA-003).

9.3. Method Blank (MB): One method blank must be processed with each batch of 20 or less samples. The method blank consists of reagent water that is carried through the entire analytical procedure, including filtration as applicable. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data.

9.3.1. A reagent water blank is prepared and analyzed with each batch of samples. The volume of reagent water used should be the same as the minimum sample volume used for the test (Section 8.3).

9.3.2. Corrective Action for Blanks

9.3.2.1. If the analyte level in the method blank is ≥ the RL for the analyte of interest in the sample, all associated samples with reportable levels of analyte are reprepared and reanalyzed. If this is not possible due to limited sample quantity or other considerations, the corresponding sample data **must be addressed in the project narrative**. Refer to QA-003 for further details of corrective actions.

9.3.2.2. High blank results indicate contamination. Make sure the glassware and filtering apparatus is clean.

9.3.2.3. Low blanks typically indicate that the balance was not operating correctly or the filter/weighing dish was not completely dry when the initial weights were measured.

9.3.2.4. If there is no analyte greater than the RL in the samples associated with an unacceptable method blank, the data may reported with qualifiers. **Such action must be taken in consultation with the client and must be addressed in the project narrative.**

9.4. Laboratory Control Sample (LCS): One LCS must be processed with each batch of 20 or less samples. The LCS must be carried through the entire analytical procedure. The LCS is used to monitor the accuracy of the analytical process. On-going monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines. No LCS is available for volatile residue procedures.

9.4.1. An LCS is prepared and analyzed with each analytical batch of samples. Refer to Section 7.2 for instructions on how to prepare the LCS solution. The LCS should be the same volume as the method blank.

9.4.2. Corrective Action for LCS

9.4.2.1. A control limit of 80-120% recovery must be applied until in-house control limits are established. If the result is outside

established control limits, the system is out of control and corrective action must occur.

9.4.2.2. Corrective action will include repreparation and reanalysis of the batch unless the client agrees that another corrective action is acceptable. If this is not possible due to limited sample quantity or other considerations, the corresponding sample data **must be addressed in the project narrative.**

9.4.2.3. Poor LCS results are usually the result of contamination or poor mixing.

9.5. A Matrix Spike/Matrix Spike Duplicate (MS/MSD or MS/SD) pair must be extracted with every process batch of similar matrix, not to exceed twenty (20) samples. An MS/MSD pair are aliquots of a selected field sample spiked with analytes of known identity and concentration. The MS/MSD pair must be processed in the same manner and at the same time as the associated samples. Spiked analytes with recoveries or precision outside control limits must be within control limits in the LCS. Corrective actions must be documented on a Non-Conformance memo, then implemented when recoveries of any spike analyte is outside control limits provided on the LIMS or by the client. Re-extraction of the blank, an LCS, the selected field sample, the MS/MSD may be required after evaluation and review.

9.6. A duplicate control sample (LCSD or DCS) must be substituted when insufficient sample volume is provided to process an MS/MSD pair. The LCSD is evaluated in the same manner as the LCS. See Quanterra Inc. QA Policy 003 for specific acceptance criteria. Sample results should agree within 20% if both the sample and sample duplicate results are > 5x RL.

10. CALIBRATION AND STANDARDIZATION

10.1. Since this method is based on gravimetric techniques, there is no calibration in the usual sense. Proper balance operation will be verified daily or prior to sample analysis by following the lab-specific balance calibration SOP. Analytical balance calibration must be performed daily (every 24 hours).

10.2. Oven temperature must be checked daily and recorded either on the benchsheet or in an oven temperature logbook.

10.3. Conductivity of the water must be monitored and recorded in the Conductivity Logbook daily. The maximum permissible conductivity is 1.0 umhos/cm (at 25

°C). If the conductivity reading on the water system exceeds this level, do not use the water for these procedures and notify the supervisor immediately.

11. PROCEDURE

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

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

11.3. All samples are to be checked out of sample control with the chain of custody documentation filled out completely.

11.4. Proper sample identification is extremely important in any analytical procedure. Labeling of evaporating dishes and filters holders must be done in a manner to ensure connection with the proper sample.

11.5. If possible, analyze all the samples of a project at the same time to minimize the QC required and streamline the flow of the project through the lab and reporting group.

11.6. Non-representative particulates such as leaves, sticks, fish, and lumps of fecal matter should be excluded from the sample if it is determined that their inclusion is not desired in the final result. The presence/removal of these artifacts should be noted on the benchsheet.

11.7. If samples are visibly oily, this should be noted on the benchsheet.

11.8. If there is limited sample volume or high solids content, smaller amounts of sample may need to be processed than detailed in the following sections. This occurrence must be noted on the benchsheet and reporting limits must be adjusted appropriately.

11.9. Proceed to the appropriate section for the desired method as follows:

Total Solids	11.10
Total Dissolved Solids	11.11
Total Suspended Solids	11.12
Volatile Solids	11.13
Volatile Dissolved Solids	11.14
Volatile Suspended Solids	11.15

11.10. Total Solids

11.10.1. Preparation of Evaporating Dish

11.10.1.1. If only total solids are to be measured, heat clean dish to 103-105 °C for one hour. If volatile solids are to be measured in addition to total solids, ignite the clean evaporating dish at 550 °C for one hour in a muffle furnace.

11.10.1.2. Remove the dish from the muffle furnace using tongs and heat resistant gloves.

11.10.1.3. Cool and store dish in desiccator until dish reaches room temperature or until needed.

11.10.1.4. Weigh immediately before use to the nearest 0.1 mg. Record the weight on the benchsheet.

11.10.2. Sample Analysis

11.10.2.1. Transfer a measured aliquot of well-mixed sample to the pre-weighed, labeled dish. Record the volume of sample (to the nearest mL) on the benchsheet. All benchsheet entries are entered into the Total Solids and Total Dissolved Solids Benchsheet Software program.

11.10.2.1.1. Choose an aliquot of sample sufficient to contain a residue of at least 25 mg but less than 200 mg. To obtain a weighable residue, successive aliquots of sample may be added to the same dish after evaporation.

11.10.2.1.2. If the sample is known to contain > 2000 mg/L dissolved solids, it should be diluted. Prescreening may be performed using a conductivity meter to determine the required sample volume or dilution. For this purpose, the following formula should be used to approximate the required sample volume:

(2000 x 100) / Conductivity = mL sample to use

Note: To avoid "oddball" reporting limits, round the determined sample volume to one of the following volume increments: 5, 10, 20, 25, 50, or 100 mL.

11.10.2.1.3. For the LCS, measure 100 mL of the LCS Solution (Section 7.2) and pour into the dish.

11.10.2.1.4. For the MB, measure 100 mL of reagent water and pour into the dish.

11.10.2.2. Evaporate the sample to dryness on a steam bath or in a drying oven. If evaporation is performed in a drying oven, the temperature should be lowered as necessary to prevent boiling and splattering of the sample.

11.10.2.3. Dry the evaporated sample for at least one hour at 103-105 °C.

11.10.2.4. Record the date, time, and oven temperature on the benchsheet when the samples are initially placed in the oven and again when they are removed from the oven.

11.10.2.5. Using tongs, remove the weighing dish from the oven and place in a desiccator and cool to room temperature.

11.10.2.6. Weigh the dish to the nearest 0.1 mg. Record the weight on the benchsheet. All benchsheet entries are entered into the Total Solids and Total Dissolved Solids Benchsheet Software program.

11.10.2.7. Return the samples to the oven for another hour, cool in a desiccator, and reweigh. Repeat the drying, cooling, desiccating, and weighing cycle until a constant weight is obtained or weight loss is less than 1.0 mg. If more than three cycles are required, prepare a Nonconformance Memo.

Note: When weighing dried sample, be alert to change in weight due to air exposure and/or sample degradation.

11.10.2.8. If volatile solids are to be determined, treat the residue according to Section 11.13.

11.10.2.9. Calculate results according to the equation provided in Section 12.1. All benchsheet entries are entered into the Total Solids and Total Dissolved Solids Benchsheet Software program. The software program completes all calculations. Use the lowest final weight achieved for calculating TS.

11.11. Total Dissolved Solids

Measure and record the conductance of each sample before preparation and analysis for TDS. Conductance multiplied by a factor between 0.55 and 0.81 gives a rough approximation of TDS. See calculations in Section 12.3. The measured TDS should be within the range of the expected TDS result calculated from conductance. If the measured TDS is not within the expected TDS range calculated from conductance, follow the corrective action protocol in Figure 1.

11.11.1. Preparation of Glass Fiber Filter Disc: Refer to Section 17.1.2.

11.11.2. Preparation of Evaporating Dishes

11.11.2.1. If only total dissolved solids are to be measured, heat clean dish to 180 ± 2 °C for one hour. If volatile dissolved solids are to be measured in addition to TDS, ignite the clean evaporating dish at 550 ± 50 °C for one hour in a muffle furnace.

11.11.2.2. Heat resistant gloves and tongs must be used when removing items from the muffle furnace.

11.11.2.3. Store and cool dish in desiccator until dish reaches room temperature or until needed.

Note: Always transfer the dish with gloves or tongs to prevent added weight due to oil from fingerprints.

11.11.2.4. Weigh immediately before use to the nearest 0.1 mg. All benchsheet entries are entered into the Total Solids and Total Dissolved Solids Benchsheet Software program.

11.11.3. Sample Analysis

11.11.3.1. Thoroughly rinse the entire filtration apparatus with reagent water before filtering each sample.

11.11.3.2. Assemble the filtering apparatus, place a glass fiber filter in the apparatus, pre-wet the filter using reagent water, and begin suction.

Note: If the sample also requires TSS, preweigh the prepared filter and refer to Section 11.12 for additional guidance.

11.11.3.3. Shake the sample vigorously and rapidly transfer 100 mL (or a smaller portion which will yield between 10 and 200 mg dried residue) to the funnel by means of a graduated cylinder. If total dissolved solids content is low, a larger volume may be filtered. If more than 10 minutes are required to complete filtration, decrease sample size.

Note: Multiple filters may be used if performing only TDS analysis.

11.11.3.3.1. The conductance of each sample may be used to determine the appropriate sample volume to process.

Note: TDS is typically 55-90% of the conductance result. The exact relationship depends on the compounds present in the samples and may not hold for very high concentrations or samples containing non-ionic species or samples with conductances

greater than 10,000 umho/cm or less than 50 umho/cm.

11.11.3.3.1.1. If the sample has a conductance less than 3,000 umhos/cm, 100 mL should be used.

11.11.3.3.1.2. If the conductance is 3,000 to 10,000 umhos/cm, use 20 mL of sample.

11.11.3.3.1.3. If the conductance is extremely high (> 10,000 umhos/cm) smaller volumes may be used.

11.11.3.3.2. All benchsheet entries are entered into the Total Solids and Total Dissolved Solids Benchsheet Software program.

11.11.3.3.3. For the method blank, process 100 mL of reagent water as the sample.

11.11.3.3.4. For the LCS, process 100 mL of the LCS Solution. Refer to Section 7.2 for instructions on how to prepare the LCS.

11.11.3.4. Filter the sample through the glass fiber filter.

11.11.3.5. Rinse the graduated cylinder, funnel walls, and filter with three successive 10 mL portions of reagent water and allow for complete drainage between washings. Continue to apply vacuum for about three minutes after filtration is complete to remove as much water as possible.

11.11.3.6. Transfer the filtrate (including the washings) to a pre-weighed evaporating dish. Rinse the receiving flask with 10-25 mL of reagent water and transfer washings into the dish to ensure complete transfer of the sample.

11.11.3.7. Evaporate the samples to dryness on a steam bath or in an oven set slightly below boiling. If filtrate volume exceeds

dish capacity, add successive portions to the same dish after evaporation.

11.11.3.8. Dry the evaporated sample in an oven for at least one hour at 180 ± 2 °C.

11.11.3.9. Record the date, time, and oven temperature on the benchsheet when the samples are initially placed in the oven and again when they are removed from the oven.

11.11.3.10. Using tongs, remove the weighing dish from the oven and place in a desiccator and cool to room temperature.

11.11.3.11. Weigh the dish to the nearest 0.1 mg. All benchsheet entries are entered into the Total Solids and Total Dissolved Solids Benchsheet Software program.

11.11.3.12. Return the samples to the oven for another hour, cool in a desiccator, and reweigh. Repeat the drying, cooling, desiccating and weighing cycle until a constant weight is obtained or weight loss is less than 1.0 mg. If more than three cycles are required, prepare a Nonconformance Memo.

11.11.3.13. Calculate results according to the equation in Section 12.2. All benchsheet entries are entered into the Total Solids and Total Dissolved Solids Benchsheet Software program. The software program completes all calculations. Use the lowest final weight achieved for calculating TDS.

11.12. Total Suspended Solids

11.12.1. Preparation of Glass Fiber Filter Disc

Note: As an alternative to the steps outlined in Sections 11.12.1.1 through 11.12.1.7, prepared and pre-weighed filters may be purchased for use under this method.

11.12.1.1. Place the glass fiber filter discs, one at a time, on the membrane filter apparatus or insert into bottom of a suitable Gooch crucible with wrinkled surface up.

11.12.1.2. While vacuum is applied, wash the disc with three successive 20 mL volumes of distilled water.

11.12.1.3. Remove all traces of water by continuing to apply vacuum after water has passed through and discard washings.

11.12.1.4. Remove filter from membrane filter apparatus and place in a labeled, glass weighing dish (or remove both the crucible and filter if a Gooch crucible is used), and dry in an oven at 103-105 °C for one hour.

Note: If volatile suspended solids (VSS) are to be determined as well as total suspended solids, the filter must be heated at 550 °C for one hour, instead of 103-105 °C.

11.12.1.5. Using tongs, remove the weighing dish from the oven and place in a desiccator and cool to room temperature.

11.12.1.6. Weigh the cooled filter using an analytical balance to the nearest 0.1 mg. Handle the filters or crucible/filter only with forceps. All benchsheet entries are entered into the Total Solids and Total Dissolved Solids Benchsheet Software program.

11.12.1.7. Repeat the drying, cooling, and weighing cycle until a constant weight is obtained or weight loss is less than 1.0 mg.

11.12.2. Selection of Sample Volume

11.12.2.1. For a 47 mm diameter filter, filter 100 mL of sample.

11.12.2.2. If during filtration of this initial volume, the filtration rate drops rapidly or if filtration time exceeds 5-10 minutes, a smaller volume of sample should be processed.

Note: If the sample appears high in TSS, start with a sample volume < 100 mL.

11.12.3. Sample Analysis

11.12.3.1. Assemble the filtering apparatus, place the pre-weighed glass fiber filter in the apparatus, pre-wet the filter using reagent water and begin suction.

Note: Handle the filters or crucible/filter only with forceps.

11.12.3.2. Shake the sample vigorously and quantitatively transfer 100 mL (or an appropriate aliquot) of the sample to the filter using a graduated cylinder or pipet. A smaller amount should be filtered if the sample is high in TSS or is otherwise slow to filter. Filter 25 mL at a time until filtration slows. Record the volume of sample filtered (to the nearest mL) on the benchsheet.

Note: If Total Dissolved Solids (TDS) is also required, the filtrate may be used. Refer to Section 11.11 for additional guidance.

11.12.3.3. Remove all traces of water by continuing to apply vacuum after the sample has passed through.

11.12.3.4. With suction on, rinse the graduated cylinder, filter, suspended solids residue, and filter funnel wall with three 10 mL portions of reagent water allowing complete drainage between washings.

11.12.3.5. Remove all traces of water by continuing to apply vacuum for about three minutes after the sample has passed through.

11.12.3.6. Carefully remove the filter from the filter support and transfer to an glass weighing dish. Alternatively, remove the crucible and filter from the crucible adapter.

11.12.3.7. Dry the filter at least one hour at 103-105 °C.

11.12.3.8. Record the date, time, and oven temperature on the benchsheet when the samples are initially placed in the oven and again when they are removed from the oven.

11.12.3.9. Using tongs, remove the weighing dish from the oven and place in a desiccator and cool to room temperature.

11.12.3.10. Cool the samples in a desiccator, weigh (to the nearest 0.1 mg). Enter weight into the Total Solids and Total Dissolved Solids Benchsheet Software program.

11.12.3.11. Return the samples to the oven for another hour, cool in a desiccator, and reweigh. Repeat the drying, cooling, desiccating, and weighing cycle until a constant weight is obtained or weight loss is less than 1.0 mg. If more than three cycles are required, prepare a Nonconformance Memo.

11.12.3.12. If volatile suspended solids are to be determined, treat the residue according to Section 11.15.

11.12.3.13. Calculate the results using the formula given in Section 12.3. All benchsheet entries are entered into the Total Solids and Total Dissolved Solids Benchsheet Software program. Use the lowest final weight achieved for calculating TSS.

11.13. Volatile Solids

11.13.1. Heat muffle furnace up to temperature (550 ± 50 °C).

11.13.2. Place evaporating dish containing residue generated by Total Solids protocol (Section 11.10) in muffle furnace to ignite the residue.

11.13.3. Record the date, time, and oven temperature on the benchsheet when the samples are initially placed in the oven and again when they are removed from the oven.

11.13.4. Typically, 15-20 minutes ignition are required for 200 mg of residue. However, more than one sample and/or heavier residues may necessitate longer ignition times.

11.13.5. Let dish cool partially in air until most of the heat has dissipated before transferring to a desiccator for final cooling.

11.13.6. Using tongs and heat resistant gloves, remove the weighing dish from the muffle furnace and place in a desiccator and cool to room temperature.

11.13.7. Weigh dish as soon as it has cooled to room temperature.

11.13.8. Return the samples to the oven for another hour, cool in a desiccator and reweigh. Repeat the drying, cooling, desiccating, and weighing cycle until a constant weight is obtained or weight loss is less than 1.0 mg. If more than three cycles are required, prepare a Nonconformance Memo.

11.13.9. Calculate the results using the formula given in Section 12.4. Use the lowest final weight achieved for calculating VS.

11.14. Volatile Dissolved Solids

11.14.1. Heat muffle furnace up to temperature (550 ± 50 °C).

11.14.2. Place evaporating dish containing residue generated by Total Dissolved Solids protocol (Section 11.3) in muffle furnace to ignite the residue.

11.14.3. Record the date, time, and oven temperature on the benchsheet when the samples are initially placed in the oven and again when they are removed from the oven.

11.14.4. Typically, 15-20 minutes ignition are required for 200 mg of residue. However, more than one sample and/or heavier residues may necessitate longer ignition times.

11.14.5. Let dish cool partially in air until most of the heat has dissipated before transferring to a desiccator for final cooling.

11.14.6. Using tongs and heat resistant gloves, remove the weighing dish from the muffle furnace and place in a desiccator and cool to room temperature.

11.14.7. Weigh dish as soon as it has cooled to room temperature.

11.14.8. Return the samples to the oven for another hour, cool in a desiccator and reweigh. Repeat the drying, cooling, desiccating and weighing cycle until a constant weight is obtained or weight loss is less than 1.0 mg. If more than three cycles are required, prepare a Nonconformance Memo.

11.14.9. Calculate the results using the formula given in Section 12.5. Use the lowest final weight achieved for calculating VDS.

11.15. Volatile Suspended Solids

11.15.1. Heat muffle furnace up to temperature (550 ± 50 °C).

11.15.2. Place glass fiber filter disc containing residue generated by Total Suspended Solids protocol (Section 11.12) in muffle furnace to ignite the residue.

11.15.3. Record the date, time, and oven temperature on the benchsheet when the samples are initially placed in the oven and again when they are removed from the oven.

11.15.4. Typically, 15-20 minutes ignition are required for 200 mg of residue. However, more than one sample and/or heavier residues may necessitate longer ignition times.

11.15.5. Let filter cool partially in air until most of the heat has dissipated before transferring to a desiccator for final cooling.

11.15.6. Using tongs and heat resistant gloves, remove the weighing dish from the muffle furnace and place in a desiccator and cool to room temperature.

11.15.7. Weigh filter as soon as it has cooled to room temperature.

11.15.8. Return the samples to the oven for another hour, cool in a desiccator, and reweigh. Repeat the drying, cooling, desiccating, and weighing cycle until a constant weight is obtained or weight loss is less than 1.0 mg. If more than three cycles are required, prepare a Nonconformance Memo.

11.15.9. Calculate the results using the formula given in Section 12.6. Use the lowest final weight achieved for calculating VSS.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Calculate **Total Solids** as follows:

$$\text{Total Solids, mg / L} = \frac{(A - B) \times 1000}{C}$$

where: A = weight of dried residue + dish (mg)
B = weight of dish (mg)
C = volume of sample (mL)

12.2. Calculate **Total Dissolved Solids** as follows:

$$\text{Total Dissolved Solids, mg / L} = \frac{(A - B) \times 1000}{C}$$

where: A = weight of dried residue + dish (mg)
B = weight of dish (mg)
C = volume of sample (mL)

12.3. Calculate **Total Suspended Solids** as follows:

$$\text{Total Suspended Solids, mg / L} = \frac{(A - B) \times 1000}{C}$$

where: A = weight of filter + residue (mg)
B = weight of filter (mg)
C = volume of sample filtered (mL)

Calculate the expected TDS range from conductance (as umho/cm).

Expected TDS_{low} (mg/L) = (Conductance x 0.55)

Expected TDS_{high} (mg/L) = (Conductance x 0.81)

12.4. Calculate **Volatile Solids** as follows:

$$\text{Volatile Solids, mg / L} = \frac{(A - B) \times 1000}{C}$$

where: A = weight of residue + dish before ignition (mg)
B = weight of residue + dish after ignition (mg)
C = volume of sample (mL)

12.5. Calculate **Volatile Dissolved Solids** as follows:

$$\text{Volatile Dissolved Solids, mg / L} = \frac{(A - B) \times 1000}{C}$$

where: A = weight of residue + dish before ignition (mg)
B = weight of residue + dish after ignition (mg)
C = volume of sample (mL)

12.6. Calculate **Volatile Suspended Solids** as follows:

$$\text{Volatile Suspended Solids, mg / L} = \frac{(A - B) \times 1000}{C}$$

where: A = weight of residue + filter before ignition (mg)
B = weight of residue + filter after ignition (mg)
C = volume of sample (mL)

12.7. The relative percent difference (RPD) of duplicates are calculated according to the following equation:

$$RPD = 100\left[\frac{|DU1 - DU2|}{\left(\frac{DU1 + DU2}{2}\right)}\right]$$

Where: DU1 = Sample result
DU2 = Sample duplicate result

12.8. If multiple weighing cycles are required, the lowest final sample weight is used for calculating solids content.

12.9. If smaller or larger sample volumes are processed than are specified in the method, the reporting limit must be adjusted accordingly.

12.10. Sample results should be reported according to the following significant figure rules:

Significant Figures	Sample Result
2	< 10
3	≥ 10

13. METHOD PERFORMANCE

13.1. The reporting limit is 10 mg/L for TS, TDS, VS, VDS, and VSS.

13.2. The reporting limit is 5 mg/L for TSS.

13.3. The initial demonstration study as detailed in Section 9.1.2 must be acceptable before the analysis of field samples under this SOP may begin.

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

14. POLLUTION PREVENTION

14.1. No solvents of any kind or in any amount are to be disposed of in the sinks or evaporated in the hoods.

15. WASTE MANAGEMENT

15.1. Waste management in the procedure must be segregated and disposed according to the facility hazardous waste procedures. The Environmental Health and Safety Coordinator or Hazardous Materials Technician should be contacted if additional information is required.

15.2. All waste must be disposed of according to the facility hazardous waste management procedures, Attachment C of the Chemical Hygiene Plan, Section WS002, Table 1.

15.3. Samples and other solutions containing high concentrations of toxic materials must be disposed of according to the facility hazardous waste management procedures, Attachment C of the Chemical Hygiene Plan, Section WS003, Disposal of Samples After Analysis.

16. REFERENCES

16.1. Methods for the Chemical Analysis of Water and Wastes, EPA-600/4-79-020, March 1979: Methods 160.1, 160.2, 160.3, and 160.4.

16.2. Standard Methods for the Examination of Water and Wastewater, 18th Edition, 1992: Methods 2540A, 2540B, 2540C, 2540D, and 2540E.

16.3. QA Policy QA-003, Quanterra QC Program.

16.4. QA Policy QA-004, Rounding and Significant Figures.

16.5. QA Policy QA-005, Method Detection Limits.

16.6. SAC-QA-0041 Calibration and Calibration Check of Balances, Revision 1.1.

16.7. LP-CAL-0001 Laboratory Documentation Practices, Revision 3.0.

16.8. QA Policy 003 Quanterra Quality Control Program, Revision 1.

16.9. QA Policy 008 Data Recording Requirements, Rev. 0.

16.10. Chemical Hygiene Plan, Attachment C, Hazardous Waste Management Procedures, Revision 0.

17. MISCELLANEOUS (TABLES, APPENDICES, ETC.)

17.1. Modifications/Interpretations From Reference Method

17.1.1. Samples are not taken through the drying, cooling, and weighing cycle more than three times. If the sample fails to reach constant weight within three cycles, this is documented in a Nonconformance Memo and the lowest final weight is used for the calculation.

17.1.2. The filter cleaning procedure outlined in Method 160.1 is not applied. This SOP requires the use of method blanks to document system cleanliness.

17.2. Modifications From Previous SOP

17.2.1. Constant weight shall be determined after weight loss is less than 1.0 mg rather than 0.5 mg as specified in Sections 11.10.2.7 and 11.11.3.12.

17.2.2. All entries shall be entered into the TDS and TS Benchsheet Software program. Software program completes all calculations.

17.2.3. Section 7.2 includes LCS solutions used at Quanterra, West Sacramento. Sections 7.3 and 9.6 describe the spike solutions and quality control for matrix spikes.

17.2.4. Sections 8.3, 13.2, 11.12.2.1, and 11.12.3.2 describe the 100 mL sample used for TSS rather than 250 mL used in previous SOP and raises the Reporting Limit to 5 ppm, rather than 4 ppm.

17.2.5. TSS filters are placed in a glass weighing dish rather than an aluminum one in Sections 11.12.1.4 and 11.12.3.6.

17.2.6. Figure 1: Decision Tree for Failed EC/TDS Ratio has been included.

Figure 1: Decision Tree for Failed EC/TDS Ratio

STEP 1: Investigate the physical properties of the sample.

1. Take good notes on your benchsheet during pouring and after drying.

   1.1. Floatables, sediment content, viscosity, color, reaction to air exposure, smell, etc. are enough to cause a failed EC/TDS ratio.

   1.2. What happened during the process? Did the sample spill, splatter, too much sediment?

      1.2.1. If analyst error occurs, stop and reanalyze immediately.

   1.3. What is the sample's appearance after drying? Hygroscopic crystals apparent? Hard to dry? Ring around crucible? Fine sediment persistent?

   NOTE: For samples high in sediment, do not shake the samples prior to analysis.

STEP 2: Always check initial EC, when in doubt reanalyze for EC.

1. If EC is < 20 umhos/cm with TDS of < 100 ppm: Report result with an anomaly stating the EC value is <100 umhos/cm and therefore the EC/TDS ratio criteria does not apply.

2. If EC is between 20 umhos/cm and 100 umhos/cm with TDS < 150% of EC: Report result with an anomaly stating the EC value is <100 umhos/cm and therefore the EC/TDS ratio criteria does not apply.

3. If EC is between 20 umhos/cm and 100 umhos/cm with TDS > 150% of EC: Reanalyze TDS.

   3.1. If reanalysis confirms the original TDS result, report the original run with documentation on both datasheets that reanalysis confirms TDS results with failed EC/TDS ratio.

   3.2. If reanalysis does not confirm the original TDS result, and the rerun analysis passes EC/TDS criteria, report the rerun. If holding times were violated, file an anomaly stating the internal QC failed in the original run.

4. If EC is > 20,000 umhos/cm (oversaturated), dilute the sample at 10x and rerun for EC. Recalculate EC/TDS ratio using the rerun results for EC.

   4.1. If new ratio passes, report results with documentation in the data that the sample was diluted and rerun for EC due to high EC and possibly saturation of salts.

   4.2. If new ratio still fails, rerun TDS.

      4.2.1. If reanalysis confirms the original TDS result, report the original run with notes on both datasheets that reanalysis confirms TDS results with failed EC/TDS ratio.

4.2.2. If reanalysis does not confirm the original TDS result, and the rerun analysis passes EC/TDS criteria, report the rerun. If holding times were violated, file an anomaly stating the internal QC failed in the original run.

5. For failed TDS with EC > 100 umhos/cm, but < 20,000 umhos/cm, rerun EC first before reanalyzing TDS. Keep TDS holding times (7 days) in mind.

5.1. If rerun EC confirms original, and EC/TDS ratio still fails, rerun TDS.

5.2. If rerun EC > 20% RPD from the original EC result, and EC/TDS ratio passes, report TDS result and record new EC result.

5.3. If EC/TDS still fails, rerun TDS.

STEP 3: Only when the EC evaluations and reanalyses are complete should you determine whether to reanalyze for TDS.

STEP 4: If none of the above scenarios apply and you need additional guidance, see your supervisor.

17.3. Tables

Table 1: Summary of Quality Control Requirements for Solids Determinations

QC Samples	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	1 per batch of up to 20 samples	< ± RL	Reanalyze all samples associated with unacceptable MB
Laboratory Control Sample (LCS)	1 per batch of up to 20 samples	80 - 120 % until in-house limits established	Reanalyze all samples associated with unacceptable LCS
Matrix Spikes (MS/MSD)	1 pair per batch of up to 20 samples	75-125% , RPD ≤ 20%	Spiked analytes must be within control limits in the LCS. Re-extraction of the blank, an LCS, the selected field sample, the MS/MSD may be required after evaluation and review.
Duplicate (D)	1 per batch of up to 20 samples, substituted when insufficient sample volume is provided to process an MS/MSD pair	≤ 20% RPD when results > 5x RL	Narrate the results

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Enseco
A CORNING Company

STANDARD OPERATING PROCEDURE

Subject or Title:
Chemical Oxygen Demand (Method 410.4)

SOP No.:	Revision No.:	Effective Date:
LM-CAL-1019	Original	March 19, 1990

Supersedes: None

1. SCOPE AND APPLICATION

1.1 Analytes

This method measures the oxygen equivalent of materials present in water that are subject to oxidation by a strong chemical oxidant.

1.2 Reporting Limit

The reporting limit is 5 mg/L.

1.3 Applicable Matrices

This method is applicable to surface waters, as well as industrial and domestic wastewaters.

1.4 Dynamic Range

This method covers the ranges between 5 and 1500 mg/L COD. Higher concentrations can be determined by dilution.

1.5 Analysis Time

The analytical time is approximately 4 hours for a batch of 20 samples.

Prepared By: John Disney	Date: 3/16/90
Management Approval:	Date: 3/19/90
QA Officer Approval:	Date: 3/19/90

2. SUMMARY OF METHOD

2.1 Organic and oxidizable inorganic substances in an aqueous sample are digested and oxidized by potassium dichromate in 50% sulfuric acid in a sealed vial at 150°C. After two hours of digestion, tubes are removed from the digestor, cooled and measured colorimetrically at both high and low level ranges. The measurement is an indication of excess dichromate remaining in the vial after digestion.

2.2 The low level curve ranges from 5 to 150 mg/L and is read at 420 nm. The high level curve ranges from 100 to 1500 mg/L and is read at 620 nm wavelength. Different Hach COD vials are used for each level.

3. COMMENTS

3.1 Interferences

3.1.1 Low levels of organic matter contamination in water or on glassware may give bad results.

3.1.2 Volatile materials may be lost during sampling and storage. Samples should completely fill a glass bottle and be preserved to pH <2 with H_2SO_4.

3.1.3 Chloride may give false positive results. Mercuric sulfate in the Hach vials will prevent interference of up to 2000 ppm chloride. If chloride is higher than 2000 ppm, dilutions must be made to reduce this interference.

3.2 Helpful Hints

3.2.1 This method is based on the reduction of Cr (VI) to Cr (III). Readings at 420 nm represent the reduction of Cr (VI) in the low curve. The high curve reads at 620 nm to represent the increase in Cr (III).

3.2.2 COD is related to TOC and BOD. For rough comparison purposes, COD = 1.6 x BOD; BOD = 1.6 x TOC.

3.2.3 Some inorganic compounds may contribute to COD thus indicating a higher level of organic contamination.

4. SAFETY ISSUES

4.1 All Cal employees are expected to follow the Enseco GGC policy. Lab coats, gloves, and glasses are required when working in the lab.

4.2 COD vials contain sulfuric acid as well as salts of mercury, silver, and dichromate. Consult MSDS and handle with extreme care. Return used tubes to the original containers so they can be sent off-site for disposal.

4.3 Run analysis in a fume hood.

4.4 When mixing samples in the vials, wrap paper towels around the vials to avoid burns and possible breakage.

5. SAMPLE COLLECTION AND PRESERVATION

5.1 Samples should be collected in glass bottles, preserved to pH less than 2 and stored at 4°C. Minimize head space.

5.2 Holding time for COD is 28 days.

6. APPARATUS

6.1 Hach block digestor for 16 mm tubes set at 150°C ± 2°C.

6.2 Spectrophotometer to measure 16 mm tubes at 420 and 620 nm.

7. REAGENTS AND STANDARDS

7.1 Premixed Hach low range COD tubes #21258-15.

7.2 Premixed Hach high range + COD tubes #21259-15.

7.3 Deionized water which contains less than 0.3 mg/L organic carbon. Do not store water in plastic bottles. Distilled water may be used.

7.4 1500 ppm COD stock standard. Dry potassium phthalate (KHP) and desiccate. Use 1.275 g to 1000 mL and preserve with 2 mL H_2SO_4.

7.5 1000 ppm COD stock standard. Use 0.85 g KHP to 1 liter with 2 mL H_2SO_4.

7.6 COD working standards. Dilute stock solutions in volumetrics as follows:

Concentration (mg/L)	Volume of Stock (mL)	Final Volume (mL)
0	0	100
5	5 mL 100 ppm	100
10	10 mL 100 ppm	100
50	50 mL 100 ppm	100
100	100 mL 100 ppm	100
150	15 mL 1000 ppm	100
500	50 mL 1000 ppm	100
1000	100 mL 1000 ppm	100
1500	100 mL 1500 ppm	100

7.7 Reference Solution

Use ERA demand sample diluted according to instructions supplied with the vial.

8. PROCEDURE

8.1 Preheat block digestor to 150°C.

8.2 Prepare both a high range curve consisting of a blank, 100, 150, 500, 1000, and 1500 mg/L standards and a low range curve consisting of a blank, 5.0, 10, 50, 100, and 150 mg/L. Mid-range ERA reference standards are to be run on both ranges.

8.3 Pipet 2.0 mL of standard/sample into each vial (one low range, one high range). Replace cap tightly, wrap in paper towel and shake vigorously. Place vials in digestor block for 2 hours.

8.4 Turn on spectrophotometer and allow 20 minutes for warm-up.

8.5 Remove vials to a cooling rack for approximately 20 minutes.

8.6 Read absorbance for the low range vials at 420 nm and high range vials at 620 nm.

8.7 Record absorbances to 0.001 units. Perform standard checks every 10 samples for each curve.

9. QA/QC REQUIREMENTS

9.1 A blank is required for each curve and must accompany no more than 20 samples.

9.2 Duplicate Control Samples (DCS) are required for every 20 samples. Single Control Samples (SCS) are required for every batch which doesn't include DCS. Separate DCS/SCS are prepared for each curve.

9.3 A blank and standard check must bracket every run and be after every 10 samples.

9.4 If QC spikes are required, use a spike concentration of 100 mg/L.

9.5 Acceptance Criteria

9.5.1 Blanks must fall below the reporting limit (5 mg/L).

9.5.2 DCS and SCS recoveries must fall within 85-115%.

9.5.3 Relative Percent Difference (RPD) of the DCS must be less than 10 for the high curve and less than 15 for the low curve.

9.5.4 Standard checks must be within 10%.

9.5.5 Calibration standards must give a correlation coefficient of ± 0.995 or better.

10. CALCULATIONS

10.1 Enter concentrations and absorbances of the standards into a linear least squares program. Notice the slope of the low range curve is negative while that of the high range curve is positive.

10.2 Calculate sample concentrations from the calibration curves and multiply by dilution factors.

10.3 Note that negative absorbances on the low range curve indicate an over-range reading. Therefore the high range reading should be used.

11. REPORTING

11.1 Results are reported in mg/L.

11.2 For all dilutions, the reporting limit is multiplied by the dilution factor.

11.3 Use 3 significant figures for values of 10 or greater. Report all DCS and SCS with 3 significant figures.

11.4 Document all non-standard procedures such as odd dilutions, interference problems, etc.

12. REFERENCES

12.1 Sources

12.1.1 EPA Method 410.4.

12.1.2 The Hach Corporation recommended method.

Curtis & Tompkins, Ltd.

SOP FOR BIOCHEMICAL OXYGEN DEMAND (BOD)

SCOPE: This SOP covers determination of Biochemical Oxygen Demand (BOD) in samples. BOD is the amount of dissolved oxygen required by bacteria to consume biodegradable material over a period of five days at 20 degrees C. Several dilutions of a sample are prepared with nutrient-rich dilution water. The initial dissolved oxygen is measured with an electrode. The samples are then incubated at 20 degrees C for five days. The final dissolved oxygen is measured by electrode and the BOD is calculated.

REFERENCES: EPA 405.1

LIMS PRODUCT NAME: BOD
LINKED SPREADSHEETS: NONE
REPORTING LIMIT: 5.0 mg/L

QC REQUIREMENTS: Two glucose/glutamic acid blank spikes (LCS's) are prepared and reported with each batch. A seeded blank is prepared with each batch. Final DO must be at least 1 mg/L for a dilution to be used in reporting. Oxygen depletion must be at least 1 mg/L for a dilution to be used in reporting. A duplicate sample must be run with each batch and the %RPD must be less than 10%. The temperature of the incubator must be maintained at 20 degrees C +/- 1 degree. A seed control blank must be analyzed with each batch.

SAFETY: Wear protective gloves when preparing the sample. Avoid skin and eye contact with all samples.

APPARATUS:
1. 300 mL BOD bottles with pointed ground-glass stoppers.
2. Racks for transporting BOD bottles.
3. Incubator, at 20 degrees C +/- 1 degree.
4. Class A volumetric pipets.
5. Volumetric glassware.
6. Magnetic stirrer with stir bars.
7. Graduated cylinders
8. 19-liter plastic container for dilution water.
9. Tygon tubing for siphon.
10. Aerator with pump
11. pH meter
12. Dissolved Oxygen electrode
13. 500 mL Erlenmeyer flasks.

14. Buret for sodium thiosulfate titrant.
15. Hach DPD test kit for chlorine residual determination.

REAGENTS:

1. Milli-Q water
2. Nutrient buffer pillows for BOD, commercially available from HACH; add contents of one pillow to 19 liters of Milli-Q water.
3. Glucose/glutamic acid solution:
   Dry reagent-grade glucose and glutamic acid in the 105 degree oven for one hour. Dissolve 0.15 g glucose and 0.15 g glutamic acid in deionized water and dilute to 1 L in a volumetric flask. Mix well. Store in a refrigerator.
4. Seed:
   BOD Seed Inoculum (Polyseed). Dissolve one capsule in 500 mL of the dilution water. Stir for 1 hour. prepare daily; solution will be usable for only six hours.

PROCEDURE:

1) Dilution water vessel preparation:

a. Fill vessel with hot tap water and a cup of bleach. Put the tygon tubing in the vessel.
b. Let soak for 30 minutes.
c. Empty and rinse thoroughly with hot water. It is extremely important that no trace of bleach remains.
d. Rinse thoroughly with organic free DI water.
e. Perform cleaning once per month or whenever the average dissolved oxygen depletion in the blanks exceeds 0.2.

2) Dilution water preparation:

a. Dispense the Milli-Q water into a 19 liter container.
b. Add contents of nutrient powder pillow.
c. Aerate for 45 minutes.

3) Seed Preparation:

a. Add contents of one polyseed capsule to 500 mL of prepared dilution water.
b. Mix with stir bar and aerate for one hour minimum.

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4) Sample dilution:

a. Determine COD on sample - one hour reaction time is sufficient as a screen for BOD analysis.
b. Determine the pH of the sample. It should lie between 6.5 and 8.0. If it does not, adjust the H with sulfuric acid or sodium hydroxide.
c. check for residual chlorine with potassium iodide/starch test paper, or use Hach DPD test kit, following kit instructions.
d. If residual chlorine is found, treat the sample with a predetermined amount of sodium sulfite solution.
e. Collect enough BOD bottles for the batch. Allow 2 bottles for blank, 1 bottle for a seeded blank, 2 bottles for an LCS, 3 bottles for each sample, and 3 bottles for each sample duplicate.
f. Documentation for benchbook includes: The "date off" should be five days after the "date set". Include columns for the bottle numbers, the sample ID, the initial DO, the sample volume, the depletion, and the BOD (mg/L) for each bottle.
g. Allow the sample to reach room temperature.
h. mix each sample well.
i. Prepare serial dilutions in accordance with COD values, see Appendix A.
j. Use Class A volumetric pipets to deliver difference volumes of each sample to three BOD bottles. The volumes can be selected by referring to historical results. The goal is to obtain oxygen depletions of at least 1 mg/L with a final DO of at least 1 mg/L.
k. Add 25 mL of seed directly to the seed blank.
l. Add 5 mL of the glucose/glutamic acid solution the first LCS and 10 mLs of glucose/glutamic acid solution to the second LCS.
m. Add 2 mL of seed to each of the LCS bottles and each sample bottle. Do not add seed to the blanks.
n. Carefully fill each bottle with the prepare dilution water using a siphon. Let the open end of the siphon extend below the surface of the water in the bottles during filling. Take care not to introduce air bubbles.

4. Determination of initial dissolved oxygen:

a.Connect the dissolved oxygen electrode to the pH meter.

Note: The oxygen electrode nests in a funnel with a built-in stirrer. The funnel provides an overflow reservoir for water displaced when the electrode is inserted in a bottle.

Note: There is a five position switch at the top of the electrode: OFF, BT CK (for battery check), ZERO (for zero calibration), AIR and H2O (to take readings).

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Note: When not in use, the electrode should be stored in a BOD bottle partially filled with DI water. Do not immerse the electrode. Switch the electrode to the "OFF" position.

b. With the electrode switch in the "OFF" position, switch the pH meter to pH mode. The meter will read 7.00.
c. Turn the electrode switch to "BT CK" and verify that the battery reading is 13 or greater. If less than 13, replace battery.
d. Turn the electrode switch to "zero". Use the "zero calibration control" to set the meter to read 0.00.
e. Turn the electrode switch to "AIR". Read out should be 7.60. If not, adjust with knob, wait for stable reading and probe is now calibrated.
f. Turn electrode switch to "H2O".
g. Place the BOD bottles containing the seed blanks, LCS, samples, and replicates on the magnetic stirrer one by one. Carefully insert the funnel and electrode and record the initial DO reading for each in the benchbook.
h. Carefully stopper each bottle. Make sure that the BOD bottles are water sealed and that there are no air bubbles inside. Cover each stopper with a plastic cap and write the "date off" on the cap. The "date off" is five days after today's date. Transport all BOD bottles to the incubator.

5. Determination of final dissolved oxygen:

a. Five days after they were wet, retrieve the BOD bottles from the incubator.
b. Follow meter and probe calibrations as outlined in E.4.a through f.
c. Document in benchbook.
d. Place the BOD bottles containing the seed blanks, LCS samples and replicates on the magnetic stirrer one by one. Carefully insert the funnel and electrode and record the final DO reading for each in the benchbook.

CALCULATION and Data Review:

1. Subtract the final DO from the initial DO for each bottle. Record the results in the "depletion" column in the benchbook

2. Calculate the average initial DO of the regular blanks (DO.AVG). Calculate the average final DO of the seeded blanks (SEED.AVG). Calculate the seed correction factor (SCF) as follows:

$$SCF = \frac{(DO.AVE) - (SEED.AVG)}{5}$$

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3. Calculate the BOD for each bottle as follows:

$$BOD = \frac{(\text{Initial DO}) - (\text{Final DO}) - (\text{SCF})}{\text{Sample volume (in mL)}} \times 300$$

4. The reported BOD for a sample is the average of the BOD's for each sample dilution that satisfies the QC requirements. If only one dilution is satisfactory, its BOD will be the reported BOD.

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QUANTERRA STANDARD OPERATING PROCEDURE

TITLE: GC/MS ANALYSIS BASED ON METHODS 8270C AND 625

(SUPERSEDES: Revision 1)

Prepared by: Richard Burrows

Reviewed by:_____
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C4-443

TABLE OF CONTENTS

1 SCOPE AND APPLICATION 1
2 SUMMARY OF METHOD 1
3 DEFINITIONS 1
4 INTERFERENCES 1
5 SAFETY PRECAUTIONS 1
6 EQUIPMENT AND SUPPLIES 1
7 REAGENTS AND STANDARDS 1
8 SAMPLE PRESERVATION AND STORAGE 1
9 QUALITY CONTROL 1
10 CALIBRATION AND STANDARDIZATION 1
11 PROCEDURE 1
12 DATA ANALYSIS AND CALCULATIONS 1
13 METHOD PERFORMANCE 1
14 POLLUTION PREVENTION 1
15 WASTE MANAGEMENT 1
16 REFERENCES 1
17 MISCELLANEOUS 1
18 REQUIREMENTS FOR METHOD 625 1

LIST OF TABLES

TABLE 1	Quanterra Primary Standard and Standard Reporting Limits
TABLE 2	Quanterra Appendix IX Standard and Standard Reporting Limits
TABLE 3	Reportable Analytes for Quanterra Standard Tests, Primary Standard
TABLE 4	Reportable analytes for Quanterra Standard Tests, Appendix IX Standard
TABLE 5	Recommended Instrument Conditions
TABLE 6	DFTPP Ion Abundance Criteria
TABLE 7	Characteristic Ions, Primary Standard
TABLE 8	Characteristic Ions, Appendix IX Standard
TABLE 9	8270B LCS Compounds
TABLE 10	TCLP LCS Compounds
TABLE 11	8270B Surrogate Compounds
TABLE 12	Calibration Levels, Primary Standard
TABLE 13	Calibration Levels, Appendix IX Standard
TABLE 14	Initial Demonstration Accuracy and Precision Limits
TABLE A-1	Method 625 Reporting List and Limits
TABLE-A-2	Method 625 LCS and MS Compounds and Spike Concentrations

1

SCOPE AND APPLICATION

1.1 This method is based upon SW846 8270C, and is applicable to the determination of the concentration of semivolatile organic compounds in extracts prepared from solid and aqueous matrices. The modifications presented in Attachment A may be followed for analysis of wastewater following method 625. Direct injection of a sample may be used in limited applications. Refer to Tables 1, 2, 3 and 4 for the list of compounds applicable for this method. Note that the compounds are listed in approximate retention time order. Additional compounds may be amenable to this method. If non-standard analytes are required, they must be validated by the procedures described in section 13 before sample analysis.

1.2 The following compounds may require special treatment when being determined by this method:

- Benzidine can be subject to oxidative losses during solvent concentration and exhibits poor chromatography. Neutral extraction should be performed if this compound is expected.
- Hexachlorocyclopentadiene is subject to thermal decomposition in the inlet of the gas chromatograph, chemical reaction in acetone solution, and photochemical decomposition.
- N-Nitrosodiphenylamine decomposes in the gas chromatographic inlet and cannot be distinguished from diphenylamine.
- Pentachlorophenol, 2,4-dinitrophenol, 4-nitrophenol, 4,6-dinitro-2-methylphenol, 4-chloro-3-methylphenol, benzoic acid, 2-nitroaniline, 3-nitroaniline, 4-chloroaniline, and benzyl alcohol are subject to erratic chromatographic behavior, especially if the GC system is contaminated with high boiling material.
- Hexachlorophene is not amenable to analysis by this method.
- 3-Methylphenol cannot be separated from 4-methylphenol by the conditions specified in this method.

1.3 The standard reporting limit (SRL) of this method for determining an individual compound is approximately 0.33 mg/kg (wet weight) for soil/sediment samples, 1 - 200 mg/kg for wastes (dependent on matrix and method of preparation), and 10 µg/L for groundwater samples. Some compounds have higher reporting limits. Refer to Tables 1 and 2 for specific SRLs. Reporting limits will be proportionately higher for sample extracts that require dilution.

2 SUMMARY OF METHOD

2.1 Aqueous samples are extracted with methylene chloride using a separatory funnel, a continuous extractor or Accelerated One-Step™. Solid samples are extracted with methylene chloride / acetone using sonication, soxhlet, accelerated soxhlet or

pressurized fluid extraction. Waste dilution is used for samples that are miscible with the solvent. The extract is dried, concentrated to a volume of 1 mL, and analyzed by GC/MS. Extraction procedures are detailed in SOP# CORP-OP-0001. Qualitative identification of the parameters in the extract is performed using the retention time and the relative abundance of characteristic ions. Quantitative analysis is performed using the internal standard technique with a single characteristic ion.

3 DEFINITIONS

3.1 CCC (Calibration Check Compounds) - A subset of target compounds used tc evaluate the calibration stability of the GC/MS system. A maximum percent deviation of the CCC's is specified for calibration acceptance.

3.2 SPCC (System Performance Check Compounds) - Target compounds designated to monitor chromatographic performance, sensitivity, and compound instability or degradation on active sites. Minimum response factors are specified for acceptable performance.

3.3 Batch - The batch is a set of up to 20 samples of the same matrix processed using the same procedures and reagents within the same time period. The Quality Control batch must contain a matrix spike / spike duplicate (MS/MSD), a Laboratory Control Sample (LCS), and a method blank. In some cases, at client request, the MS/MSD may be replaced with a matrix spike and sample duplicate. Batches are defined at the sample preparation stage. Batches should be kept together through the whole analytical process to the extent possible, but it is not mandatory to analyze prepared extracts on the same instrument or in the same sequence. Refer to the Quanterra QC Program document (QA-003) for further details of the batch definition.

3.4 Method Blank - An analytical control consisting of all reagents, internal standards and surrogate standards, that is carried through the entire analytical procedure. The method blank is used to define the level of laboratory background and reagent contamination.

3.5 LCS (Laboratory Control Sample) - A blank spiked with the parameters of interest that is carried through the entire analytical procedure. Analysis of this sample with acceptable recoveries of the spiked materials demonstrates that the laboratory techniques for this method are acceptable.

3.6 MS (Matrix Spike)- aliquot of a matrix (water or soil) fortified (spiked) with known quantities of specific compounds and subjected to the entire analytical procedure in order to indicate the appropriateness of the method for the matrix by measuring recovery.

3.7 MSD (Matrix Spike Duplicate)- a second aliquot of the same sample as the matrix spike (above) that is spiked in order to determine the precision of the method.

4 INTERFERENCES

4.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Raw GC/MS data from all blanks, samples, and spikes must be evaluated for interferences. If an interference is detected it is necessary to determine if the source of interference is in the preparation and/or cleanup of the samples; then take corrective action to eliminate the problem.

4.2 The use of high purity reagents, solvents, and gases helps to minimize interference problems.

4.3 Matrix interferences may be caused by contaminants that are coextracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature of the sample.

4.4 Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. To reduce carryover, the sample syringe must be rinsed with solvent between samples. Whenever an unusually concentrated sample is encountered, it should be followed by the analysis of solvent to check for cross contamination.

4.5 Phthalate contamination is commonly observed in this analysis and its occurrence should be carefully evaluated as an indicator of a contamination problem in the sample preparation step of the analysis.

5 SAFETY PRECAUTIONS

5.1 Procedures shall be carried out in a manner that protects the health and safety of all Quanterra associates. The following requirements must be met:

5.1.1 Eye protection that satisfies ANSI Z87.1 (as per the Chemical Hygiene Plan), laboratory coat, and appropriate gloves must be worn while samples, standards, solvents and reagents are being handled. Disposable gloves that have become contaminated will be removed and discarded; other gloves will be cleaned immediately.

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

5.1.3 Chemicals that have been classified as carcinogens, or potential carcinogens,

under OSHA include: Benzo(a)anthracene, benzidine, 3,3'-dichlorobenzidine, benzo(a)pyrene, dibenzo(a,h)anthracene, and n-nitrosodimethylamine. Primary standards should be purchased in solution. If neat materials must be obtained, they shall be handled in a hood.

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

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

6 EQUIPMENT AND SUPPLIES

6.1 Gas Chromatograph/Mass Spectrometer System: An analytical system complete with a temperature-programmable gas chromatograph suitable for split/splitless injection and all required accessories, including syringes, analytical columns, and gases. The capillary column should be directly coupled to the source.

6.2 Column: 30 m x 0.32 mm I.D. (or 0.25 mm I.D.) 0.5-µm film thickness silicon-coated fused-silica capillary column (J & W Scientific DB-5.625 or equivalent). Alternate columns are acceptable if they provide acceptable performance.

6.3 Mass Spectrometer: Capable of scanning from 35 to 500 AMU every one second or less, using 70 volts (nominal) electron energy in the electron impact ionization mode. The mass spectrometer must be capable of producing a mass spectrum for decafluorotriphenylphosphine (DFTPP) which meets all of the criteria in Table 6 when 50 ng of the GC/MS tuning standard is injected through the GC.

6.4 GC/MS Interface: Any GC-to-MS interface that gives acceptable calibration points and achieves acceptable tuning performance criteria may be used.

6.5 Data System: A computer system must be interfaced to the mass spectrometer. The system must allow the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer must have software that can search any GC/MS data file for ions of a specific mass and that can plot such ion abundances versus time or scan number. This type of plot is defined as the Extracted Ion Current Profile (EICP). Software must also be available that allows integrating the abundances in any EICP between specified time or scan-number limits. The most recent version of the EPA/NIH Mass Spectral Library is recommended.

6.6 Syringe: 10 µL Hamilton Laboratory grade syringes or equivalent.

6.7 Carrier gas: Ultra high purity helium.

7 REAGENTS AND STANDARDS

7.1 A minimum five point calibration curve is prepared. The low point should be at or below the reporting limit. Refer to Tables 12 and 13 for typical calibration levels for all analytes. Other calibration levels may be used, depending on instrument capability, but the low standard must support the reporting limit and the high standard defines the range of the calibration.

7.2 An Internal Standard solution is prepared. Compounds in the I.S. Mix are: acenaphthene-d10, chrysene-d12, 1,4-dichlorobenzene-d4, naphthalene-d8, perylene-d12, and phenanthrene-d10.

7.2.1 Internal Standards are added to all standards and extracts to result in 40ng injected onto the column. For example, if the volume of an extract used was 200 µL, 20 µL of a 400 µg/mL internal standard solution would be added for a 1 µL injection.

7.3 Surrogate Standard Spiking Solution: Prepare as indicated in the preparative methods. See appropriate preparation SOP. Surrogate compounds and levels are listed in Table 11.

7.4 GC/MS Tuning Standard: A methylene chloride solution containing 50 µg/mL of decafluorotriphenylphosphine (DFTPP) is prepared. Pentachlorophenol, benzidine, and DDT, should also be included in the Tuning Standard at 50 µg/mL.

7.5 Laboratory Control Spiking Solution: Prepare as indicated in the preparative methods. See appropriate preparation SOP. LCS compounds and levels are listed in Tables 9 and 10.

7.6 Matrix Spike Solution: Prepare as indicated in the preparative methods. See preparation SOP. The matrix spike compounds and levels are the same as the LCS compounds.

7.7 The standards listed in 7.1 to 7.6 should be refrigerated at $\leq 6^{\circ}C$ when not in use. Refrigeration at $-10^{\circ}C$ to $-20^{\circ}C$ may be used if it can be demonstrated that analytes do not fall out of solution at this temperature. The standards must be replaced at least once a year. The continuing calibration standard must be replaced every week and is stored at $\leq 6^{\circ}C$.

8 SAMPLE PRESERVATION AND STORAGE

8.1 Reference appropriate facility SOP for sample bottle preservation and storage.

8.2 Samples are stored at $4 \pm 2^{\circ}C$. Samples and extracts should be stored in suitable glass containers with Teflon lined caps. (Extracts will normally be stored for 30 days after invoicing.)

8.3 Water samples are extracted within seven days of sampling and the extracts are analyzed within forty days of extraction. Solids, sludges, and organic liquids are extracted within fourteen days of sampling and the extracts are analyzed within forty days of extraction.

9 QUALITY CONTROL

9.1 Initial Demonstration of Capability

9.1.1 For the standard analyte list, the initial demonstration and method detection limit (MDL) studies described in section 13 must be acceptable before analysis of samples may begin. Refer to the flow chart in section 17.4.1.

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

9.2 Control Limits

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

9.2.1 These limits do not apply to dilutions (except for tests without a separate extraction), but surrogate and matrix spike recoveries will be reported unless the dilution is more than 5X.

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

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

9.3 Method Blank

A method blank is prepared and analyzed with each batch of samples. The method blank consists of reagent water for aqueous samples, and sodium sulfate for soil samples (Refer to SOP No. CORP-OP-0001 for details). Surrogates are added and the method blank is carried through the entire analytical procedure. The method blank must not contain any analyte of interest at or above the reporting limit (except common laboratory contaminants, see below) or at or above 5% of the measured concentration of that analyte in the associated samples, whichever is higher.

- If the analyte is a common laboratory contaminant (phthalate esters), the data may be reported with qualifiers if the concentration of the analyte is less than five times the RL. Such action must be taken in consultation with the client.
- Reanalysis of any samples with reportable concentrations of analytes found in the method blank is required unless other actions are agreed with the client.
- If there is no target analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. Such action should be taken in consultation with the client.

9.3.1 The method blank must have acceptable surrogate recoveries. If surrogate recoveries are not acceptable, the data must be evaluated to determine if the method blank has served the purpose of demonstrating that the analysis is free of contamination. If surrogate recoveries are low and there are reportable analytes in the associated samples, re-extraction of the blank and affected samples will normally be required. Consultation with the client should take place.

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

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

9.3.4 Sample results are NOT blank subtracted unless specific requests and arrangements have been made with a client or agency.

9.4 Instrument Blank

9.4.1 Instruments must be evaluated for contamination during each 12 hour analytical run. This may be accomplished by analysis of a method blank. If a method blank is not available, an instrument blank must be analyzed. An instrument blank consists of methylene chloride with the internal standards added. It is evaluated in the same way as the method blank.

9.5 Laboratory Control Sample (LCS)

9.5.1 A laboratory control sample (LCS) is prepared and analyzed with every batch of samples. All analytes must be within established control limits. The LCS is spiked with the compounds listed in Tables 9 and 10 unless specified by a client or agency. The compounds must be spiked at a concentration equivalent to 100 or 150 ng on-column depending on the analyte.

9.5.2 If any analyte in the LCS is outside the laboratory established historical control limits, corrective action must occur. Corrective action may include re-extraction and reanalysis of the batch.

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

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

9.5.3 Ongoing monitoring of the LCS provides evidence that the laboratory is performing the method within accepted QC guidelines for accuracy and precision.

9.6 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

A matrix spike/matrix spike duplicate (MS/MSD) is prepared and analyzed with every batch of samples. The MS/MSD is spiked with the same subset of analytes as the LCS (See Tables 9 and 10). Compare the percent recovery and relative percent difference (RPD) to that in the laboratory specific historically generated limits.

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

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

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

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

9.7 Surrogates

9.7.1 Every sample, blank, and QC sample is spiked with surrogate standards. Surrogate spike recoveries must be evaluated by determining whether the concentration (measured as percent recovery) falls within the required recovery limits. Surrogate compounds must be spiked at either 100 or 150 ng on-column, depending on the surrogate. The compounds routinely included in the surrogate spiking solution, along with recommended standard concentrations, are listed in Table 11.

9.7.2 If any surrogates are outside limits the following corrective actions must take

place (except for dilutions):

- Check all calculations for error.
- Ensure that instrument performance is acceptable.
- Recalculate the data and/or reanalyze the extract if either of the above checks reveal a problem.
- Re-extract and reanalyze the sample or flag the data as "Estimated Concentration" if neither of the above resolves the problem.

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

9.7.3 If the sample with surrogate recoveries outside the recovery limits was a sample used for an MS/MSD and the surrogate recoveries in the MS/MSD are also outside of the control limits, then the sample, the MS, and the MSD do not require reanalysis as this phenomenon would indicate a possible matrix problem.

9.7.4 If the sample is reanalyzed and the surrogate recoveries in the reanalysis are acceptable, then the problem was within the analyst's control and only the reanalyzed data should be reported. (Unless the reanalysis was outside holding times, in which case reporting both sets of results may be appropriate.)

9.7.5 If the reanalysis does confirm the original results, the original analysis is reported and the data flagged as estimated due to matrix effect.

9.8 Nonconformance and Corrective Action

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

9.9 Quality Assurance Summaries

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

9.10 Quanterra QC Program

Further details of QC and corrective action guidelines are presented in the Quanterra

QC Program document (QA-003). Refer to this document if in doubt regarding corrective actions.

10 CALIBRATION AND STANDARDIZATION

10.1 Summary

10.1.1 The instrument is tuned for DFTPP, calibrated initially with a five-point calibration curve, and verified each 12-hour shift with one or more continuing calibration standard(s). Recommended instrument conditions are listed in Table 5.

10.2 All standards and extracts are allowed to warm to room temperature before injecting.

10.3 Instrument Tuning

At the beginning of every twelve hour shift when analyses are to be performed, the GC/MS system must be checked to see if acceptable performance criteria (Table 6) is achieved for DFTPP (decafluorotriphenylphosphine).

10.3.1 Inject 50 ng of the GC/MS tuning standard (Section 7.4) into the GC/MS system. Obtain a background-corrected mass spectra of DFTPP and confirm that all the key m/z criteria in Table 6 are achieved. If all the criteria are not achieved, the analyst must retune the mass spectrometer and repeat the test until all criteria are achieved. The performance criteria must be achieved before any samples, blanks, or standards are analyzed.

10.3.2 The GC/MS tuning standard should also be used to evaluate the inertness of the chromatographic system. Benzidine and pentachlorophenol should not exhibit excessive tailing. If DDT is an analyte of interest, it must be included in the tuning standard, and its breakdown must be < 20%. Refer to section 12 for the appropriate calculations.

10.4 Initial Calibration

10.4.1 Internal Standard Calibration Procedure: Internal standards are listed in Table 7. Use the base peak m/z as the primary m/z for quantitation of the standards. If interferences are noted, use one of the next two most intense masses for quantitation.

10.4.2 Compounds should be assigned to the IS with the closest retention time.

10.4.3 Prepare calibration standards at a minimum of five concentration levels for each parameter of interest. Six standards must be used for a quadratic least squares calibration. It may also be useful to analyze six calibration levels and use the

lower five for most analytes and the upper five for analytes that have poor response. Add the internal standard mixture to result in 40 ng on column. (For example, if the volume of the calibration standard used is 1 mL, add 100 µL of the 400 µg/mL internal standard solution for a 1 µL injection). The concentrations of all analytes are listed in tables 12 and 13.

10.4.4 Analyze each calibration standard and tabulate the area of the primary characteristic m/z against concentration for each compound and internal standard. Calculate response factors (RF), average response factors, and the percent RSD of the response factors for each compound using the equations in section 12 and verify that the CCC and SPCC criteria in section 10.4.5 and 10.4.6 are met. **No sample analysis may be performed unless these criteria are met.**

10.4.5 System Performance Check Compounds (SPCCs): The minimum average RF for semivolatile SPCCs is 0.050. If the minimum response factors are not met, the system must be evaluated and corrective action must be taken before sample analysis begins. Some possible problems are standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or chromatographic system. This check must be met before analysis begins.

SPCC Compounds:

N-nitroso-di-n-propylamine
Hexachlorocyclopentadiene
2,4-Dinitrophenol
4-Nitrophenol

10.4.6 Calibration Check Compounds (CCCs): The %RSD of the response factors for each CCC in the initial calibration must be less than 30% for the initial calibration to be considered valid. This criterion must be met before sample analysis begins. Problems similar to those listed under SPCCs could affect this criterion.

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

10.4.6.2 CCC Compounds:

Phenol
Acenaphthene
1,4-Dichlorobenzene
N-nitrosodiphenylamine
2-Nitrophenol
Pentachlorophenol
2,4-Dichlorophenol

Fluoranthene
Hexachlorobutadiene
Di-n-octylphthalate
4-Chloro-3-methylphenol
Benzo(a)pyrene
2,4,6-Trichlorophenol

10.4.7 If the average of all %RSDs in the initial calibration is ≤ 15%, then all analytes may use average response factor for calibration.

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

10.4.7.2 If the average of all the %RSDs in the initial calibration is > 15%, then calibration on a curve must be used for those analytes with %RSD > 15%. Linear or quadratic curve fits may be used. Use of 1/Concentration^2 weighting is recommended to improve the accuracy of quantitation at the low end of the curve. The analyst should consider instrument maintenance to improve the linearity of response. If Relative Standard Error (RSE) is used to evaluate the curve it must be better than 15%. Otherwise the correlation coefficient (coefficient of determination for non-linear curves) must be ≥ 0.990.

10.4.8 Weighting of data points

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

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

10.4.10 Quantitation is performed using the calibration curve or average response factor form the initial curve, not the continuing calibration.

10.5 Continuing Calibration

10.5.1 At the start of each 12-hour period, the GC/MS tuning standard must be analyzed. A 50 ng injection of DFTPP must result in a mass spectrum for DFTPP which meets the criteria given in Table 6.

10.5.2 Following a successful DFTPP analysis the continuing calibration standard(s) are analyzed. The standards must contain all semivolatile analytes, including all required surrogates. A mid level calibration standard is used for the continuing calibration.

10.5.3 The following criteria must be met for the continuing calibration to be acceptable:

- The SPCC compounds must have a response factor of ≥ 0.05.
- The percent difference or drift of the CCC compounds from the initial calibration must be $\leq 20\%$. (see section 12 for calculations) In addition, the percent difference or drift of all analytes must be $\leq 50\%$, with allowance being made for up to six target compounds to have percent drift greater than 50%.
- The internal standard response must be within 50-200% of the response in the mid level of the initial calibration.
- The internal standard retention times must be within 30 seconds of the retention times in the mid-level of the initial calibration.

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

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

11 PROCEDURE

11.1 Sample Preparation

Samples are prepared following SOP CORP-OP-0001.

11.2 Sample Analysis Procedure

11.2.1 Calibrate the instrument as described in section 10. Depending on the target compounds required by the client, it may be necessary to use more than one calibration standard.

11.2.2 All samples must be analyzed using the same instrument conditions as the preceeding continuing calibration standard.

11.2.3 Add internal standard to the extract to result in 40 ng injected on column (for example, 50 µL internal standard solution in 0.5 mL of extract for a 1 µL injection). Mix thoroughly before injection into the instrument.

11.2.4 Inject the sample extract into the GC/MS system using the same injection technique as used for the standards.

11.2.5 The data system will determine the concentration of each analyte in the extract using calculations equivalent to those in section 12. Quantitation is based on the initial calibration, not the continuing calibration.

11.2.6 Identified compounds are reviewed for proper integration. Manual integrations are performed if necessary and are documented by the analyst or automatically by the data system.

11.2.7 Target compounds identified by the data system are evaluated using the criteria listed in section 12.1.

11.2.8 Library searches of peaks present in the chromatogram that are not target compounds (Tentatively Identified Compounds, TIC) may be performed if required by the client. They are evaluated using the criteria in section 12.3.

11.3 Dilutions

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

11.3.1 Guidance for Dilutions Due to Matrix

If the sample is initially run at a dilution and the baseline rise is less than the

height of the internal standards, or if individual non-target peaks are less than two times the height of the internal standards, the sample should be reanalyzed at a more concentrated dilution. This requirement is approximate and subject to analyst judgement. For example, samples containing organic acids may need to be analyzed at a higher dilution to avoid destroying the column.

11.3.2 Reporting Dilutions

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

11.4 Perform all qualitative and quantitative measurements. When the extracts are not being used for analyses, refrigerate them at 4 ± 2°C, protected from light in screw cap vials equipped with unpierced Teflon lined septa.

11.5 Retention time criteria for samples

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

11.5.1 If the retention time of any internal standard in any sample varies by more than 0.1 minute from the preceeding continuing calibration standard, the data must be carefully evaluated to ensure that no analytes have shifted outside their retention time windows.

11.6 Percent Moisture

Analytical results may be reported as dry or wet weight, as required by the client. Percent moisture must be determined if results will be reported as dry weight. Refer to the facility specific SOP for determination of percent moisture.

11.7 Procedural Variations

11.7.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file. Any unauthorized deviations from this procedure must also be documented as a non-conformance, with a cause and corrective action described.

11.8 Troubleshooting Guide

11.8.1 Daily Instrument Maintenance

In addition to the checks listed in the instrument maintenance schedule in the Quanterra QAMP, the following daily maintenance should be performed.

- Clip Column as necessary.
- Install new or cleaned injection port liner as necessary.
- Install new septum as necessary.
- Perform mass calibration as necessary.

11.8.2 Major Maintenance

A new initial calibration is necessary following major maintenance. Major maintenance includes changing the column, cleaning the ion volume or repeller, cleaning the source, and replacing the multiplier. Refer to the manufacturer's manual for specific guidance.

12 DATA ANALYSIS AND CALCULATIONS

12.1 Qualitative identification

An analyte is identified by retention time and by comparison of the sample mass spectrum with the mass spectrum of a standard of the suspected compound (standard reference spectrum). Mass spectra for standard reference may be obtained on the user's GC/MS by analysis of the calibration standards or from the NBS library. Two criteria must be satisfied to verify identification: (1) elution of sample component at the same GC retention time as the standard component; and (2) correspondence of the sample component and the standard component characteristic ions. (Note: Care must be taken to ensure that spectral distortion due to co-elution is evaluated.)

- The sample component retention time must compare to within ± 0.2 min. of the retention time of the standard component. For reference, the standard must be run within the same twelve hours as the sample.
- All ions present in the standard mass spectra at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) should be present in the sample spectrum.
- The characteristic ions of a compound must maximize in the same scan or

within one scan of each other.

- The relative intensities of ions should agree to within ±30% between the standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample abundance must be between 20% and 80%.)

12.1.1 If a compound cannot be verified by all the above criteria, but in the technical judgment of the analyst the identification is correct, the analyst shall report that identification and proceed with quantitation.

12.2 Mass chromatogram searches.

Certain compounds are unstable in the calibration standard and cannot be calibrated in the normal way. In particular, the compound hexachlorophene (CAS 70-30-4) falls into this category, and is required for Appendix IX analysis. For this analyte a mass chromatogram search is made.

12.2.1 Hexachlorophene

Display the mass chromatograms for mass 196 and mass 198 for the region of the chromatogram from at least 2 minutes before chrysene-d12 to at least 4 minutes after chrysene-d12. If peaks for both ions coincide then the analyst evaluates the spectrum for the presence of hexachlorophene. No quantitation is possible.

12.3 For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the type of analyses being conducted. Computer generated library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. Only after visual comparison of sample spectra with the nearest library searches shall the mass spectral interpretation specialist assign a tentative identification. Guidelines for making tentative identification are:

- Relative intensities of major ions in the reference spectrum (ions >10% of the most abundant ion) should be present in the sample spectrum.

- The relative intensities of the major ions should agree within ±20%. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance should be between 30%and 70%.)

- Molecular ions present in the reference spectrum should be present in the sample spectrum.

- Ions present in the sample spectrum, but not in the reference spectrum, should be

reviewed for possible background contamination or presence of coeluting compounds.

- Ions present in the reference spectrum, but not in the sample spectrum, should be reviewed for possible subtraction from the sample spectrum because of background contamination or coeluting peaks. Data system library reduction programs can sometimes create these discrepancies.

- Automatic background subtraction can severely distort spectra from samples with unresolved hydrocarbons.

12.4 Anyone evaluating data is trained to know how to handle isomers with identical mass spectra and close elution times. These include:

Dichlorobenzenes
Methylphenols
Trichlorophenols
Phenanthrene, anthracene
Fluoranthene, pyrene
Benzo(b) and (k)fluoranthene
Chrysene, benzo(a)anthracene

Extra precautions concerning these compounds are to more closely scrutinize retention time vs. the calibration standard and also to check that all isomers have distinct retention times.

A second category of problem compounds would be the poor responders or compounds that chromatograph poorly. Included in this category would be:

Benzoic acid
Chloroanilines
Nitroanilines
2,4-Dinitrophenol
4-Nitrophenol
Pentachlorophenol
3,3'-Dichlorobenzidine
Benzyl alcohol
4,6-Dinitro-2-methylphenol

Manually checking the integrations would be appropriate for these compounds.

12.5 Calculations

12.5.1 Percent Relative Standard Deviation for Initial Calibration

$$\%RSD = \frac{SD}{\overline{RF}} \times 100$$

RF = Mean of RFs from intial caibration for a compound

SD = Standard deviation of RFs from initial calibration for a compound,

$$= \sqrt{\sum_{i=1}^{N} \frac{\left(RFi - \overline{RF}\right)^2}{N-1}}$$

RFi = RF for each of the calibration levels

N = Number of RF values

12.5.2 Continuing calibration percent drift

$$\%Drift = \frac{C_{actual} - C_{found}}{C_{actual}} \times 100\%$$

C_{actual} = Known concentration in standard

C_{found} = Measured concentration using selected quantitation method

12.5.3 Concentration in the extract

The concentration of each identified analyte and surrogate in the extract is calculated from the linear or quadratic curve fitted to the initial calibration points, or from the average RF of the initial calibration.

12.5.3.1 Average response factor

If the average of all the %RSDs of the response factors in the initial calibration is ≤ 15%, the average response factor from the initial calibration may be used for quantitation.

$$C_{ex} = \frac{R_x C_{is}}{R_{is} \overline{RF}}$$

12.5.3.2 Linear fit

$$C_{ex} = A + B\frac{(R_x C_{is})}{R_{is}}$$

C_{ex}= Concentration in extract, µg/mL

R_x= Response for analyte

R_{is}= Response for internal standard

C_{is}= Concentration of internal standard

A= Intercept

B= Slope

12.5.3.3 Quadratic fit

$$C_{ex} = A + B\left(\frac{R_x C_{is}}{R_{is}}\right) + C\left(\frac{R_x C_{is}}{R_{is}}\right)$$

C= Curvature

12.5.4 The concentration in the sample is then calculated.

12.5.4.1 Aqueous Calculation

$$\text{Concentration, } \mu g / \text{L} = \frac{C_{ex} V_t}{V_o}$$

Where:

V_t = Volume of total extract, µL, taking into account dilutions (i.e., a 1-to-10 dilution of a 1 mL extract will mean V_t = 10,000 µL. If half of the base/neutral extract and half of the acid extract are combined, V_t = 2,000.)

V_o = Volume of water extracted (mL)

12.5.5 Sediment/Soil, Sludge (on a dry-weight basis) and Waste (normally on a wet-weight basis:

$$\text{Concentration, } \mu g / \text{kg} = \frac{C_{ex} V_t}{W_s D}$$

W_s = Weight of sample extracted or diluted in grams

D = (100 - % moisture in sample)/100, for a dry weight basis or 1 for a wet weight basis

12.6 MS/MSD percent recovery calculation.

$$\text{Matrix Spike Recovery} = \frac{S_{SR} - S_R}{S_A} \times 100\%$$

S_{SR} = Spike sample result

S_R=Sample result

S_A = Spike added

12.7 Relative % Difference calculation for the MS/MSD

$$RPD = \frac{MS_R - MSD_R}{1/2(MS_R + MSD_R)} \times 100$$

RPD = Relative percent difference

MS_R = Matrix spike result

MSD_R = Matrix spike duplicate result

12.8 Relative response factor calculation.

$$RF = \frac{A_x C_{is}}{A_{is} C_x}$$

A_x=Area of the characteristic ion for the compound being measured

A_{is}=Area of the characteristic ion for the specific internal standard

C_x=Concentration of the compound being measured (µg/L)

C_{is} =Concentration of the specific internal standard (µg/L)

12.9 Calculation of TICs: The calculation of TICs (tentatively identified compounds) is identical to the above calculations with the following exceptions:

A_x=Area of the total ion chromatogram for the compound being measured

A_{is}=Area of the total ion chromatogram for the nearest internal standard without interference

RF=1

12.10 Percent DDT breakdown

$$\% \text{ DDT breakdown} = \frac{\text{DDEarea} + \text{DDDarea}}{\text{DDTarea} + \text{DDEarea} + \text{DDarea}}$$

The total ion current areas are used for this calculation

13 METHOD PERFORMANCE

13.1 Method Detection Limit

Each laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in QA Policy #: QA-005.

13.2 Initial Demonstration

Each laboratory must make an initial demonstration of capability for each individual method. Demonstration of capability for both soil and water matrices is required. This requires analysis of QC check samples containing all of the standard analytes for the method. For some tests it may be necessary to use more than one QC check mix to cover all analytes of interest.

13.2.1 Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to the level 4 calibration standard.

13.2.2 Calculate the average recovery and standard deviation of the recovery for each analyte of interest. Compare these results with the acceptance criteria given in table 14.

13.2.3 If any analyte does not meet the acceptance criteria the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

13.3 Non-standard analytes

For non-standard analytes, an MDL study must be performed and calibration curve generated before analyzing any samples, unless lesser requirements are previously agreed to with the client. In any event, the minimum initial demonstration required is analysis of an extracted standard at the reporting limit and a single point calibration.

13.4 Training Qualification

The group/team leader has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

13.5 Data Quality Objectives (DQO). Refer to project-specific Quality Assurance plans for DQO information.

14 POLLUTION PREVENTION

14.1 This section is not applicable to this procedure.

15 WASTE MANAGEMENT

15.1 Waste generated during aliquotting and from used vials must be disposed of in accordance with the facility hazardous waste procedures. The Health and Safety Director should be contacted if additional information is required.

16 REFERENCES

16.1 SW846, Test Methods for Evaluating Solid Waste, Third Edition, Update II, October 1994, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS): Capillary Column Technique, Method 8270B.

16.2 J. W. Eichelberger, L. E. Harris, and W. L. Budde, "Reference Compound to Calibrate Ion Abundance Measurement in Gas Chromatography/Mass Spectrometry," Analytical Chemistry, 47, 995 (1975)

17 MISCELLANEOUS

17.1 Modifications from Reference Method

17.1.1 A retention time window of 0.2 minutes is used for all components, since some data systems do not have the capability of using the relative retention time units specified in the reference method.

17.1.2 The quantitation and qualifier ions for some compounds have been changed from those recommended in SW-846 in order to improve the reliability of qualitative identification.

17.2 Modifications from Previous Revision

17.2.1 This SOP has been substantially revised to meet the requirements of method 8270C.

17.2.2 Directions for analysis be method 625 have been added as an attachment.

17.3 Facility Specific SOPs

Each facility shall attach a list of facility specific SOPs or approved attachments (if

applicable) which are required to implement this SOP or which are used in conjunction with this SOP. If no facility specific SOPs or amendments are to be attached, a statement must be attached specifying that there are none.

17.4

Tables

Table 1

Quanterra Primary Standard[1] and Standard Reporting Limits

Analytes	CAS Number	Standard Reporting Limits	
		Aqueous µg/L	Low Soil/Sediment µg/kg
Pyridine	110-86-1	20	660
N-nitrosodimethylamine	62-75-9	10	330
Aniline	62-53-3	10	330
Phenol	108-95-2	10	330
Bis(2-chloroethyl)ether	111-44-4	10	330
2-Chlorophenol	95-57-8	10	330
1,3-Dichlorobenzene	541-73-1	10	330
1,4-Dichlorobenzene	106-46-7	10	330
Benzyl alcohol	100-51-6	10	330
1,2-Dichlorobenzene	95-50-1	10	330
2-Methylphenol	95-48-7	10	330
2,2'-oxybis(1-chloropropane)[2]	108-60-1	10	330
4-Methylphenol	106-44-5	10	330
N-Nitroso-di-n-propylamine	621-64-7	10	330
Hexachloroethane	67-72-1	10	330
Nitrobenzene	98-95-3	10	330
Isophorone	78-59-1	10	330
2-Nitrophenol	88-75-5	10	330
2,4-Dimethylphenol	105-67-9	10	330
Benzoic acid	65-85-0	50	1600
Bis(2-chloroethoxy)methane	111-91-1	10	330
2,4-Dichlorophenol	120-83-2	10	330
1,2,4-Trichlorobenzene	120-82-1	10	330
Naphthalene	91-20-3	10	330
4-Chloroaniline	106-47-8	10	330
Hexachlorobutadiene	87-68-3	10	330
4-Chloro-3-methylphenol	59-50-7	10	330
2-Methylnaphthalene	91-57-6	10	330
Hexachlorocyclopentadiene	77-47-4	50	1600
2,4,6-Trichlorophenol	88-06-2	10	330
2,4,5-Trichlorophenol	95-95-4	10	330
2-Chloronaphthalene	91-58-7	10	330
2-Nitroaniline	88-74-4	50	1600
Dimethyl phthalate	131-11-3	10	330
Acenaphthylene	208-96-8	10	330
3-Nitroaniline	99-09-2	50	1600
Acenaphthene	83-32-9	10	330
2,4-Dinitrophenol	51-28-5	50	1600
4-Nitrophenol	100-02-7	50	1600
Dibenzofuran	132-64-9	10	330
2,4-Dinitrotoluene	121-14-2	10	330
2,6-Dinitrotoluene	606-20-2	10	330
Diethylphthalate	84-66-2	10	330

Table 1

Quanterra Primary Standard[1] and Standard Reporting Limits

Analytes	CAS Number	Standard Reporting Limits	
		Aqueous µg/L	Low Soil/Sediment µg/kg
4-Chlorophenyl phenyl ether	7005-72-3	10	330
Fluorene	86-73-7	10	330
4-Nitroaniline	100-01-6	50	1600
4,6-Dinitro-2-methylphenol	534-52-1	50	1600
N-Nitrosodiphenylamine	86-30-6	10	330
Azobenzene	103-33-3	10	330
4-Bromophenyl phenyl ether	101-55-3	10	330
Hexachlorobenzene	118-74-1	10	330
Pentachlorophenol	87-86-5	50	1600
Phenanthrene	85-01-8	10	330
Anthracene	120-12-7	10	330
Carbazole	86-74-8	10	330
Di-n-butyl phthalate	84-74-2	10	330
Fluoranthene	206-44-0	10	330
Benzidine	92-87-5	100	3300
Pyrene	129-00-0	10	330
Butyl benzyl phthalate	85-68-7	10	330
3,3'-Dichlorobenzidine	91-94-1	50	1600
Benzo(a)anthracene	56-55-3	10	330
Bis(2-ethylhexyl)phthalate	117-81-7	10	330
Chrysene	218-01-9	10	330
Di-n-octylphthalate	117-84-0	10	330
Benzo(b)fluoranthene	205-99-2	10	330
Benzo(k)fluoranthene	207-08-9	10	330
Benzo(a)pyrene	50-32-8	10	330
Indeno(1,2,3-cd)pyrene	193-39-5	10	330
Dibenz(a,h)anthracene	53-70-3	10	330
Benzo(g,h,i)perylene	191-24-2	10	330

[1] The Quanterra primary standard is the standard normally used at Quanterra. Additional standards, such as the Appendix IX standard may be necessary to include all target analytes required for some clients.

[2] 2,2'oxybis(1-chloropropane) was formally known as bis(2-chloroisopropyl)ether

Table 2

Quanterra Appendix IX[1] Standard Reporting Limits

Semivolatiles	CAS Number	Standard Reporting Limits	
		Aqueous µg/L	Low Soil/Sediment µg/kg
2-Picoline	109-06-8	20	660
N-Nitrosomethylethylamine	10595-95-6	10	330
Methyl methanesulfonate	66-27-3	10	330
N-Nitrosodiethylamine	55-18-5	10	330
Ethyl methanesulfonate	62-50-0	10	330
Pentachloroethane	76-01-7	50	1600
Acetophenone	98-86-2	10	330
N-Nitrosopyrrolidine	930-55-2	10	330
N-Nitrosomorpholine	59-89-2	10	330
o-Toluidine	95-53-4	20	660
3-Methylphenol	108-39-4	10	330
N-Nitrosopiperidine	100-75-4	10	330
o,o,o-Triethyl-Phosphorothioate[2]	126-68-1	50	1600
a,a-Dimethyl-phenethylamine	122-09-8	50	1600
2,6-Dichlorophenol	87-65-0	10	330
Hexachloropropene	1888-71-7	100	3300
p-Phenylenediamine	106-50-3	100	3300
n-Nitrosodi-n-butylamine	924-16-3	10	330
Safrole	94-59-7	20	660
1,2,4,5-Tetrachlorobenzene	95-94-3	10	330
Isosafrole	120-58-1	20	660
1,4-Dinitrobenzene	100-25-4	10	330
1,4-Naphthoquinone	130-15-4	50	1600
1,3-Dinitrobenzene	99-65-0	10	330
Pentachlorobenzene	608-93-5	10	330
1-Naphthylamine	134-32-7	10	330
2-Naphthylamine	91-59-8	10	330
2,3,4,6-Tetrachlorophenol	58-90-2	50	1600
5-Nitro-o-toluidine	99-55-8	20	660
Thionazin[2]	297-97-2	50	1600
1,3,5-Trinitrobenzene	99-35-4	50	1600
Sulfotepp[2]	3689-24-5	50	1600
Phorate[2]	298-02-2	50	1600
Phenacetin	62-44-2	20	660
Diallate[3]	2303-16-4	20	660
Dimethoate[2]	60-51-5	20	660
4-Aminobiphenyl	92-67-1	50	1600
Pentachloronitrobenzene	82-68-8	50	1600
Pronamide	23950-58-5	20	660
Disulfoton[2]	298-04-4	50	1600
2-secbutyl-4,6-dinitrophenol (Dinoseb)	88-85-7	20	660
Methyl Parathion[2]	298-00-0	50	1600
4-Nitroquinoline-1-oxide	56-57-5	100	3300
Parathion[2]	56-38-2	50	1600

Table 2

Quanterra Appendix IX[1] Standard Reporting Limits

Semivolatiles	CAS Number	Standard Reporting Limits	
		Aqueous µg/L	Low Soil/Sediment µg/kg
Methapyrilene	91-80-5	50	1600
Aramite	140-57-8	20	660
Isodrin[3]	465-73-6	10	330
Kepone[2]	143-50-0	100	3300
Famphur[3]	52-85-7	100	3300
p-(Dimethylamino)azobenzene	60-11-7	20	660
p-Chlorobenzilate[3]	510-15-6	10	330
3,3'-Dimethylbenzidine	119-93-7	50	1600
2-Acetylaminofluorene	53-96-3	100	3300
Dibenz(a,j)acridine	224-42-0	20	660
7,12-Dimethylbenz(a)anthracene	57-97-6	20	660
3-Methylcholanthrene	56-49-5	20	660

1 The Appendix IX standard contains additional analytes required for the Appendix IX list. The Quanterra primary standard must also be analyzed to include all of the Appendix IX list.

2 May also be analyzed by method 8140 or 8141, which can achieve lower reporting limits.

3 May also be analyzed by method 8080 or 8081, which can achieve lower reporting limits

Table 3

Reportable Analytes for Quanterra Standard Tests, Primary Standard

Analyte	CAS Number	Quanterra Standard List	TCLP	TCL	Appendix IX
Pyridine	110-86-1		X		X
N-nitrosodimethylamine	62-75-9				X
Aniline	62-53-3				X
Phenol	108-95-2	X		X	X
Bis(2-chloroethyl)ether	111-44-4	X		X	X
2-Chlorophenol	95-57-8	X		X	X
1,3-Dichlorobenzene	541-73-1	X		X	X
1,4-Dichlorobenzene	106-46-7	X	X	X	X
Benzyl alcohol	100-51-6				X
1,2-Dichlorobenzene	95-50-1	X		X	X
2-Methylphenol	95-48-7	X	X	X	X
2,2'-oxybis(1-chloropropane)[1]	180-60-1	X		X	X
4-Methylphenol	106-44-5	X	X	X	X
N-Nitroso-di-n-propylamine	621-64-7	X		X	X
Hexachloroethane	67-72-1	X	X	X	X
Nitrobenzene	98-95-3	X	X	X	X
Isophorone	78-59-1	X		X	X
2-Nitrophenol	88-75-5	X		X	X
2,4-Dimethylphenol	105-67-9	X		X	X
Benzoic acid	65-85-0				
Bis(2-chloroethoxy)methane	111-91-1	X		X	X
2,4-Dichlorophenol	120-83-2	X		X	X
1,2,4-Trichlorobenzene	120-82-1	X		X	X
Naphthalene	91-20-3	X		X	X
4-Chloroaniline	106-47-8	X		X	X
Hexachlorobutadiene	87-68-3	X	X	X	X
4-Chloro-3-methylphenol	59-50-7	X		X	X
2-Methylnaphthalene	91-57-6	X		X	X
Hexachlorocyclopentadiene	77-47-4	X		X	X
2,4,6-Trichlorophenol	88-06-2	X	X	X	X
2,4,5-Trichlorophenol	95-95-4	X	X	X	X
2-Chloronaphthalene	91-58-7	X		X	X
2-Nitroaniline	88-74-4	X		X	X
Dimethyl phthalate	131-11-3	X		X	X
Acenaphthylene	208-96-8	X		X	X
3-Nitroaniline	99-09-2	X		X	X
Acenaphthene	83-32-9	X		X	X
2,4-Dinitrophenol	51-28-5	X		X	X
4-Nitrophenol	100-02-7	X		X	X
Dibenzofuran	132-64-9	X		X	X
2,4-Dinitrotoluene	121-14-2	X	X	X	X
2,6-Dinitrotoluene	606-20-2	X		X	X
Diethylphthalate	84-66-2	X		X	X
4-Chlorophenyl phenyl ether	7005-72-3	X		X	X
Fluorene	86-73-7	X		X	X

Table 3

Reportable Analytes for Quanterra Standard Tests, Primary Standard

Analyte	CAS Number	Quanterra *Standard List*	TCLP	TCL	Appendix IX
4-Nitroaniline	100-01-6	X		X	X
4,6-Dinitro-2-methylphenol	534-52-1	X		X	X
N-Nitrosodiphenylamine	86-30-6	X		X	X
Azobenzene[4]	103-33-3				
4-Bromophenyl phenyl ether	101-55-3	X		X	X
Hexachlorobenzene	118-74-1	X	X	X	X
Pentachlorophenol	87-86-5	X	X	X	X
Phenanthrene	85-01-8	X		X	X
Anthracene	120-12-7	X		X	X
Carbazole	86-74-8	X		X	
Di-n-butyl phthalate	84-74-2	X		X	X
Fluoranthene	206-44-0	X		X	X
Benzidine	92-87-5				
Pyrene	129-00-0	X		X	X
Butyl benzyl phthalate	85-68-7	X		X	X
3,3'-Dichlorobenzidine	91-94-1	X		X	X
Benzo(a)anthracene	56-55-3	X		X	X
Bis(2-ethylhexyl)phthalate	117-81-7	X		X	X
Chrysene	218-01-9	X		X	X
Di-n-octylphthalate	117-84-0	X		X	X
Benzo(b)fluoranthene	205-99-2	X		X	X
Benzo(k)fluoranthene	207-08-9	X		X	X
Benzo(a)pyrene	50-32-8	X		X	X
Indeno(1,2,3-cd)pyrene	193-39-5	X		X	X
Dibenz(a,h)anthracene	53-70-3	X		X	X
Benzo(g,h,i)perylene	191-24-2	X		X	X

[1] 2,2'oxybis(1-chloropropane) was formally known as bis(2-chloroisopropyl)ether

[2] Azobenzene is formed by decomposition of 1,2-diphenlyhydrazine. If 1,2-diphenylhydrazine is requested, it will be analyzed as azobenzene.

Table 4

Reportable analytes for Quanterra Standard Tests, Appendix IX Standard

Semivolatiles	CAS Number	Quanterra Standard List	TCLP	TCL	Appendix IX
2-Picoline	109-06-8				X
N-Nitrosomethylethylamine	10595-95-6				X
Methyl methanesulfonate	66-27-3				X
N-Nitrosodiethylamine	55-18-5				X
Ethyl methanesulfonate	62-50-0				X
Pentachloroethane	76-01-7				X
Acetophenone	98-86-2				X
N-Nitrosopyrrolidine	930-55-2				X
N-Nitrosomorpholine	59-89-2				X
o-Toluidine	95-53-4				X
3-Methylphenol	108-39-4				X
N-Nitrosopiperidine	100-75-4				X
o,o,o-Triethyl-Phosphorothioate[2]	126-68-1				X
a,a-Dimethyl-phenethylamine	122-09-8				X
2,6-Dichlorophenol	87-65-0				X
Hexachloropropene	1888-71-7				X
p-Phenylenediamine	106-50-3				X
n-Nitrosodi-n-butylamine	924-16-3				X
Safrole	94-59-7				X
1,2,4,5-Tetrachlorobenzene	95-94-3				X
Isosafrole	120-58-1				X
1,4-Dinitrobenzene	100-25-4				
1,4-Naphthoquinone	130-15-4				X
1,3-Dinitrobenzene	99-65-0				X
Pentachlorobenzene	608-93-5				X
1-Naphthylamine	134-32-7				X
2-Naphthylamine	91-59-8				X
2,3,4,6-Tetrachlorophenol	58-90-2				X
5-Nitro-o-toluidine	99-55-8				X
Thionazin[2]	297-97-2				X
1,3,5-Trinitrobenzene	99-35-4				X
Sulfotepp[2]	3689-24-5				X
Phorate[2]	298-02-2				X
Phenacetin	62-44-2				X
Diallate	2303-16-4				X
Dimethoate[2]	60-51-5				X
4-Aminobiphenyl	92-67-1				X
Pentachloronitrobenzene	82-68-8				X
Pronamide	23950-58-5				X
Disulfoton[2]	298-04-4				X
2-secbutyl-4,6-dinitrophenol (Dinoseb)[2]	88-85-7				X
Methyl parathion[2]	298-00-0				X
4-Nitroquinoline-1-oxide	56-57-5				X
Parathion[2]	56-38-2				X

Table 4

Reportable analytes for Quanterra Standard Tests, Appendix IX Standard

Semivolatiles	CAS Number	Quanterra Standard List	TCLP	TCL	Appendix IX
Isodrin[3]	465-73-6				X
Kepone [2]	143-50-0				X
Famphur[2]	52-85-7				X
Methapyrilene	91-80-5				X
Aramite	140-57-8				X
p-(Dimethylamino)azobenzene	60-11-7				X
p-Chlorobenzilate[3]	510-15-6				X
3,3'-Dimethylbenzidine	119-93-7				X
2-Acetylaminofluorene	53-96-3				X
Dibenz(a,j)acridine	224-42-0				
7,12-Dimethylbenz(a)anthracene	57-97-6				X
3-Methylcholanthrene	56-49-5				X
Hexachlorophene[4]	70-30-4				X
Diphenylamine[5]	122-39-4				X

[2] May also be analyzed by method 8140 or 8141, which can achieve lower reporting limits.

[3] May also be analyzed by method 8080 or 8081, which can achieve lower reporting limits

[4] Hexachlorophene is a required analyte for Appendix IX. This compound is not stable, and therefore not included in the calibration standard. The characteristic ions for hexachlorophene are searched for in the chromatogram. (See section 12.2.1)

[5] Diphenylamine is a required compound for Appendix IX. N-nitrosodiphenylamine decomposes in the injection port to form diphenylamine. Therefore these two compounds cannot be distinguished. Diphenylamine is not included in the calibration standard.

Table 5

Suggested Instrumental Conditions

Mass Range	35-500 amu
Scan Time	≤1 second/scan
Initial Column Temperature/Hold Time	40°C for 2 minutes
Column Temperature Program	40 - 320°C at 11.5°C/min
Final Column Temperature/Hold Time	320°C (until at least one minute after benzo(g,h,i)perylene has eluted)
Injector Temperature	250 - 300°C
Transfer Line Temperature	250 - 300°C
Source Temperature	According to manufacturer's specifications
Injector	Grob-type, split / splitless
Sample Volume	1 or 2 µl
Carrier Gas	Helium at 30 cm/sec

Table 6

DFTPP Key Ions and Ion Abundance Criteria

Mass	Ion Abundance Criteria
51	30 - 60% of mass 198
68	<2% of mass 69
70	<2% of mass 69
127	40 - 60% of mass 198
197	<1% of mass 198
198	Base peak, 100% relative abundance
199	5 - 9% of mass 198
275	10 - 30% of mass 198
365	>1% of mass 198
441	Present, but less than mass 443
442	>40% of mass 198
443	17 - 23% of mass 442

Table 7

Analytes in Approximate Retention Time Order and Characteristic Ions, Primary Standard

Analyte	Primary	Secondary	Tertiary
N-nitrosodimethylamine	74	42	
Pyridine	79	52	
2-Fluorophenol (Surrogate Standard)	112	64	63
Phenol-d5 (Surrogate Standard)	99	42	71
Aniline	93	66	
Phenol	94	65	66
Bis(2-chloroethyl)ether	93	63	95
2-Chlorophenol	128	64	130
1,3-Dichlorobenzene	146	148	113
1,4-Dichlorobenzene-d4 (Internal Standard)	152	150	115
1,4-Dichlorobenzene	146	148	113
Benzyl Alcohol	108	79	77
1,2-Dichlorobenzene	146	148	113
2-Methylphenol	108	107	79
2,2'-oxybis(1-chloropropane)[1]	45	77	79
4-Methylphenol	108	107	79
N-Nitroso-di-n-propylamine	70	42	101,130
Hexachloroethane	117	201	199
Nitrobenzene-d5 (Surrogate Standard)	82	128	54
Nitrobenzene	77	123	65
Isophorone	82	95	138
2-Nitrophenol	139	65	109
2,4-Dimethylphenol	107	121	122
Benzoic Acid	122	105	77
Bis(2-chloroethoxy)methane	93	95	123
2,4-Dichlorophenol	162	164	98
1,2,4-Trichlorobenzene	180	182	145
Naphthalene-d8 (Internal Standard)	136	68	54
Naphthalene	128	129	127
4-Chloroaniline	127	129	65
Hexachlorobutadiene	225	223	227
4-Chloro-3-methylphenol	107	144	142
2-Methylnaphthalene	142	141	115
Hexachlorocyclopentadiene	237	235	272
2,4,6-Trichlorophenol	196	198	200
2,4,5-Trichlorophenol	196	198	200
2-Fluorobiphenyl (Surrogate Standard)	172	171	170
2-Chloronaphthalene	162	164	127
2-Nitroaniline	65	92	138
Dimethylphthalate	163	194	164
Acenaphthylene	152	151	153
2,6-Dinitrotoluene	165	63	89

Table 7

Analytes in Approximate Retention Time Order and Characteristic Ions, Primary Standard

Analyte	Primary	Secondary	Tertiary
Acenaphthene-d10 (Internal Standard)	164	162	160
3-Nitroaniline	138	108	92
Acenaphthene	153	152	154
2,4-Dinitrophenol	184	63	154
Dibenzofuran	168	139	84
4-Nitrophenol	109	139	65
2,4-Dinitrotoluene	165	63	89
Diethylphthalate	149	177	150
Fluorene	166	165	167
4-Chlorophenylphenylether	204	206	141
4-Nitroaniline	138	92	108
4,6-Dinitro-2-methylphenol	198	182	77
N-Nitrosodiphenylamine	169	168	167
2,4,6-Tribromophenol (Surrogate Standard)	330	332	141
Azobenzene	77	182	105
4-Bromophenylphenylether	248	250	141
Hexachlorobenzene	284	142	249
Pentachlorophenol	266	264	268
Phenanthrene-d10 (Internal Standard)	188	94	80
Phenanthrene	178	179	176
Anthracene	178	179	176
Carbazole	167	166	139
Di-n-butylphthalate	149	150	104
Fluoranthene	202	101	100
Benzidine	184	92	185
Pyrene	202	101	100
Terphenyl-d14 (Surrogate Standard)	244	122	212
Butylbenzylphthalate	149	91	206
Benzo(a)Anthracene	228	229	226
Chrysene-d12 (Internal Standard)	240	120	236
3,3'-Dichlorobenzidine	252	254	126
Chrysene	228	226	229
Bis(2-ethylhexyl)phthalate	149	167	279
Di-n-octylphthalate	149	167	43
Benzo(b)fluoranthene	252	253	125
Benzo(k)fluoranthene	252	253	125
Benzo(a)pyrene	252	253	125
Perylene-d12 (Internal Standard)	264	260	265
Indeno(1,2,3-cd)pyrene	276	138	277
Dibenz(a,h)anthracene	278	139	279
Benzo(g,h,i)perylene	276	138	277

Table 8

Analytes in Approximate Retention Time Order and Characteristic Ions, Appendix IX Standard

Analyte	Primary	Secondary	Tertiary
2-Picoline	93	66	92
N-Nitrosomethylethylamine	88	42	43
Methyl methanesulfonate	80	79	65
N-Nitrosodiethylamine	102	44	57
Ethyl methanesulfonate	79	109	97
Pentachloroethane	117	119	167
Acetophenone	105	77	120
N-Nitrosopyrrolidine	100	41	42
N-Nitrosomorpholine	116	56	86
o-Toluidine	106	107	
3-Methylphenol	108	107	77
N-Nitrosopiperidine	114	42	55
o,o,o-Triethyl-Phosphorothioate	198	121	93
a,a-Dimethyl-phenethylamine	58	91	
2,6-Dichlorophenol	162	164	63
Hexachloropropene	213	215	211
p-Phenylenediamine	108	80	
n-Nitrosodi-n-butylamine	84	57	41
Safrole	162	104	77
1,2,4,5-Tetrachlorobenzene	216	214	218
Isosafrole 1	162	104	131
Isosafrole 2	162	104	131
1,4-Dinitrobenzene	168	75	122
1,4-Naphthoquinone	158	104	102
1,3-Dinitrobenzene	168	75	76
Pentachlorobenzene	250	248	252
1-Naphthylamine	143	115	
2-Naphthylamine	143	115	
2,3,4,6-Tetrachlorophenol	232	230	131
5-Nitro-o-toluidine	152	77	106
Thionazin	97	96	143
1,3,5-Trinitrobenzene	213	75	120
Sulfotepp	97	322	202
Phorate	75	97	121
Phenacetin	108	179	109
Diallate	86	234	
Dimethoate	87	93	125
4-Aminobiphenyl	169		
Pentachloronitrobenzene	237	142	214
Pronamide	173	175	255
Disulfoton	88	97	89
2-secbutyl-4,6-dinitrophenol (Dinoseb)	211	163	147
Methyl parathion	109	125	263
4-Nitroquinoline-1-oxide	190	128	160
Parathion	109	97	291
Isodrin	193	66	195

Table 8

Analytes in Approximate Retention Time Order and Characteristic Ions, Appendix IX Standard

Analyte	Primary	Secondary	Tertiary
Kepone	272	274	237
Famphur	218	125	93
Methapyrilene	97	58	
Aramite 1	185	319	
Aramite 2	185	319	
p-(Dimethylamino)azobenzene	120	225	77
p-Chlorobenzilate	251	139	253
3,3'-Dimethylbenzidine	212	106	
2-Acetylaminofluorene	181	180	223
Dibenz(a,j)acridine	279	280	
7,12-Dimethylbenz(a)anthracene	256	241	120
3-Methylcholanthrene	268	252	253

Table 9

8270C LCS Compounds

LCS Compounds	Spiking Level, ng/µL in extract[1]
1,2,4-Trichlorobenzene	100
Acenaphthene	100
2,4-Dinitrotoluene	100
Pyrene	100
N-Nitroso-di-n-propylamine	100
1,4-Dichlorobenzene	100
Pentachlorophenol	150
Phenol	150
2-Chlorophenol	150
4-Chloro-3-methylphenol	150
4-Nitrophenol	150

[1] Levels are 50 and 75 ng/µL if 2 µL injection is used

Table 10

TCLP LCS Compounds

LCS Compounds	Spiking Level, ng/µL in extract[1]
1,4-Dichlorobenzene	100
2,4-Dinitrotoluene	100
Hexachlorobenzene	100
Hexachlorobutadiene	100
Hexachloroethane	100
2-Methylphenol	100
3-Methylphenol	100
4-Methylphenol	100
Nitrobenzene	100
Pentachlorophenol	100
Pyridine	100
2,4,5-Trichlorophenol	100
2,4,6-Trichlorophenol	100

[1] Levels are 50 ng/µL if 2 µL injection is used

Recovery limits for the LCS and for matrix spikes are generated from historical data and are maintained by the QA department.

Table 11

8270C Surrogate Compounds

Surrogate Compounds	Spiking Level, ng/µL in extract[2]
Nitrobenzene-d5	100
2-Fluorobiphenyl	100
Terphenyl-d14	100
1,2-Dichlorobenzene-d4[1]	100
Phenol-d5	150
2-Fluorophenol	150
2,4,6-Tribromophenol	150
2-Chlorophenol-d4[1]	150

[1] Included in standard mix, but not routinely evaluated for method 8270B

[2] Levels are 50 and 75 ng/µL if 2 µL injection is used

Recovery limits for surrogates are generated from historical data and are maintained by the QA department.

Table 12

Calibration Levels, Primary Standard, µg/mL

Analyte	Level 1	Level 2	Level 3	Level 4	Level 5
Pyridine	10	20	50	100	160
N-nitrosodimethylamine	10	20	50	100	160
Aniline	10	20	50	100	160
Phenol	10	20	50	100	160
Bis(2-chloroethyl)ether	10	20	50	100	160
2-Chlorophenol	10	20	50	100	160
1,3-Dichlorobenzene	10	20	50	100	160
1,4-Dichlorobenzene	10	20	50	100	160
Benzyl alcohol	10	20	50	100	160
1,2-Dichlorobenzene	10	20	50	100	160
2-Methylphenol	10	20	50	100	160
2,2'-oxybis(1-chloropropane)[1]	10	20	50	100	160
4-Methylphenol	10	20	50	100	160
N-Nitroso-di-n-propylamine	10	20	50	100	160
Hexachloroethane	10	20	50	100	160
Nitrobenzene	10	20	50	100	160
Isophorone	10	20	50	100	160
2-Nitrophenol	10	20	50	100	160
2,4-Dimethylphenol	10	20	50	100	160
Benzoic acid	20	50	100	200	320
Bis(2-chloroethoxy)methane	10	20	50	100	160
2,4-Dichlorophenol	10	20	50	100	160
1,2,4-Trichlorobenzene	10	20	50	100	160
Naphthalene	10	20	50	100	160
4-Chloroaniline	10	20	50	100	160
Hexachlorobutadiene	10	20	50	100	160
4-Chloro-3-methylphenol	10	20	50	100	160
2-Methylnaphthalene	10	20	50	100	160
Hexachlorocyclopentadiene	10	20	50	100	160
2,4,6-Trichlorophenol	10	20	50	100	160
2,4,5-Trichlorophenol	10	20	50	100	160
2-Chloronaphthalene	10	20	50	100	160
2-Nitroaniline	20	50	100	200	320
Dimethyl phthalate	10	20	50	100	160
Acenaphthylene	10	20	50	100	160
3-Nitroaniline	20	50	100	200	320
Acenaphthene	10	20	50	100	160
2,4-Dinitrophenol	20	50	100	200	320
4-Nitrophenol	20	50	100	200	320
Dibenzofuran	10	20	50	100	160
2,4-Dinitrotoluene	10	20	50	100	160
2,6-Dinitrotoluene	10	20	50	100	160
Diethylphthalate	10	20	50	100	160
4-Chlorophenyl phenyl ether	10	20	50	100	160
Fluorene	10	20	50	100	160
4-Nitroaniline	10	20	50	100	160

Table 12

Calibration Levels, Primary Standard, µg/mL

Analyte	Level 1	Level 2	Level 3	Level 4	Level 5
4,6-Dinitro-2-methylphenol	20	50	100	200	320
N-Nitrosodiphenylamine	10	20	50	100	160
Azobenzene[2]	10	20	50	100	160
4-Bromophenyl phenyl ether	10	20	50	100	160
Hexachlorobenzene	10	20	50	100	160
Pentachlorophenol	20	50	100	200	320
Phenanthrene	10	20	50	100	160
Anthracene	10	20	50	100	160
Carbazole	10	20	50	100	160
Di-n-butyl phthalate	10	20	50	100	160
Fluoranthene	10	20	50	100	160
Benzidine	20	50	100	200	320
Pyrene	10	20	50	100	160
Butyl benzyl phthalate	10	20	50	100	160
3,3'-Dichlorobenzidine	20	50	100	200	320
Benzo(a)anthracene	10	20	50	100	160
Bis(2-ethylhexyl)phthalate	10	20	50	100	160
Chrysene	10	20	50	100	160
Di-n-octylphthalate	10	20	50	100	160
Benzo(b)fluoranthene	10	20	50	100	160
Benzo(k)fluoranthene	10	20	50	100	160
Benzo(a)pyrene	10	20	50	100	160
Indeno(1,2,3-cd)pyrene	10	20	50	100	160
Dibenz(a,h)anthracene	10	20	50	100	160
Benzo(g,h,i)perylene	10	20	50	100	160

[1] 2,2'oxybis(1-chloropropane) was formally known as bis(2-chloroisopropyl)ether

[2]Azobenzene is formed by decomposition of 1,2-diphenlyhydrazine. If 1,2-diphenylhydrazine is requested, it will be analyzed as azobenzene.

Table 13

Calibration Levels, Appendix IX Standard, µg/mL

Semivolatiles	Level 1	Level 2	Level 3	Level 4	Level 5
2-Picoline	10	20	50	100	160
N-Nitrosomethylethylamine	10	20	50	100	160
Methyl methanesulfonate	10	20	50	100	160
N-Nitrosodiethylamine	10	20	50	100	160
Ethyl methanesulfonate	10	20	50	100	160
Pentachloroethane	10	20	50	100	160
Acetophenone	10	20	50	100	160
N-Nitrosopyrrolidine	10	20	50	100	160
N-Nitrosomorpholine	10	20	50	100	160
o-Toluidine	10	20	50	100	160
3-Methylphenol	10	20	50	100	160
N-Nitrosopiperidine	10	20	50	100	160
o,o,o-Triethyl-Phosphorothioate	20	50	100	200	320
a,a-Dimethyl-phenethylamine	10	20	50	100	160
2,6-Dichlorophenol	10	20	50	100	160
Hexachloropropene	20	50	100	200	320
p-Phenylenediamine	10	20	50	100	160
n-Nitrosodi-n-butylamine	10	20	50	100	160
Safrole	10	20	50	100	160
1,2,4,5-Tetrachlorobenzene	10	20	50	100	160
Isosafrole 1 + 2	20	50	100	200	320
1,4-Dinitrobenzene	10	20	50	100	160
1,4-Naphthoquinone	10	20	50	100	160
1,3-Dinitrobenzene	10	20	50	100	160
Pentachlorobenzene	10	20	50	100	160
1-Naphthylamine	10	20	50	100	160
2-Naphthylamine	10	20	50	100	160
2,3,4,6-Tetrachlorophenol	10	20	50	100	160
5-Nitro-o-toluidine	10	20	50	100	160
Thionazin	10	20	50	100	160
1,3,5-Trinitrobenzene	20	50	100	200	320
Sulfotepp	10	20	50	100	160
Phorate	10	20	50	100	160
Phenacetin	10	20	50	100	160
Diallate 1 + 2	20	50	100	200	320
Dimethoate	10	20	50	100	160
4-Aminobiphenyl	10	20	50	100	160
Pentachloronitrobenzene	20	50	100	200	320
Pronamide	10	20	50	100	160
Disulfoton	10	20	50	100	160
2-secbutyl-4,6-dinitrophenol (Dinoseb)	20	50	100	200	320
Methyl parathion	10	20	50	100	160
4-Nitroquinoline-1-oxide	20	40	100	200	320
Parathion	10	20	50	100	160
Isodrin	10	20	50	100	160
Kepone	20	50	100	200	320

Table 13

Calibration Levels, Appendix IX Standard, µg/mL

Semivolatiles	Level 1	Level 2	Level 3	Level 4	Level 5
Famphur	20	50	100	200	320
Methapyrilene	10	20	50	100	160
Aramite 1 and 2	20	50	100	200	320
p-(Dimethylamino)azobenzene	10	20	50	100	160
p-Chlorobenzilate	10	20	50	100	160
3,3'-Dimethylbenzidine	10	20	50	100	160
2-Acetylaminofluorene	10	20	50	100	160
Dibenz (a,j)acridine	10	20	50	100	160
7,12-Dimethylbenz(a)anthracene	10	20	50	100	160
3-Methylcholanthrene	10	20	50	100	160

Table 14
Initial demonstration recovery and precision limits

Compound	Spiking concentration µg/L	Limit for Relative Standard Deviation	Limit for average recovery, %
Acenaphthene	60	27.6	60.1-132.3
Acenaphthylene	60	40.2	53.5-126.0
Aldrin[1]	60	39.0	7.2-152.2
Anthracene	60	32.0	43.4-118.0
Benz(a)anthracene	60	27.6	41.8-133.0
Benzo(b)fluoranthene	60	38.8	42.0-140.4
Benzo(k)fluoranthene	60	32.3	25.2-145.7
Benzo(a)pyrene	60	39.0	31.7-148.0
Benzo(ghi)perylene	60	58.9	D-195.0
Benzylbutyl phthalate	60	23.4	D-139.9
B-BHC[1]	60	31.5	41.5-130.6
d-BHC[1]	60	21.6	D-100.0
Bis(2-chloroethyl) ether	60	55.0	42.9-126.0
Bis(2-chloroethoxy)methane	60	34.5	49.2-164.7
Bis(2-chloroisopropyl) ether	60	46.3	62.8-138.6
Bis(2-ethylhexyl) phthalate	60	41.1	28.9-136.8
4-Bromophenyl phenyl ether	60	23.0	64.9-114.4
2-Chloronaphthalene	60	13.0	64.5-113.5
4-Chlorophenyl phenyl ether	60	33.4	38.4-144.7
Chrysene	60	48.3	44.1-139.9
4,4'-DDD[1]	60	31.0	D-134.5
4,4'-DDE[1]	60	32.0	19.2-119.7
4,4'-DDT[1]	60	61.6	D-170.6
Dibenzo(a,h)anthracene	60	70.0	D-199.7
Di-n-butyl phthalate	60	16.7	8.4-111.0
1,2-Dichlorobenzene	60	30.9	48.6-112.0
1,3-Dichlorobenzene	60	41.7	16.7-153.9
1,4-Dichlorobenzene	60	32.1	37.3-105.7

Table 14
Initial demonstration recovery and precision limits

Compound	Spiking concentration µg/L	Limit for Relative Standard Deviation	Limit for average recovery, %
3,3'-Dichlorobenzidine	60	71.4	8.2-212.5
Dieldrin[1]	60	30.7	44.3-119.3
Diethyl phthalate	60	26.5	D-100.0
Dimethyl phthalate	60	23.2	D-100.0
2,4-Dinitrotoluene	60	21.8	47.5-126.9
2,6-Dinitrotoluene	60	29.6	68.1-136.7
Di-n-octylphthalate	60	31.4	18.6-131.8
Endosulfan sulfate[1]	60	16.7	D-103.5
Endrin aldehyde	60	32.5	D-188.8
Fluoranthene	60	32.8	42.9-121.3
Fluorene	60	20.7	71.6-108.4
Heptachlor[1]	60	37.2	D-172.2
Heptachlor epoxide[1]	60	54.7	70.9-109.4
Hexachlorobenzene	60	24.9	7.8-141.5
Hexachlorobutadiene	60	26.3	37.8-102.2
Hexachloroethane	60	24.5	55.2-100.0
Indeno(1,2,3-cd)pyrene	60	44.6	D-150.9
Isophorone	60	63.3	46.6-180.2
Naphthalene	60	30.1	35.6-119.6
Nitrobenzene	60	39.3	54.3-157.6
N-Nitrosodi-n-propylamine	60	55.4	13.6-197.9
PCB-1260[1]	60	54.2	19.3-121.0
Phenanthrene	60	20.6	65.2-108.7
Pyrene	60	25.2	69.6-100.0
1,2,4-Trichlorobenzene	60	28.1	57.3-129.2
4-Chloro-3-methylphenol	60	37.2	40.8-127.9
2-Chlorophenol	60	28.7	36.2-120.4
2,4-Chlorophenol	60	26.4	52.5-121.7
2,4-Dimethylphenol	60	26.1	41.8-109.0
2,4-Dinitrophenol	60	49.8	D-172.9
2-Methyl-4,6-dinitrophenol	60	93.2	53.0-100.0
2-Nitrophenol	60	35.2	45.0-166.7
4-Nitrophenol	60	47.2	13.0-106.5
Pentachlorophenol	60	48.9	38.1-151.8
Phenol	60	22.6	16.6-100.0
2,4,6-Trichlorophenol	60	31.7	52.4-129.2

[1]Since the organochlorine pesticides and PCBs are normally determined by method 8080 at Quanterra, they will not be included in the initial demonstration of capability for method 8270B.

ATTACHMENT A

MODIFICATIONS REQUIRED FOR ANALYSIS OF WASTEWATER FOLLOWING METHOD 625

18

REQUIREMENTS FOR METHOD 625

18.1 Method 625 is required for demonstration of compliance with NPDES wastewater discharge permits. The standard analyte list and reporting limits are listed in Table A-1.

18.2 This method can be applied only to aqueous matrices.

18.3 The tune period for this method is defined as 24 hours.

18.4 Initial calibration curve requirements:

18.4.1 The initial calibration curve for this method requires at least three points.

18.4.2 Target compounds must have RSD ≤ 35%.

18.4.3 If this requirement can not be met, a regression curve must be constructed for the non-compliant compounds.

18.5 Continuing calibration verification requirements: All target compounds must have %D ≤ 20%.

18.6 Matrix Spike and LCS requirements:

18.6.1 A full analyte spike is required for method 625. The spiking levels are given in Table A-2.

Table A-1. Quanterra Method 625 standard reporting list and reporting limits.

Analytes	CAS Number	Aqueous µg/L
Phenol	108-95-2	10
Bis(2-chloroethyl)ether	111-44-4	10
2-Chlorophenol	95-57-8	10
1,3-Dichlorobenzene	541-73-1	10
1,4-Dichlorobenzene	106-46-7	10
1,2-Dichlorobenzene	95-50-1	10
2,2'-oxybis(1-chloropropane)	108-60-1	10
N-Nitroso-di-n-propylamine	621-64-7	10
Hexachloroethane	67-72-1	10
Nitrobenzene	98-95-3	10
Isophorone	78-59-1	10
2-Nitrophenol	88-75-5	10
2,4-Dimethylphenol	105-67-9	10
Bis(2-chloroethoxy)methane	111-91-1	10
2,4-Dichlorophenol	120-83-2	10
1,2,4-Trichlorobenzene	120-82-1	10
Naphthalene	91-20-3	10
Hexachlorobutadiene	87-68-3	10
4-Chloro-3-methylphenol	59-50-7	10
Hexachlorocyclopentadiene	77-47-4	50
2,4,6-Trichlorophenol	88-06-2	10
2-Chloronaphthalene	91-58-7	10
Dimethyl phthalate	131-11-3	10
Acenaphthylene	208-96-8	10
Acenaphthene	83-32-9	10
2,4-Dinitrophenol	51-28-5	50
4-Nitrophenol	100-02-7	50
2,4-Dinitrotoluene	121-14-2	10
2,6-Dinitrotoluene	606-20-2	10
Diethylphthalate	84-66-2	10
4-Chlorophenyl phenyl ether	7005-72-3	10
Fluorene	86-73-7	10
4,6-Dinitro-2-methylphenol	534-52-1	50
N-Nitrosodiphenylamine	86-30-6	10
4-Bromophenyl phenyl ether	101-55-3	10
Hexachlorobenzene	118-74-1	10
Pentachlorophenol	87-86-5	50
Phenanthrene	85-01-8	10
Anthracene	120-12-7	10
Di-n-butyl phthalate	84-74-2	10
Fluoranthene	206-44-0	10
Benzidine	92-87-5	100
Pyrene	129-00-0	10
Butyl benzyl phthalate	85-68-7	10
3,3'-Dichlorobenzidine	91-94-1	50

Analytes	CAS Number	Aqueous µg/L
Benzo(a)anthracene	56-55-3	10
Bis(2-ethylhexyl)phthalate	117-81-7	10
Chrysene	218-01-9	10
Di-n-octylphthalate	117-84-0	10
Benzo(b)fluoranthene	205-99-2	10
Benzo(k)fluoranthene	207-08-9	10
Benzo(a)pyrene	50-32-8	10
Indeno(1,2,3-cd)pyrene	193-39-5	10
Dibenz(a,h)anthracene	53-70-3	10
Benzo(g,h,i)perylene	191-24-2	10

Table A-2. Method 625 LCS and MS compounds and spike concentrations.

LCS Compounds	Spiking Level, ng/µL in extract[1]
Phenol	100
Bis(2-chloroethyl)ether	100
2-Chlorophenol	100
1,3-Dichlorobenzene	100
1,4-Dichlorobenzene	100
1,2-Dichlorobenzene	100
2,2'-oxybis(1-chloropropane)	100
N-Nitroso-di-n-propylamine	100
Hexachloroethane	100
Nitrobenzene	100
Isophorone	100
2-Nitrophenol	100
2,4-Dimethylphenol	100
Bis(2-chloroethoxy)methane	100
2,4-Dichlorophenol	100
1,2,4-Trichlorobenzene	100
Naphthalene	100
Hexachlorobutadiene	100
4-Chloro-3-methylphenol	100
Hexachlorocyclopentadiene	100
2,4,6-Trichlorophenol	100
2-Chloronaphthalene	100
Dimethyl phthalate	100
Acenaphthylene	100
Acenaphthene	100
2,4-Dinitrophenol	100
4-Nitrophenol	100
2,4-Dinitrotoluene	100
2,6-Dinitrotoluene	100
Diethylphthalate	100
4-Chlorophenyl phenyl ether	100
Fluorene	100
4,6-Dinitro-2-methylphenol	100
N-Nitrosodiphenylamine	100
4-Bromophenyl phenyl ether	100
Hexachlorobenzene	100
Pentachlorophenol	100
Phenanthrene	100
Anthracene	100
Di-n-butyl phthalate	100
Fluoranthene	100
Benzidine	100
Pyrene	100
Butyl benzyl phthalate	100
3,3'-Dichlorobenzidine	100
Benzo(a)anthracene	100
Bis(2-ethylhexyl)phthalate	100

LCS Compounds	Spiking Level, ng/µL in extract[1]
Chrysene	100
Di-n-octylphthalate	100
Benzo(b)fluoranthene	100
Benzo(k)fluoranthene	100
Benzo(a)pyrene	100
Indeno(1,2,3-cd)pyrene	100
Dibenz(a,h)anthracene	100
Benzo(g,h,i)perylene	100

[1] **Levels are 50 and 75 ng/µL if 2 µL injection is used**

QUANTERRA STANDARD OPERATING PROCEDURE

TITLE: GAS CHROMATOGRAPHIC ANALYSIS BASED ON METHOD 8000B, 8021B, 8081A, 8082 and 8151A, SW-846

(SUPERSEDES: Revision 3)

Prepared by: Richard Burrows

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C4-497

TABLE OF CONTENTS

1.	Scope and Application	4
2.	Summary of Method	4
3.	Definitions	4
4.	Interferences	4
5.	Safety	4
6.	Equipment and Supplies	4
7.	Reagents and Standards	5
8.	Sample Preservation and Storage	5
9.	Quality Control	5
10.	Calibration and Standardization	8
11.	Procedure	14
12.	Data Analysis and Calculations	16
13.	Method Performance	19
14.	Pollution Prevention	20
15.	Waste Management	20
16.	References	20
17.	Miscellaneous	20

List of Appendices:

Appendix A Analysis of Volatile Organics based on Method 8021B

Appendix B Analysis of Organochlorine Pesticides based on Method 8081A

Appendix C Analysis of Organochlorine Pesticides and PCBs based on Method 8082

Appendix D Analysis of Phenoxy Acid Herbicides based on Method 8151A

List of Tables

Table A1	Standard analyte lists for 8021B
Table A2-A4	Recommended conditions for method 8021B
Table A5	Surrogate and Internal standard concentrations for aqueous and low level soil samples, method 8021B
Table A6	Concentrations for LCS and MS/MSD compounds, low level soil and aqueous, method 8021B.
Table B1	Standard analyte and reporting limits, method 8081A
Table B2	Recommended conditions, method 8081A
Table B3	Calibration levels, method 8081A
Table B4	Column degradation evaluation mix, method 8081A
Table B5	LCS/matrix spike and surrogate levels, method 8081A
Table B6	LCS/matrix spike and surrogate levels for TCLP, method 8081A
Table B7	Suggested analytical sequence, method 8081A
Table B8	Performance limits, method 8081A
Table C1	Standard analyte list, method 8082
Table C2	Instrumental conditions, method 8082
Table C3	Calibration standards, method 8082
Table C4	LCS/Matrix spike and surrogate levels, method 8082
Table C5	Suggested Analytical Sequence, Method 8082
Table D1	Standard Analyte List, method 8151A
Table D2	Instrumental conditions, method 8151A
Table D3	Calibration standards, method 8151A
Table D4	LCS/Matrix spike and surrogate levels, method 8151B
Table D5	Performance limits, method 8151B

1.

SCOPE AND APPLICATION

This SOP describes procedures for analysis of organic analytes by Gas Chromatography (GC). The procedures are based on SW-846 methodology and are applicable for measurements made to comply with the Resource Conservation and Recovery Act (RCRA). Individual analytes and methods are described in the appendices.

2. SUMMARY OF METHOD

In general, semivolatile analytes in aqueous samples are prepared for analysis using continuous or separatory funnel liquid / liquid extraction or solid phase extraction (SOP # CORP-OP-0001) Solid samples are prepared using sonication, Soxhlet or pressurized fluid extraction (SOP # CORP-OP-0001). Volatile analytes are prepared for analysis using purge and trap methodology (Appendix A).

After the initial preparation step, the sample is introduced to the GC and concentrations of target analytes are measured by the detector response within a defined retention time window, relative to the response to standard concentrations. Internal or external standardization procedures are used as specified in the method appendices.

3. DEFINITIONS

Definitions of terms used in this SOP may be found in the glossary of the Quality Assurance Management Plan (QAMP).

4. INTERFERENCES

Contamination by carryover can occur when a low concentration sample is analyzed after a high concentration sample. In addition, some purge and trap autosamplers are susceptible to port specific contamination. Co-elution of target analytes with non-targets can occur, resulting in false positives or biased high results. In particular, this is a problem with non-selective detectors such as the Flame Ionization Detector (FID). See the appendices for interferences specific to individual tests and suggested corrective actions.

5. SAFETY

5.1. Procedures shall be carried out in a manner that protects the health and safety of all Quanterra associates. The following requirements must be met:

Eye protection that satisfies ANSI Z87.1 (as per the Chemical Hygiene Plan), laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have become contaminated will be removed and discarded; other gloves will be cleaned immediately. Refer to the Quanterra Chemical Hygiene plan for a complete description of personal protection equipment.

The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the MSDS files maintained in the laboratory. Specific hazards are covered in the appendices.

5.1.1. Opened containers of neat standards will be handled in a fume hood.

6. EQUIPMENT AND SUPPLIES

An analytical system complete with a gas chromatograph is required. A data system capable of measuring peak area and/or height is required. Recommended equipment and supplies for individual methods are listed in each method appendix.

7. REAGENTS AND STANDARDS

7.1. Stock Standards

Stock standards are purchased as certified solutions or prepared from pure solutions. Stock standards for method 8021B are stored at -10 to -20°C. Other stock standard solutions are stored at ≤6°C. All stock standards must be protected from light. Stock standard solutions should be brought to room temperature before using.

Semivolatile stock standard solutions must be replaced after one year. Stock standards of gases must be replaced at t every week, unless the acceptability of the standard is demonstrated (Less than 20% drift from the initial calibration.). Other volatile stock standards must be replaced every 6 or sooner if comparison with check standards prepared from an independent source indicates a problem.

7.1.1. Expiration times for all standards are measured from the time the standard is prepared or from the time that the standard ampoule is opened, if the standard is supplied in a sealed ampoule. If a vendor supplied standard has an earlier expiration date then that date is used.

7.2. Calibration Standards

7.2.1. Volatile Calibration Standards

The procedure for preparation of volatile standards is given in Appendix A.

7.2.2. Semivolatile Calibration Standards

Semivolatile calibration standards are prepared as dilutions of the stock standards. Surrogates and internal standards are used as specified in the method appendices. Semivolatile calibration solutions must be refrigerated at ≤6°C and protected from light. The standards must be replaced at least every six months or sooner if comparison with check standards indicates a problem.

7.3. Gases for carrier and make-up: Hydrogen, Helium, Nitrogen, Argon/Methane.

7.4. Quality control (QC) Standards

QC standards (matrix spiking and LCS standards) are prepared and stored in the same way as calibration standards. They must be made from a stock independent from the calibration standards.

8. SAMPLE PRESERVATION AND STORAGE

Semivolatile extracts must be refrigerated at ≤6°C and analyzed within 40 days of the start of the extraction. Volatile sample storage conditions and holding times are given in Appendix A.

9. QUALITY CONTROL

9.1. Initial Demonstration of Capability

9.1.1. For the standard analyte list, the initial demonstration and method detection limit (MDL) studies described in section 13 must be acceptable before analysis of samples may begin.

9.1.2. For non-standard analytes, a MDL study must be performed and calibration curve generated before analyzing any samples, unless lesser requirements are previously agreed to with the client. In any event the minimum initial demonstration required is analysis of an extracted standard at the reporting limit and a single point calibration.

9.2. Batch Definition

Batches are defined at the sample preparation stage. Batches should be kept together through the whole analytical process as far as possible, but it is not mandatory to analyze prepared extracts on the same instrument or in the same sequence. Refer to the Quanterra QC Program document (QA-003) for further details of the batch definition.

9.2.1. Quality Control Batch

The batch is a set of up to 20 samples of the same matrix processed using the same procedures and reagents within the same time period. The Quality Control batch must contain a matrix spike / spike duplicate (MS/MSD), a Laboratory Control Sample (LCS), and a method blank. Laboratory generated QC samples (Blank, LCS, MS/MSD) do not count towards the maximum 20 samples in a batch. Field QC samples are included in the batch count. In some cases, at client request, the MS/MSD may be replaced with a matrix spike and sample duplicate. If insufficient sample is available for an MS/MSD a LCSD may be substituted.

9.3. Control Limits

In-house historical control limits must be determined for surrogates, matrix spikes, and laboratory control samples (LCS). These limits must be determined at least semi-annually. The recovery limits are mean recovery +/- 3 standard deviations, unless that limit is tighter than the calibration criteria, in which case limits may be widened. Refer to policy QA-003 for more details.

9.3.1. These limits do not apply to dilutions (except for tests without a separate extraction), but surrogate and matrix spike recoveries will be reported unless the dilution is more than 5X.

9.3.2. All surrogate, LCS, and MS recoveries (except for dilutions) must be entered into QuantIMS (when available) or other database so that accurate historical control limits can be generated. For tests without a separate extraction, surrogates and matrix spikes will be reported for all dilutions.

9.3.3. Refer to the QC Program document (QA-003) for further details of control limits.

9.4. Surrogates

All methods must use surrogates to the extent possible. Surrogate recoveries in samples and QC samples must be assessed to ensure that recoveries are within established limits. If any surrogates are outside limits, the following corrective actions must take place (except for dilutions):

- Check all calculations for error.
- Ensure that instrument performance is acceptable.
- Recalculate the data and/or reanalyze the extract if either of the above checks reveal a problem.
- Reprepare and reanalyze the sample or flag the data as "Estimated Concentration" if neither of the above resolves the problem. Repreparation is not necessary is there is obvious chromatographic interference.
- The decision to reanalyze or flag the data should be made in consultation with the client. It is only necessary to reprepare / reanalyze a sample once to demonstrate that poor surrogate recovery is due to matrix effect, unless the analyst believes that the repeated out of control results are not due to matrix effect.

9.4.1. If dual column analysis is used the choice of which result to report in made in the same way as for samples (Section 12.1.2) unless one column is out of control, in which case the in control result is reported.

9.4.2. If the surrogates are out of control for the sample, matrix spike, and matrix spike duplicate, then matrix effect has been demonstrated for that sample and repreparation is not necessary. If the sample is out of control and the MS and/or MSD is in control, then repreparation or flagging of the data is required.

9.4.3. Refer to the Quanterra QC Program document (QA-003) for further details of the corrective actions.

9.5. Method Blanks

For each batch of samples, analyze a method blank. The method blank consists of reagent water for aqueous semivolatiles samples, and sodium sulfate for semivolatiles soils tests (Refer to SOP No. CORP-OP-0001 for details). For low level volatiles, the method blank consists of reagent water. For medium level volatiles, the method blank consists of methanol as described in Appendix A. Surrogates are added and the method blank is carried through the entire analytical procedure. The method blank must not contain any analyte of interest at or above the reporting limit (except common laboratory contaminants, see below) or at or above 5% of the measured concentration of that analyte in the associated samples, whichever is higher.

If the analyte is a common laboratory contaminant (methylene chloride, acetone, 2-butanone, phthalate esters) the data may be reported with qualifiers if the concentration of the analyte is less than five times the reporting limit. Such action must be taken in consultation with the client.

Re-extraction and reanalysis of samples associated with an unacceptable method blank is required when reportable concentrations are determined in the samples.

If there is no target analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. Such action should be taken in consultation with the client.

9.5.1. Refer to the Quanterra QC Program document (QA-003) for further details of the corrective actions.

9.6. Instrument Blanks

9.6.1. An instrument blank must be analyzed during any 12 hour period of analysis that does not contain a method blank.

9.6.2. An instrument blank consists of the appropriate solvent with internal standards added. If internal standards are not used the surrogates should be added.

9.6.3. Control criteria are the same as for the method blank, except that only reanalysis of affected samples would be required, not re-extraction.

9.7. Laboratory Control Samples (LCS)

For each batch of samples, analyze a LCS. The LCS contains a representative subset of the analytes of interest, and must contain the same analytes as the matrix spike. The LCS may also contain the full set of analytes. If any analyte or surrogate is outside established control limits, the system is out of control and corrective action must occur. Corrective action will normally be repreparation and reanalysis of the batch; however, if the matrix spike and matrix spike duplicate are within limits, the batch may be acceptable.

9.7.1. Refer to the Quanterra QC Program document (QA-003) for further details of the corrective action.

9.7.2. If dual column analysis is used the choice of which result to report in made in the same way as for samples (Section 12.1.2) unless one column is out of control, in which case the in control result is reported.

9.7.3. LCS compound lists are included in the appendices.

9.7.4. If full analyte spike lists are used at client request, it will be necessary to allow a percentage of the components to be outside control limits as this would be expected statistically. These requirements should be negotiated with the client.

9.8. Matrix Spikes

For each QC batch, analyze a matrix spike and matrix spike duplicate. Spiking compounds and levels are given in the appendices. Compare the percent recovery and relative percent difference (RPD) to those in the laboratory specific historically generated limits.

- If any individual recovery or RPD falls outside the acceptable range, corrective action must occur. The initial corrective action will be to check the recovery of that analyte in the Laboratory Control Sample (LCS). Generally, if the recovery of the analyte in the LCS is within limits, then the laboratory operation is in control and analysis may proceed.
- If the recovery for any component is outside QC limits for both the Matrix spike / spike duplicate and the LCS, the laboratory is out of control and corrective action must be taken. Corrective action will normally include repreparation and reanalysis of the batch.
- If a MS/MSD is not possible due to limited sample, then a LCS duplicate should be analyzed.
- The matrix spike / duplicate must be analyzed at the same dilution as the unspiked sample, unless the matrix spike components would then be above the calibration range.

9.8.1. If dual column analysis is used the choice of which result to report in made in the same way as for samples (Section 12.1.2) unless one column is out of control, in which case the in control result is reported.

9.9. Quality Assurance Summaries

Certain clients may require specific project or program QC which may supersede these method requirements. Quality Assurance Summaries should be developed to address these requirements.

9.10. Quanterra QC Program

Further details of QC and corrective action guidelines are presented in the Quanterra QC Program document (QA-003). Refer to this document if in doubt regarding corrective actions.

10. CALIBRATION AND STANDARDIZATION

Internal or external calibration may be used. Internal calibration is recommended unless the sample matrix is likely to interfere with the quantitation of the internal standard. In either event prepare standards containing each analyte of interest at a minimum of five concentration levels. The low level standard should be at or below the reporting limit. The other standards define the working range of the detector. Recommended calibration levels are given in the appendices.

10.1. A new calibration curve must be generated after major changes to the system or when the continuing calibration criteria cannot be met. Major changes include new columns, changing PID lamps or FID jets or replacing the ECD detector. A new calibration is not required after clipping the column, replacing the septum or syringe, or other minor maintenance.

10.2. With the exception of 10.3 below, it is NOT acceptable to remove points from a calibration curve for the purpose of meeting criteria, unless the points are the highest or lowest on the curve AND the reporting limit and/or linear range is adjusted accordingly. In any event, at least 5 points must be included in the calibration curve. Quadratic (second order) calibrations require at least six points. Third order calibrations require at least seven points.

10.3. A level may be removed from the calibration if the reason can be clearly documented, for example a broken vial or no purge run. A minimum of five levels must remain in the calibration. The documentation must be retained with the initial calibration. Alternatively, if the analyst believes that a point on the curve is inaccurate, the point may be reanalyzed and the reanalysis used for the calibration. All initial calibration points must be analyzed without any changes to instrument conditions, and all points must be analyzed within 24 hours.

10.4. External standard calibration

Quantitation by the external standard method assumes a proportional relationship between the calibration run and the analyte in the sample. To use this approach introduce each calibration standard into the GC using the technique that will be used for samples. The ratio of the peak height or area response to the mass or concentration injected may be used to prepare a calibration curve.

$$Calibration\ Factor\ (CF) = \frac{Area\ or\ Height\ of\ Peak}{Mass\ Injected\ (ng)}$$

Some data systems may use the inverse of this formula. This is acceptable so long as the same formula is used for standards and samples. It is also possible to use the concentration of the standard rather than the mass injected. (This would require changes in the equations used to calculate the sample concentrations). Use of peak area or height must be consistent. However, if matrix interferences would make quantitation using peak area inaccurate for a particular sample, then peak height may be used as a substitute.

10.5. Internal standard calibration

10.5.1. The internal standard approach assumes that variations in instrument sensitivity, amount injected etc. can be corrected by determining the ratio of the response of the analyte to the response of an internal standard that has been added to the extract. To use this approach, select one or more internal standard(s) that are similar in analytical behavior to the compounds of interest. Recommended internal standards are given in the appendices. The analyst must demonstrate that the measurement of the internal standard is not affected by method or matrix interferences. If the sample matrix interferes with quantitation of the internal standard, then the external standard approach must be used instead. In this event use the response factors from the previous continuing calibration to quantitate the analytes in the sample with the interference (applies only to the sample with the interference).

10.5.2. Introduce each calibration standard into the GC using the technique that will be used for samples. Response factors (RF) for each compound are calculated as follows:

$$RF = \frac{A_s \times C_{is}}{A_{is} \times C_s}$$

Where:

A_s = Response for the analyte to be measured
A_{is} = Response for the internal standard
C_{is} = Concentration of internal standard
C_s = Concentration of the analyte to be determined in the standard

10.6. Calibration curve fits

Average response factor, linear regression, or quadratic curves may be used to fit the data. Average response factor may be used if the average % RSD of the response factors or calibration factors of all the analytes in the calibration standard taken together is ≤ 20%. The average %RSD is calculated by summing the RSD value for each analyte and dividing by the total number of analytes.

10.6.1. In general, for environmental analysis, average response factors are the most appropriate calibration model. Linear or curved regression fits should only be used if the analyst has reason to believe that the average RF model does not fit the normal concentration/response behavior of the detector.

10.6.2. Average response factor

The average response factor may be used if the average percent relative standard deviation (%RSD) of all the response factors taken together is ≤ 20%.

The equation for average response factor is:

$$Average\ response\ factor = \overline{RF} = \frac{\sum_{i=1}^{n} RF_i}{n}$$

Where: n = Number of calibration levels

$\sum_{i=1}^{n} RF_i$ = Sum of response factors for each calibration level

C4-506

10.6.3. Linear regression

The linear fit uses the following functions:

10.6.3.1. External Standard

$$y = ax + b$$

or

$$x = \frac{(y-b)}{a}$$

Where: y = Instrument response
x = Concentration
a = Slope
b = Intercept

10.6.3.2. Internal Standard

$$C_s = \frac{\left[\frac{A_s C_{is}}{A_{is}} - b\right]}{a}$$

Where: C_s = Concentration in the sample
A_s = Area of target peak in the sample
A_{is} = Area of internal standard in the sample
C_{is} = Concentration of the internal standard

10.6.4. Quadratic curve

The quadratic curve uses the following functions:

10.6.4.1. External standard

$$y = ax + cx^2 + b$$

Where c is the curvature

10.6.4.2. Internal Standard

$$y = a\left(\frac{A_s \times C_{is}}{A_{is}}\right) + c\left(\frac{A_s \times C_{is}}{A_{is}}\right)^2 + b$$

10.7. Evaluation of calibration curves

10.7.1. The percent relative standard error (%RSE) from the calibration curve is used to evaluate the initial calibration. This provides a measure of how much error is associated with using the calibration curve for quantitation.

10.7.2. The least squares regression line is calculated and used to calculate the predicted concentration for each level. The percent relative standard error is calculated as follows:

$$\% RSE = 100\% \times \sqrt{\frac{\sum_{i=1}^{N}\left[\frac{C_i - PC_i}{C_i}\right]^2}{(N-P)}}$$

Where:
N = Number of points in the curve
P = Number of parameters in the curve (= 1 for average response factor, 2 for linear, 3 for quadratic)
C_i = True concentration for level i
PC_i = Predicted concentration for level i

Note that when average response factors are used, %RSE is equivalent to %RSD.

10.8. The following requirements must be met for any calibration to be used:

- Response must increase with increasing concentration.
- If a curve is used, the intercept of the curve at zero response must be less than ± the reporting limit for the analyte.
- The average Relative Standard Error (RSD for average response factors) of the calibration points from the curve used must be ≤ 20%.
- Some data systems will not measure the %RSE from a linear or quadratic fit. For the linear case, the correlation coefficient may be used as an alternative to the %RSE, and must be greater than or equal to 0.990. For the quadratic case the Coefficient of Determination may be used, and must be greater or equal to 0.990.

Note: The Relative Standard Error (RSE) is superior to the Correlation Coefficient (r^2) and Coefficient of Determination (r) for testing the fit of a set of calibration points to a line. The lower points on a curve have little effect on r. As a result a curve may have a very good correlation coefficient (>0.995), while also having > 100% error at the low point. (Edgerley, "Improving Calibration Accuracy in the Environmental Laboratory", in press).

10.9. Weighting of data points

10.9.1. In a quadratic calibration fit, the points at the lower end of the calibration curve have less relative variance than points at the high concentration end of the curve. This can cause severe errors in quantitation at the low end of the calibration. However, in environmental analysis, accuracy at the low end of the curve is very important. For this reason it is preferable to increase the weighting of the lower concentration points. 1/Concentration^2 weighting (often called $1/X^2$ weighting) will improve accuracy at the low end of the curve and should be used if the data system has this capability. Use of $1/X^2$ weighting for linear fits is currently not permitted by method 8000B.

10.10. Non-standard analytes are sometimes requested. For these analytes, it may be acceptable to analyze a single standard at the reporting limit with each continuing calibration rather than a five point initial calibration. This action must be with client approval. If the analyte is detected in any of the samples, a five point initial calibration must be generated and the sample(s) reanalyzed for quantitation.

10.11. Calibration Verification

10.11.1. 12 hour Calibration

The working calibration curve or RF must be verified by the analysis of a mid point calibration standard at the beginning, after every 12 hours, and at the end of the analysis sequence. The center of each retention time window is updated with each 12 hour calibration.

10.11.2. Calibration Verification

It may be appropriate to analyze a mid point standard more frequently than every 12 hours. If these calibration verification standards are analyzed, requirements are the same as the 12 hour calibration with the exception that retention times are not updated.

10.11.3. Any analyte that is reportable as found must have a % difference of $\leq$ 15% in the preceding verification or 12 calibration, on the column used for quantitation. Refer to section 12.1.2 for which result to report.

10.11.4. Analytes not detected in client samples will ideally also have a %D of $\leq$ 15%. However, the analysis is acceptable if the average of the %D for all the analytes is $\leq$ 15%. This average is calculated by summing all the %D results in the calibration and dividing by the number of analytes.

10.11.5. Reportable as found is defined as any analyte that would be reported as anything other than a non-detect.

10.11.6. It is not necessary to run a calibration verification standard at the beginning of the sequence if samples are analyzed immediately after the completion of the initial calibration.

10.11.7. Samples quantitated by external standard methods must be bracketed by calibration verification standards that meet the criteria listed above. Bracketing is not necessary for internal standard methods.

10.11.8. % Difference calculation

% Difference for internal and external methods is calculated as follows

Internal Standard:

$$\%D = \frac{RF_c - \overline{RF}}{\overline{RF}} \times 100$$

External standard:

$$\%D = \frac{CF_c - \overline{CF}}{\overline{CF}} \times 100$$

Where RF_c and CF_c are the response and calibration factors from the continuing calibration

$\overline{RF}$ and $\overline{CF}$ are the average response and calibration factors from the initial calibration

10.11.9. % Drift calculation

% Drift is used for comparing the continuing calibration to a linear or quadratic curve. The criteria for % drift are the same as for % difference

$$\%\ Drift = \frac{Calculated\ Conc.\ -\ Theoretical\ Conc.}{Theoretical\ Conc.} \times 100\%$$

10.11.10. Corrective Actions for Continuing Calibration

If the overall average %D of all analytes is greater than ± 15% corrective action must be taken. This may include clipping the column, changing the liner or other minor instrument adjustments, followed by reanalyzing the standard. If the overall average %D still varies by more than ± 15%, a new calibration curve must be prepared.

10.11.11. Corrective Action for Samples

For internal standard methods, any samples injected after a standard not meeting the calibration criteria must be reinjected.

For external standard methods, any samples injected after the last good continuing calibration standard must be reinjected.

11. PROCEDURE

11.1. Extraction

Extraction procedures are referenced in the appendices.

11.2. Cleanup

Cleanup procedures are referenced in the appendices.

11.3. Gas Chromatography

Chromatographic conditions for individual methods are presented in the appendices.

11.4. Sample Introduction

In general, volatiles analytes are introduced using purge and trap as described in Appendix A. Semivolatile analytes are introduced by direct injection of the extract. Samples, standards,,and QC must be introduced using the same procedure.

11.5. Analytical Sequence

An analytical sequence starts with an initial calibration or a daily calibration. Refer to the individual method appendices for method specific details of daily calibrations and analytical sequences.

11.5.1. The daily calibration includes analysis of standards containing all single response analytes and updating the retention time windows.

11.5.2. If there is a break in the analytical sequence of greater than 12 hours, a new analytical sequence must be started with a daily calibration.

11.6. Retention Time Windows

11.6.1. Retention time windows must be determined for all analytes. Make an injection of all analytes of interest each day over a three day period. Calculate the standard deviation of the three retention times for each analyte (relative retention times may also be used). For multiresponse analytes (e.g., Aroclors) use the retention time of major peaks. Plus or minus three times the standard deviation of the retention times of each analyte defines the retention time window.

11.6.2. The center of the retention time window is the retention time from the last of the three standards. The centers of the windows are updated with the mid point of the initial calibration and each 12 hour calibration. The widths of the windows will remain the same until new windows are generated following the installation of a new column.

11.6.3. If the retention time window as calculated above is less than +/- 0.05 minutes, use +/- 0.05 minutes as the retention time window. This allows for slight variations in retention times caused by sample matrix.

11.6.4. The laboratory must calculate new retention time windows each time a new column is installed. The new windows must be generated within one week of the installation of the new column. Until these standards have been run on the new column, the retention time windows from the old column may be used, updated with the retention times from the new initial calibration.

11.6.5. Corrective Action for Retention Times

The retention times of all compounds in each continuing calibration must be within the retention time windows established by the 12 hour calibration. If this condition is not met, all samples analyzed after the last compliant standard must be reanalyzed unless the following conditions are met for any compound that elutes outside the retention time window:

The retention time of that compound in the standard must be within a retention time range equal to twice the original window.

No peak that would be reportable may be present on the sample chromatogram within an elution time range equal to three times the original retention time window.

11.7. Daily Retention Time Windows

The center of the retention time windows determined in section 11.6 are adjusted to the retention time of each analyte as determined in the 12 hour calibration standards. (See the method 8081A and 8082 appendices for exceptions for multi-response components.) The retention time windows must be updated at the beginning of each analytical sequence and with each 12 hour calibration, but not for any other calibration verification standards.

11.8. Percent Moisture

Analytical results may be reported as dry or wet weight, as required by the client. Percent moisture must be determined if results will be reported as dry weight. Refer to SOP CORP-OP-0001 for determination of percent moisture.

11.9. Procedural Variations

Procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and approved by a supervisor and QA/QC manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file. The nonconformance is also addressed in the case narrative. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Qualitative Identification

12.1.1. Tentative identification occurs when a peak is found within the retention time window for an analyte, at a concentration above the reporting limit, or above the MDL if J flags are required. Normally confirmation is required on a second column, but if the detector is sufficiently specific or if the sample matrix is well enough defined, single column analysis may be adequate. In some cases GC/MS confirmation may be required. Client specific requirements may also define the need for second column confirmation and / or GC/MS confirmation. Refer to the appendices for test specific requirements for confirmation. Identification is confirmed if a peak is also present in the retention time window for that analyte on the confirmatory column, at a concentration greater than the reporting limit (MDL if J flag confirmation required).

12.1.2. Dual column quantitation

For confirmed results, two approaches are available to the analyst;
A) The primary column approach
Or
B) The better result approach
Both are acceptable to avoid the reporting of erroneous or unconfirmed data.

12.1.2.1. Primary column approach:

The result from the primary column is normally reported. The result from the secondary column is reported if any of the following three bulleted possibilities are true.

- There is obvious chromatographic interference on the primary column
- The result on the primary column is > 40% greater than the result on the secondary column
- Continuing or bracketing standard fails on the primary column but is acceptable on the secondary column. (If the primary column result is > 40% higher than the secondary, and the primary column calibration fails, then the sample must be evaluated for reanalysis.)

12.1.2.2. Better result approach

The lower of the two results is normally reported. The lower result is considered better because the higher result is generally higher because of chromatographic interference. The higher result is reported if any of the following two bulleted possibilities are true.

- There is obvious chromatographic interference on the column with the lower result
- The continuing or bracketing calibration on the column with the lower result fails. (If the higher result is > 40% higher and the calibration on the column with the lower result fails, then the sample must be evaluated for reanalysis.)

12.1.3. If the Relative percent difference (RPD) between the response on the two columns is greater than 40%, or if the opinion of an experienced analyst is that the complexity of the matrix is resulting in false positives, the confirmation is suspect and the results are qualified. RPD is calculated using the following formula:

$$RPD = \frac{R_1 - R_2}{\frac{1}{2}(R_1 + R_2)}$$

Where R=Result

12.1.4. Multi-response Analytes

For multi-response analytes, the analyst should use the retention time window, but should rely primarily on pattern recognition. The pattern of peaks will normally serve as confirmation.

12.1.5. The experience of the analyst should weigh heavily in the interpretation of the chromatogram. For example, sample matrix or laboratory temperature fluctuation may result in variation of retention times.

12.2. Calibration Range

If concentrations of any analytes exceed the working range as defined by the calibration standards, then the sample must be diluted and reanalyzed. Dilutions should target the most concentrated analyte in the upper half (over 50% of the high level standard) of the calibration range. It may be necessary to dilute samples due to matrix.

12.3. Dilutions

Samples may be screened to determine the appropriate dilution for the initial run. If the initial diluted run has no hits or hits below 20% of the calibration range and the matrix allows for analysis at a lesser dilution, then the sample must be reanalyzed at a dilution targeted to bring the largest hit above 50% of the calibration range.

12.3.1. Guidance for Dilutions Due to Matrix

If the sample is initially run at a dilution and only minor matrix peaks are, then the sample should be reanalyzed at a more concentrated dilution. Analyst judgement is required to determine the most concentrated dilution that will not result in instrument contamination.

12.3.2. Reporting Dilutions

The most concentrated dilution with no target compounds above the calibration range will be reported. Other dilutions will only be reported at client request.

12.4. Interferences

If peak detection is prevented by interferences, further cleanup should be attempted. If no further cleanup is reasonable, then elevation of reporting levels and/or lack of positive identification must be addressed in the case narrative.

12.5. *Internal Standard Criteria for Continuing Calibration*

If internal standard calibration is used, then the internal standard response in a continuing calibration standard must be within 50 to 150% of the response in the mid level of the initial calibration.

12.6. Calculations

Capabilities of individual data systems may require the use of different formulas than those presented here. When this is the case, the calculations used must be shown to be equivalent and must be documented in an appendix attached to this document.

12.6.1. External Standard Calculations

12.6.1.1. Aqueous samples

$$Concentration\ (mg\ /\ L) = \frac{(A_x \times V_t \times D_f)}{(CF \times V_i \times V_s)}$$

Where:

A_x = Response for the analyte in the sample
V_i = Volume of extract injected, µL
D_f = Dilution factor
V_t = Volume of total extract, µL
V_s = Volume of sample extracted or purged, mL
CF = Calibration factor, area or height/ng, Section 10.1

12.6.1.2. Non-aqueous Samples

$$Concentration\ (mg\ /\ kg) = \frac{(A_x \times V_t \times D_f)}{(CF \times V_i \times W \times D)}$$

Where:

W = Weight of sample extracted or purged, g

$$D = \frac{100 - \%\ Moisture}{100}$$ (D = 1 if wet weight is required)

12.6.2. Internal Standard Calculations

12.6.2.1. Aqueous Samples

$$Concentration\ (mg\ /\ L) = \frac{(A_x \times C_{is} \times D_f)}{(A_{is} \times RF \times V_s)}$$

Where:

C_{is} = Amount of internal standard added, ng
A_{is} = Response of the internal standard
RF = Response factor for analyte

12.6.2.2. Non-aqueous Samples

$$Concentration\ (mg\ /\ kg) = \frac{(A_x \times C_{is} \times D_f)}{(A_{is} \times RF \times W \times D)}$$

12.6.3. Surrogate Recovery

Concentrations of surrogate compounds are calculated using the same equations as for the target compounds. The response factor from the initial calibration is used. Surrogate recovery is calculated using the following equation:

$$\%\ \text{Recovery} = \frac{Concentration\ (or\ amount)\ found}{Concentration\ (or\ amount)\ spiked} \times 100$$

13. METHOD PERFORMANCE

13.1. Method Detection Limit

Each laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in QA Policy #: QA-005.

13.2. Initial Demonstration

Each laboratory must make a one time initial demonstration of capability for each individual method. Demonstration of capability for both soils and water matrices is required. This requires analysis of QC check samples containing all of the standard analytes for the method. For some tests it may be necessary to use more than one QC check mix to cover all analytes of interest.

13.2.1. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to a mid level calibration.

13.2.2. Calculate the average recovery and standard deviation of the recovery for each analyte of interest. Compare these results with the acceptance criteria given in each appendix.

13.2.3. If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

13.3. Training Qualification

The group/team leader has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

14. POLLUTION PREVENTION

This method does not contain any specific modifications that serve to minimize or prevent pollution.

15. WASTE MANAGEMENT

Waste generated in this procedure will be segregated and disposed according to the facility hazardous waste procedures. The Environmental Health and Safety Director should be contacted if additional information is required.

16. REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition, Final Update III, December 1996, Section 8000B

17. MISCELLANEOUS

17.1. Modifications from Reference Method

17.1.1. Chapter 1 of SW-846 states that the method blank should not contain any analyte of interest at or above the Method Detection Limit. This SOP states that the Method Blank must not contain any analyte of interest at or above the reporting limit. Common lab contaminants are allowed to be up to 5 times the reporting limit in the blank following consultation with the client.

17.2. Modifications from Previous Revision

This SOP has been substantially revised to reflect changes to Method 8000 contained in Update III to SW-846.

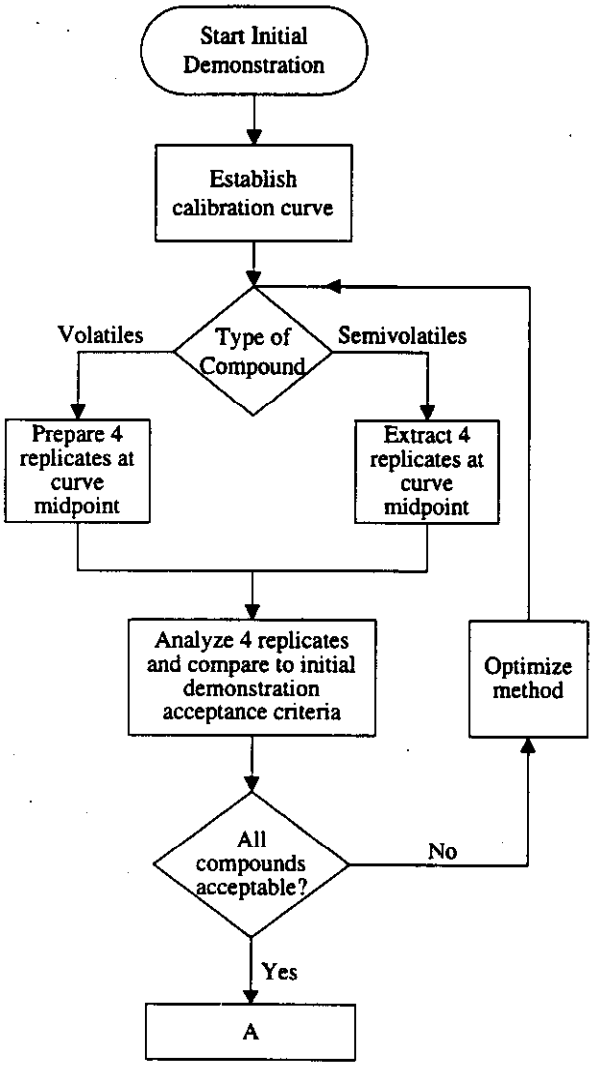
17.3. Facility Specific SOPs

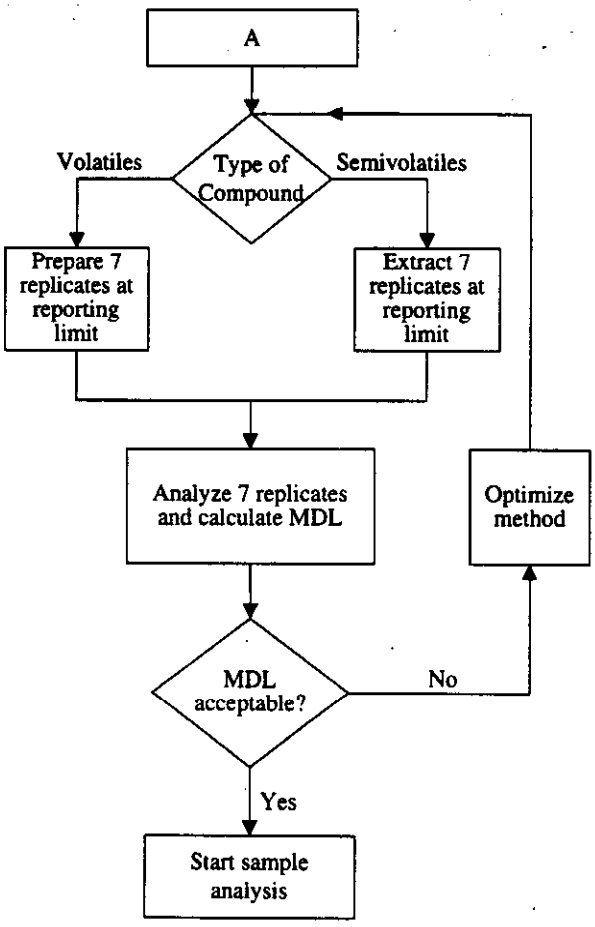
Each facility shall attach a list of facility specific SOPs or approved attachments (if applicable) which are required to implement this SOP or which are used in conjunction with this SOP. If no facility specific SOPs or amendments are to be attached, a statement must be attached specifying that there are none.

17.4.

Flow Diagrams

17.4.1. Initial demonstration and MDL[1]

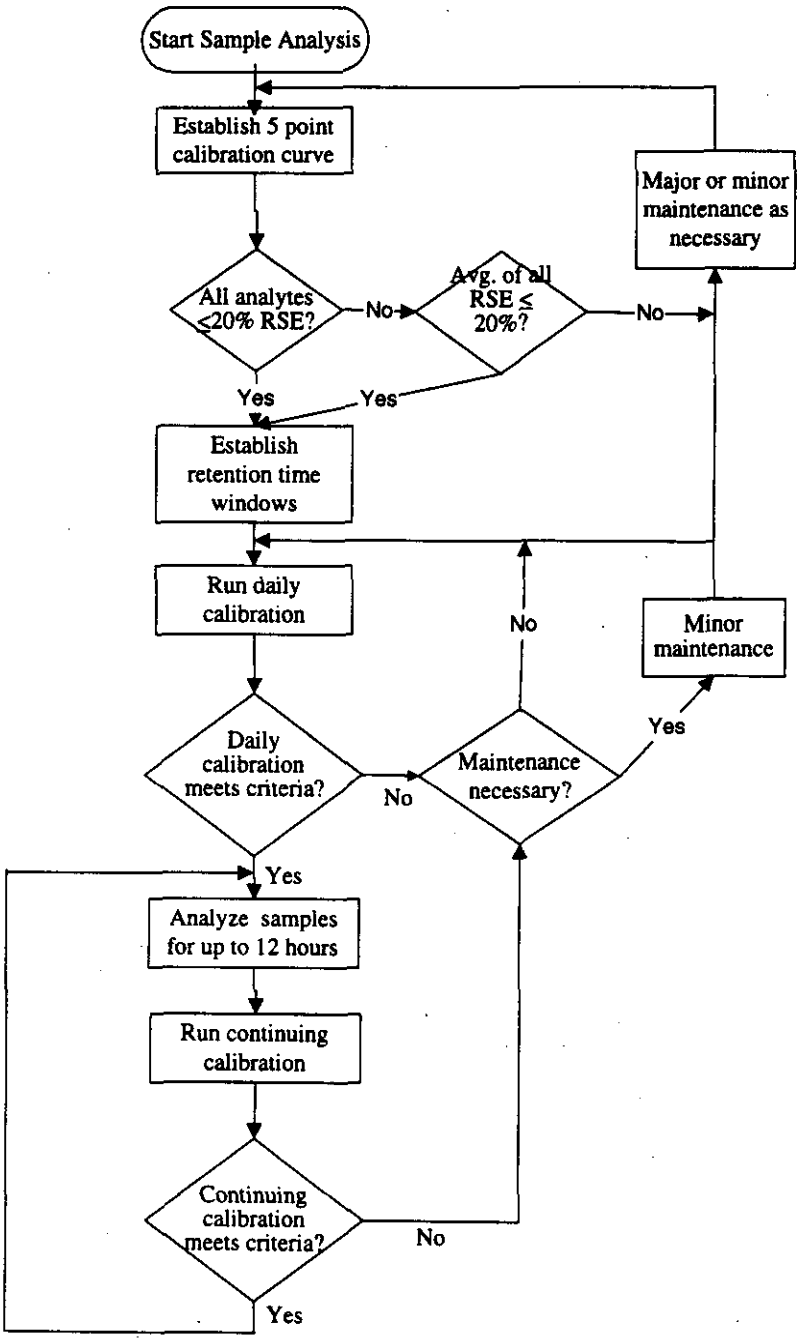




17.4.2.

[1] This flow diagram is for guidance and cannot cover all eventualities. Consult the SOP text and a supervisor if in doubt.

Sample Analysis[1]



[1] This flow diagram is for guidance and cannot cover all eventualities. Consult the SOP text and a supervisor if in doubt.

1. SCOPE AND APPLICATION

1.1. This method describes sample preparation and extraction for the analysis of volatile organics by a purge and trap procedure, following method 8021B. However, where required by a client QAPP this section may also be used to analyze aromatic volatiles by discontinued methods 8020A and 8010B. All requirements of the 8000B section of this SOP must be met except when superseded by this Appendix. Refer to Table A-1 for the individual analytes normally determined by these procedures.

1.2. Compounds within the scope of this method have boiling points below 200°C and are soluble or slightly soluble in water. Classes of compounds best suited to purge-and-trap analysis include low molecular weight halogenated hydrocarbons, aromatics, ketones, nitriles, acetates, acrylates, ethers, and sulfides.

1.3. Water samples and soils samples with low levels of contamination may be analyzed directly by purge-and-trap extraction and gas chromatography. Higher concentrations of these analytes in soil may be determined by the medium level methanol extraction procedure.

1.4. This method also describes the preparation of water-miscible liquids, non-water-miscible liquids, solids, wastes, and soils/sediments for analysis by the purge-and-trap procedure.

2. SUMMARY OF METHOD

2.1. An inert gas is bubbled through the sample at ambient temperature or at 40°C (40°C required for low level soils), and the volatile components are transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column where the volatile components are adsorbed. After purging is completed, the sorbent column is heated and backflushed with inert gas to desorb the components onto a gas chromatographic column. Analytes are detected using a photoionization Detector, an electrolytic conductivity detector or a combination of both.

2.2. For soil samples, a portion of the sample is dispersed in methanol to dissolve the volatile organic constituents. A portion of the methanolic solution is combined with water. It is then analyzed by purge-and-trap GC following the normal water method. If very low detection limits are needed for soil samples then direct purge using sodium bisulfate preservation may be necessary.

3. DEFINITIONS

Refer to the QAMP for definitions of terms used in this SOP.

4. INTERFERENCES

4.1. Refer to section 4 of the method 8000B part of this SOP for general information on chromatographic interferences.

4.2. Impurities in the purge gas, and from organic compounds out-gassing from the plumbing ahead of the trap, account for the majority of contamination problems. The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running laboratory reagent blanks. The use of non-TFE plastic tubing, non-TFE thread sealants, or flow controllers with rubber components in the purging device should be avoided.

4.3. Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) through the septum seal of the sample vial during shipment and storage. A trip blank prepared from organic-free reagent water and carried through sampling and handling protocols serves as a check on such contamination.

4.4. Contamination by carryover can occur whenever high-concentration and low-concentration samples are analyzed sequentially. Whenever an unusually concentrated sample is analyzed, it should be followed

by an analysis of organic-free reagent water to check for cross-contamination. The trap and other parts of the system are subject to contamination. Therefore, frequent bake-out and purging of the system may be required.

4.5. When utilizing an autosampler system which has multiple ports for sample analysis, it is likely that only a single stage or port may be contaminated by a highly concentrated sample. If a port is suspect, a water blank should be analyzed to verify lack of contamination. If the water blank and subsequent blanks on that port show contamination consistent with the concentrated sample, further maintenance is required. This may include replacing or cleaning the multi-port valve, transfer lines, etc.

4.6. A holding blank is kept in the sample refrigerator. This is analyzed and replaced every 14 days. If the holding blank does not meet the method blank criteria, the source of contamination must be found and corrected. Evaluation of all samples analyzed in the 14 day period prior to the analysis of the contaminated holding blank is required.

4.7. Acidification of samples may result in hydrolysis of 2-chloroethyl vinyl ether.

5. SAFETY

5.1. Refer to section 5 of the Method 8000B section of this SOP for general safety requirements.

5.2. Often, purge vessels on purge-and-trap instrumentation are pressurized by the time analysis is completed. Therefore, vent the pressure prior to removal of these vessels to prevent the contents from spraying out.

5.3. The toxicity or carcinogenicity of each chemical used in this procedure has not been fully defined. Additional health and safety information can be obtained from the MSDS files maintained in the laboratory. The following specific hazards are known:

Methanol -- Flammable and toxic

6. EQUIPMENT AND SUPPLIES

6.1. Microsyringes -- 10µL, 25µL, 100µL, 250µL, 500µL, and 1000µL. These should be equipped with a 20 gauge (0.006" ID) needle. These will be used to measure and dispense methanolic solutions and aqueous samples.

6.2. Gas tight syringes -- 5 mL and 25 mL. Used for measuring sample volumes.

6.3. Purge and Trap Apparatus -- A device capable of extracting volatile compounds, trapping on a sorbent trap, and introducing onto a gas chromatograph.

6.4. Purge and Trap Autosampler -- In order to maintain high sample throughput, an autosampler is highly recommended.

6.5. Trap -- The trap used is dependent on the class of compound to be analyzed. Refer to Table A-2 for suggested traps for specific tests.

6.6. Purge Vessels -- These are dependent on the purge and trap unit/autosampler used. Both disposable culture tubes (needle sparge units) and specially designed vessels with fritted bottoms may be used. Follow the manufacturer's suggestions for configuration.

6.7. Columns - Refer to Table A-2 for details of columns.

6.8. Volumetric flasks, Class A: 5 mL to 250 mL

6.9. pH paper

6.10. Balance capable of weighing to 0.01g for samples.

7. REAGENTS AND SUPPLIES

7.1. Refer to the method 8000B section of this SOP for general requirements for reagents and supplies.

7.2. Organic Free Water

Organic free water is defined as water in which an interferent is not observed at the reporting limit of the compounds of interest. Suggested methods for generating organic free water include:

- Filtration through a carbon bed.
- Continuously sparging water with helium or nitrogen.
- Use of commercial water purification systems.

Other methods may be used, so long as the requirement that the water not show any interferences is met. The procedure used should be documented in a lab specific attachment.

7.3. Sodium Bisulfate

7.4. Methanol -- Purge and Trap Grade

7.5. Standards

Refer to tables A-5 and A-6 for details of surrogate, matrix spiking and internal standards. Calibration standard levels are not specified, since they may depend on the sensitivity and linear range of specific detectors. However, the low level standard must be equivalent to the reporting limits specified in Table A-1.

7.5.1. Volatile standards are prepared by injecting a measured volume of the stock standard into a syringe containing the appropriate volume of organic free water. The calibration standard is then loaded into the purge device.

8. SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1. Holding times for all volatile analysis are 14 days from sample collection.

8.2. Water samples are normally preserved at pH < 2 with 1:1 hydrochloric acid. If residual chlorine is present, 2 drops of 10% sodium thiosulfate are added.

8.3. Solid samples are field preserved with sodium bisulfate solution for low level analysis, or with methanol for medium level analysis. Soil samples can also be taken using the EnCore™ sampler and preserved in the lab within 48 hours of sampling. At specific client request, unpreserved soil samples may be accepted.

8.4. There are several methods of sampling soil. The recommended method, which provides the minimum of field difficulties, is to take a EnCore sample. (The 5 g or 25 g sampler can be used, depending on client preference). Following shipment back to the lab the soil is preserved in methanol. This is the medium level procedure. If very low detection limits are needed (< 50 µg/kg for most analytes) then it will be necessary to use two additional 5 g EnCore samplers or to use field preservation.

8.5. Sample collection for medium level analysis using EnCore samplers.

8.5.1. Ship one 5 g (or 25 g) EnCore sampler per field sample position.

8.5.2. An additional bottle must be shipped for percent moisture determination.

8.5.3. When the samples are returned to the lab, extrude the (nominal) 5g (or 25 g) sample into a tared VOA vial containing 5 mL methanol (25 mL methanol for the 25 g sampler). Obtain the weight of the soil added to the vial and note on the label.

8.5.4. Add the correct amount of surrogate spiking mixture. (Add 100 µL of 250 µg/mL solution for a nominal 25 g sample, 20µL for a nominal 5 g sample.)

8.5.5. Add the correct amount of matrix spiking solution to the matrix spike and matrix spike duplicate samples. (Add 100 µL of 250 µg/mL solution for a nominal 25 g sample, 20µL for a nominal 5 g sample.) The addition of spike introduces a slight error, (0.4%) which can be neglected, into the calculations.

8.5.6. Prepare an LCS for each batch by adding the correct amount of matrix spiking solution to clean methanol. (100 µL of spike to 25 mL methanol or 20 µL spike to 5 mL methanol).

8.5.7. Shake the samples for two minutes to distribute the methanol throughout the soil.

8.5.8. Allow to settle, then remove a portion of methanol and store in a clean Teflon capped vial at 4+2°C until analysis.

8.6. Sample collection for medium level analysis using field methanol preservation

8.6.1. Prepare a VOA vial by adding 5 mL purge and trap grade methanol. (If a 25 g sample is to be used, add 25 mL methanol to the VOA vial).

8.6.2. Seal the bottle and attach a label.

8.6.3. Weigh the bottle to the nearest 0.01g and note the weight on the label.

8.6.4. Ship with appropriate sampling instructions.

8.6.5. Each sample will require an additional bottle with no preservative for percent moisture determination.

8.6.6. At client request, the methanol addition and weighing may also be performed in the field.

8.6.7. When the samples are returned to the lab, obtain the weight of the soil added to the vial and note on the label.

8.6.8. Add the correct amount of surrogate spiking mixture. (Add 100 µL of 250 µg/mL solution for a nominal 25 g sample, 20µL for a nominal 5 g sample.)

8.6.9. Add the correct amount of matrix spiking solution to the matrix spike and matrix spike duplicate samples. (Add 100 µL of 250 µg/mL solution for a nominal 25 g sample, 20µL for a nominal 5 g sample.) The addition of spike introduces a slight error, (0.4%) which can be neglected, into the calculations.

8.6.10. Prepare an LCS for each batch by adding the correct amount of matrix spiking solution to clean methanol. (100 µL of spike to 25 mL methanol or 20 µL spike to 5 mL methanol).

8.6.11. Shake the samples for two minutes to distribute the methanol throughout the soil.

8.6.12. Allow to settle, then remove a portion of methanol and store in a clean Teflon capped vial at 4+2°C until analysis.

8.7. Low level procedure

8.7.1. If low detection limits are required (typically < 50 µg/kg) sodium bisulfate preservation must be used. However, it is also necessary to take a sample for the medium level (methanol preserved) procedure, in case the concentration of analytes in the soil is above the calibration range of the low-level procedure.

8.7.2. A purge and trap autosampler capable of sampling from a sealed vial is required for analysis of samples collected using this method. (Varian Archon or O.I. 4552).

8.7.3. The soil sample is taken using a 5g EnCore sampling device and returned to the lab. It is recommended that two EnCore samplers be used for each field sample position, to allow for any reruns than may be necessary. A separate sample for % moisture determination is also necessary.

8.7.4. Prepare VOA vials by adding a magnetic stir bar, approximately 1 g of sodium bisulfate and 5 mL of reagent water.

8.7.5. Seal the vial and attach a label. The label must not cover the neck of the vial or the autosampler will malfunction.

8.7.6. Weigh the vial to the nearest 0.01g and note the weight on the label.

8.7.7. Extrude the soil sample from the EnCore sampler into the prepared VOA vial. Reweigh the vial to obtain the weight of soil and note on the label.

8.7.8. **Note:** Soils containing carbonates may effervesce when added to the sodium bisulfate solution. If this is the case at a specific site, add 5 mL of water instead, and freeze at ≥ -10°C until analysis.

8.7.9. Alternatively the sodium bisulfate preservation may be performed in the field. Ship at least two vials per sample. The field samplers must determine the weight of soil sampled. Each sample will require an additional bottle with no preservative for percent moisture determination, and an additional bottle preserved with methanol for the medium level procedure.

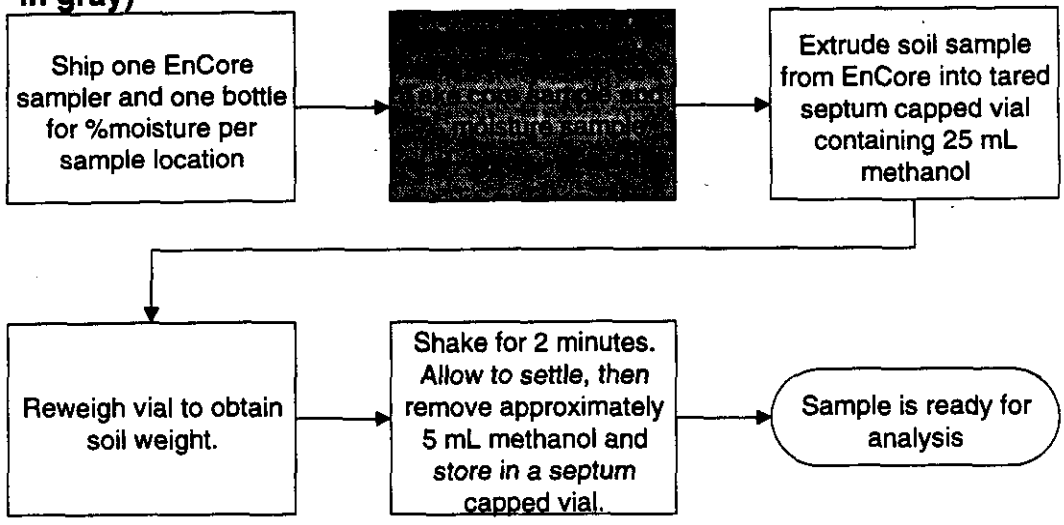
8.8. Aqueous samples are stored in glass containers with Teflon lined septa at 4°C +/- 2°C, with minimum headspace.

8.9. Medium level solid extracts are aliquoted into 2 - 5 mL glass vials with Teflon lined caps and stored at 4°C +/- 2°C. The extracts are stored with minimum headspace.

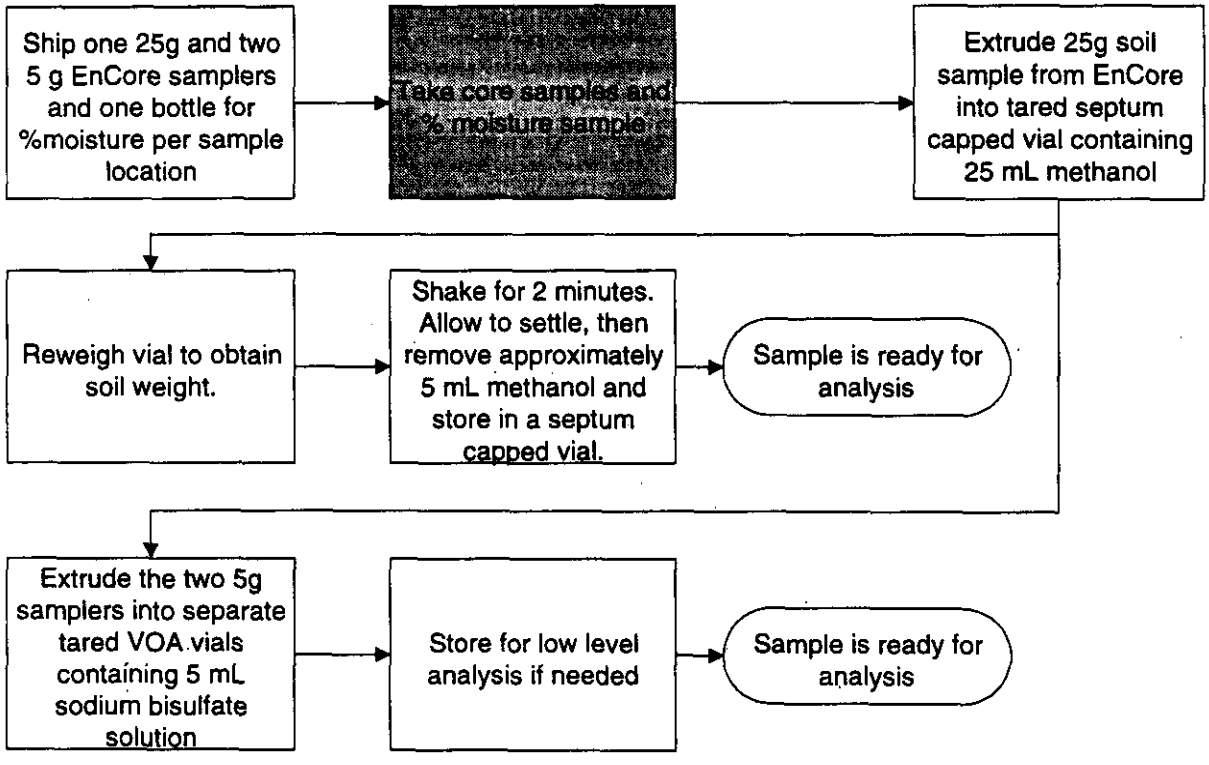
8.10. The maximum holding time is 14 days from sampling until the sample is analyzed. (Samples that are found to be unpreserved still have a 14 day holding time. However they should be analyzed as soon as possible. The lack of preservation should be addressed in the case narrative). Maximum holding time for the EnCore sampler (before the sample is added to methanol or sodium bisulfate) is 48 hours.

8.11. A holding blank is stored with the samples. This is analyzed and replaced if any of the trip blanks show any contamination. Otherwise it is replaced every 14 days.

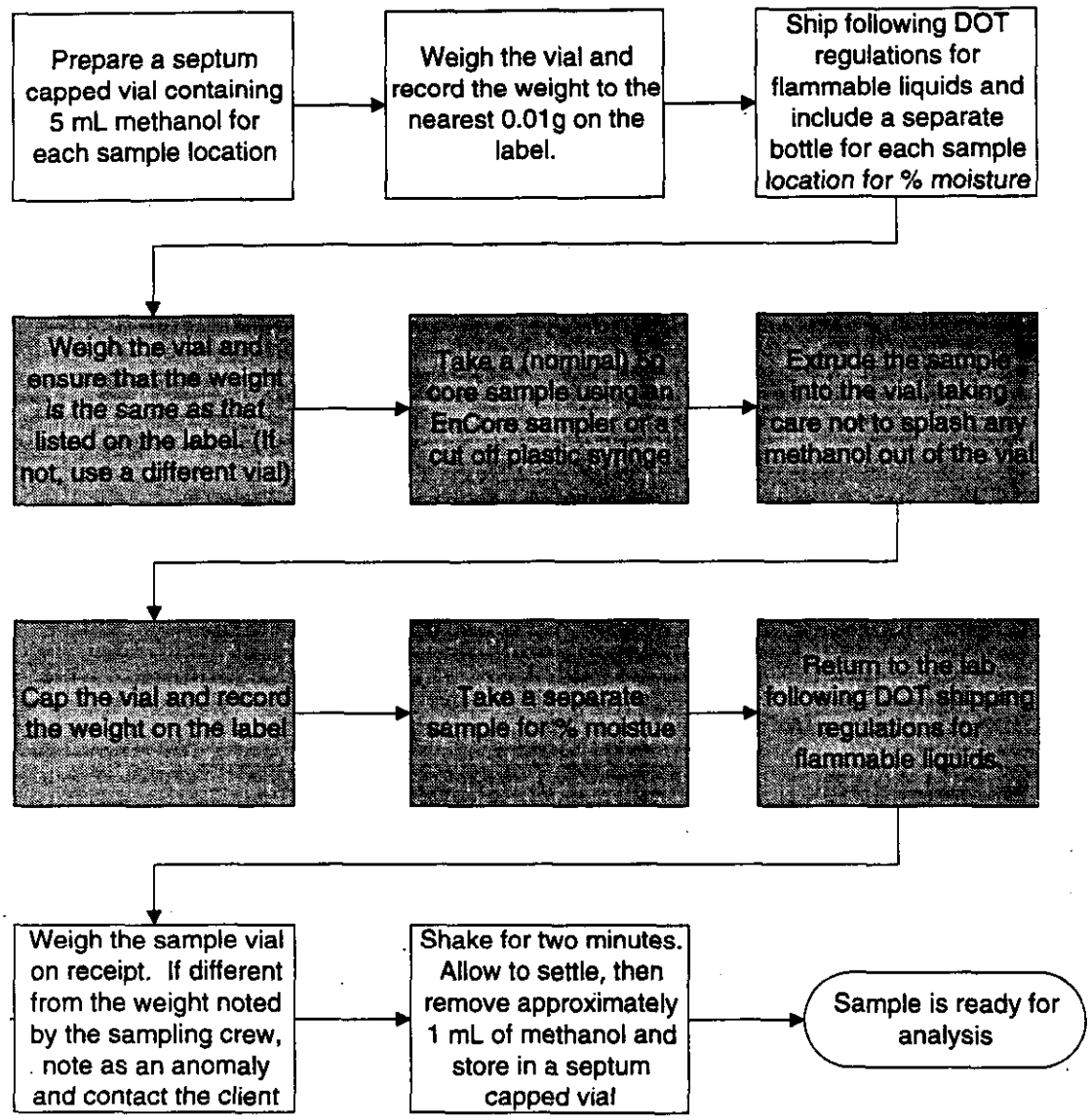
EnCore procedure when low level is not required (field steps in gray)



EnCore procedure when low level is required

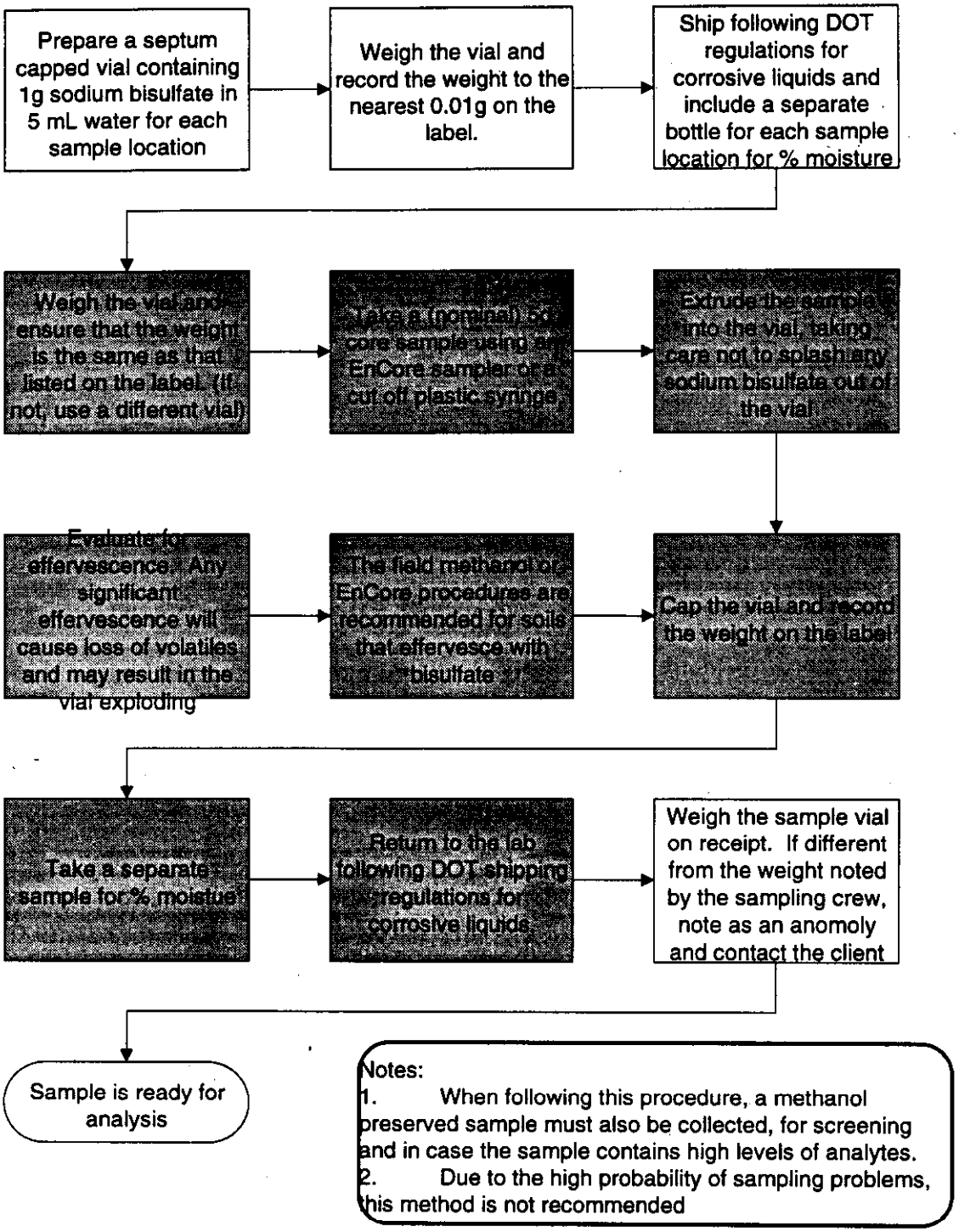


Field methanol extraction procedure (field steps in gray)



9.

Field bisulfate preservation procedure (field steps in gray)



Notes:
1. When following this procedure, a methanol preserved sample must also be collected, for screening and in case the sample contains high levels of analytes.
2. Due to the high probability of sampling problems, this method is not recommended

9. QUALITY CONTROL

9.1. Refer to the method 8000B section of this SOP, section 9, for general quality control procedures, including batch definition, requirements for method blanks, LCS, matrix spikes, surrogates, and control limits.

10. CALIBRATION AND STANDARDIZATION

10.1. Refer to the method 8000B section of this SOP, section 10, for general calibration procedures.

10.2. Gas Chromatograph Operating Conditions

Various column configurations are possible. If dual column confirmation is necessary, the sample may be split using a Y splitter at the injector end to direct the sample to two columns and two detectors. For simultaneous determination of aromatic and halogenated volatiles, a single column is used and the PID and ELCD detectors are connected in series.

10.2.1. Refer to Table A-2, A-3 and A-4 for GC operating conditions.

10.3. Initial Calibration

10.3.1. Refer to Section 10 of the 8000B section of this SOP for details of initial calibration criteria.

10.3.2. Low level soil samples must be purged at 40°C, therefore the calibration curve must also be purged at 40°C. In addition, the low level soil calibration solutions should contain approximately the same amount of sodium bisulfate as the samples.

10.3.3. The low level calibration must be at the reporting limit or below. The remaining standards encompass the working range of the detector.

10.3.4. Calibrate the instrument using the same volume that will be used during sample analysis.

10.4. Calibration Verification

10.4.1. A mid level calibration standard is used for the calibration verification. The gases have a 20%D criteria rather than the 15% used for other analytes.

10.4.2. A calibration verification run is performed after every 10 samples for this method.

10.4.3. Bracketing of samples with calibration verification runs is only necessary for external standard analysis.

11. PROCEDURE

11.1. Refer to the method 8000B section of this SOP for general procedural requirements.

11.2. Analytical Sequence

The analytical sequence starts with an initial calibration of at least five points, or a 12 hour calibration that meets % difference criteria from an existing initial calibration.

11.3. Confirmation

The PID and ELCD detectors are sufficiently selective that second column confirmation is not always necessary. Requirements for second column confirmation should be decided in consultation with the client. If the PID and ELCD are used in series confirmatory information for many analytes can be gained by comparing the relative response from the two detectors.

11.4. Aqueous Sample Analysis (Purge and Trap units using sparge vessels)

11.4.1. Depending on the sensitivity of the instrument and capabilities of the purge and trap device, 5, 10, 20, or 25 mL sample volumes may be analyzed. A 5 mL sample volume is recommended.

11.4.2. Rinse a 5 mL (or 25 mL for larger sample volumes) gas-tight syringe with organic free water. Fill the syringe with the sample to be analyzed, and compress to volume.

11.4.3. Check and document the pH of the sample remaining in the VOA vial after loading the syringe.

11.4.4. This procedure invalidates the contents of the VOA vial for further analysis, unless an aliquot is transferred to a smaller VOA vial with no headspace (e.g., 20 mL) at the same time the analysis aliquot is removed.

11.4.5. Spike with the appropriate volume of surrogate/internal standard solution and spike solution (if required) through the barrel of the syringe. The method blank is spiked with surrogates only, the LCS and matrix spikes with the surrogate and matrix spiking solutions. Refer to Tables A-5 and A-6 for volumes and concentrations of spiking solutions.

11.4.6. Load onto the purge and trap device and start the run.

11.4.7. If the initial analysis of a sample or a dilution of the sample has a concentration of analytes that exceeds the initial calibration range, the sample must be reanalyzed at a higher dilution. When a sample has a high response for a compound, analysis should be followed by an organic free water blank. It is recognized that during automated unattended analysis, this may not occur. If any potential carryover hits are present in samples following highly contaminated samples, the sample must be reanalyzed to determine if any of these hits are a result of carryover or are actually present in the sample.

11.4.8. Dilutions may be made in gas tight syringes unless the volume of sample used is less than 5 µL, in which case dilution in volumetric flasks will be necessary.

11.4.8.1. Spike with the same volume of surrogate/internal standard solution as used for undiluted samples prior to loading onto the purge and trap device.

11.4.8.2. For Matrix spike / matrix spike duplicates where the sample requires dilution, the sample is spiked <u>after</u> the dilution is performed.

11.5. Aqueous and Soil Sample Analysis (Purge and Trap units that sample directly from the VOA vial)

11.5.1. Units which sample from the VOA vial should be equipped with a module which automatically adds surrogate and internal standard solution to the sample prior to purging the sample.

11.5.2. If the autosampler uses automatic IS/SS injection, no further preparation of the VOA vial is needed. Otherwise the internal and surrogate standards must be added to the vial. *Note:* Aqueous samples with high amounts of sediment present in the vial may not be suitable for analysis on this instrumentation, or they may need to analyzed as soils.

11.5.3. Sample remaining in the vial after sampling with one of these mechanisms is no longer valid for further analysis. A fresh VOA vial must be used for further sample analysis.

11.5.4. Check the pH of the sample remaining in the VOA vial after analysis is completed.

11.6. Low-Level Solids Analysis using discrete autosamplers

Note: This technique may seriously underestimate analyte concentration and must not be used except at specific client request for the purpose of comparability with previous data. It is no longer part of SW-846.

This method is based on purging a heated sediment/soil sample mixed with reagent water containing the surrogate and, if applicable, internal and matrix spiking standards. Analyze all reagent blanks and standards under the same conditions as the samples (e.g., heated). The calibration curve is also heated during analysis. Purge temperature is 40°C.

11.6.1. Do not discard any supernatant liquids. Mix the contents of the container with a narrow metal spatula.

11.6.2. Weigh out 5 g (or other appropriate aliquot) of sample into a disposable culture tube or other purge vessel. Record the weight to the nearest 0.1 g. If method sensitivity is demonstrated, a smaller aliquot may be used. Do not use aliquots less than 1.0 g. If the sample is contaminated with analytes such that a purge amount less than 1.0 g is appropriate, use the medium level method described in section 11.7.

11.6.3. Connect the purge vessel to the purge and trap device.

11.6.4. Rinse a 5 mL gas-tight syringe with organic free water, and fill. Compress to 5 mL. Add surrogate/internal standard (and matrix spike solutions if required.) (See Tables A-5, A-6, A-7 and A-8.) Add directly to the sample from 11.6.2.

11.6.5. The above steps should be performed rapidly and without interruption to avoid loss of volatile organics.

11.6.6. *Add the heater jacket or other heating device and start the purge and trap unit.*

11.6.7. Soil samples that have low IS recovery when analyzed (<50%) should be reanalyzed once to confirm matrix effect. If external standard calibration is used, samples with surrogate recovery below the control limit should be reanalyzed once to confirm matrix effect.

11.7. Methanol Extract Soils

11.7.1. Rinse a gas-tight syringe with organic free water. Fill the syringe with the same volume of organic free water as used in the calibrations. Add no more than 2% (v/v) (100 µL for a 5 mL purge) methanolic extract (from Section 8.5 or 8.6) to the syringe. Add internal standard (if used). Load the sample onto the purge and trap device and analyze as for aqueous samples. If less than 5µL of methanolic extract is to be added to the water, dilute the methanolic extract such that a volume greater than 5µL will be added to the water in the syringe.

12. DATA ANALYSIS AND CALCULATIONS

Refer to section 12 of the 8000B section of this SOP.

13. METHOD PERFORMANCE

13.1. Performance limits for the four replicate initial demonstration of capability required under Section 13.1 of the 8000B section of this SOP are 70-130%, with %RSD $\leq$ 25% The spiking level should be 20 µg/L. These limits are for guidance, and certain "difficult" analytes may fall outside this range. Any outliers must be discussed with Quanterra's Director of Quality Assurance and/or Director of Technology before proceeding with sample analysis.

14. POLLUTION PREVENTION

This method does not contain any specific modifications that serve to minimize or prevent pollution.

15. WASTE MANAGEMENT

15.1. Waste generated in this procedure will be segregated and disposed according to the facility hazardous waste procedures. The Environmental Health and Safety Director should be contacted if additional information is required.

16. REFERENCES

16.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition, Final Update III, December 1996, Sections 5000, 5030B, 5035 and 8021B

17. MISCELLANEOUS

17.1. Modifications from Reference Method

17.2. Modifications from previous revision

17.2.1. Substantial changes to this method were made to reflect changes in Update III of SW-846.

17.3. Facility Specific SOPs

Each facility shall attach a list of facility specific SOPs or approved attachments (if applicable) which are required to implement this SOP or which are used in conjunction with this SOP. If no facility specific SOPs or amendments are to be attached, a statement must be attached specifying that there are none.

17.4.

Tables

Table A-1

Standard Analyte List

Test	Compound	CAS number	Reporting Limit, µg/L or µg/kg		
			Aqueous	Low Soil	Medium Soil
Halogenated volatiles by 8021B	Bromodichloromethane	75-27-4	1.0	1.0	50
	Bromoform	75-25-2	1.0	1.0	50
	Bromomethane	74-83-9	1.0	1.0	50
	Carbon Tetrachloride	56-23-5	1.0	1.0	50
	Chlorobenzene	108-90-7	1.0	1.0	50
	Chloroethane	70-00-3	1.0	1.0	50
	2-Chloroethyl vinyl ether	110-75-8	5.0	5.0	250
	Chloroform	67-66-3	1.0	1.0	50
	Chloromethane	74-87-3	1.0	1.0	50
	Dibromochloromethane	124-48-1	1.0	1.0	50
	1,2-Dichlorobenzene	95-50-1	1.0	1.0	50
	1,3-Dichlorobenzene	541-73-1	1.0	1.0	50
	1,4-Dichlorobenzene	106-46-7	1.0	1.0	50
	Dichlorodifluoromethane	75-71-8	1.0	1.0	50
	1,1-Dichloroethane	75-34-3	1.0	1.0	50
	1,2-Dichloroethane	107-06-2	1.0	1.0	50
	1,1-Dichloroethene	75-45-4	1.0	1.0	50
	cis-1,2 Dichloroethene	156-59-4	1.0	1.0	50
	trans-1,2-Dichloroethene	156-60-5	1.0	1.0	50
	Dichloromethane(DCM)	75-09-2	5.0	5.0	250
	1,2-Dichloropropane	78-87-5	1.0	1.0	50
	cis-1,3-Dichloropropene	10061-01-5	1.0	1.0	50
	trans-1,3-Dichloropropene	10061-02-6	1.0	1.0	50
	1,1,2,2-Tetrachloroethane	79-34-5	1.0	1.0	50
	Tetrachloroethene	127-18-4	1.0	1.0	50
	1,1,1-Trichloroethane	71-55-6	1.0	1.0	50
	1,1,2-Trichloroethane	79-00-5	1.0	1.0	50
	Trichloroethene	79-01-6	1.0	1.0	50
	Trichlorofluoromethane	75-69-4	1.0	1.0	50
	Vinyl Chloride	75-01-4	1.0	1.0	50
Additional halogenated volatiles	Benzyl Chloride	100-44-7	5.0	5.0	250
	Bromobenzene	108-86-1	1.0	1.0	50
	Dibromomethane	74-95-3	1.0	1.0	50
	1,1,1,2-Tetrachloroethane	630-20-6	1.0	1.0	50
	1,2,3-Trichloropropane	96-18-4	1.0	1.0	50
BTEX by 8021B	Benzene	71-43-2	1.0	1.0	50
	Ethyl Benzene	100-41-4	1.0	1.0	50
	Toluene	108-88-3	1.0	1.0	50
	Xylenes (total)	1330-20-7	1.0	1.0	50
Aromatic volatiles by 8021B	Benzene	71-43-2	1.0	1.0	50
	Chlorobenzene	108-90-7	1.0	1.0	50
	1,2-Dichlorobenzene	75-34-3	1.0	1.0	50
	1,3-Dichlorobenzene	107-06-2	1.0	1.0	50

Table A-1

Standard Analyte List

Test	Compound	CAS number	Reporting Limit, µg/L or µg/kg		
			Aqueous	Low Soil	Medium Soil
	1,4-Dichlorobenzene	75-45-4	1.0	1.0	50
	Ethyl Benzene	100-41-4	1.0	1.0	50
	Toluene	108-88-3	1.0	1.0	50
	Xylenes (total)	1330-20-7	1.0	1.0	50
Additional aromatic and unsaturated volatiles	1,2,4 Trimethylbenzene	95-63-6	1.0	1.0	50
	1,3,5 Trimethylbenzene	108-67-8	1.0	1.0	50
	Acetone	67-64-1	10	10	500
	MEK (2-butanone)	78-93-3	5.0	5.0	250
	MIBK (4-methyl-2-pentanone)	108-10-1	5.0	5.0	250
	Naphthalene	91-20-3	2.0	2.0	250
	Styrene	100-42-5	1.0	1.0	50
	Methyl tert-butyl ether (MTBE)	1634-04-4	1.0	1.0	50
Combined halogenated and aromatic volatiles by 8021B	Benzene	71-43-2	1.0	1.0	50
	Bromobenzene	108-86-1	1.0	1.0	50
	Bromochloromethane	74-97-5	1.0	1.0	50
	Bromodichloromethane	75-27-4	1.0	1.0	50
	Bromoform	75-25-2	1.0	1.0	50
	Bromomethane	74-83-9	1.0	1.0	50
	n-butylbenzene	104-51-8	1.0	1.0	50
	sec-Butylbenzene	135-98-8	1.0	1.0	50
	tert-Butylbenzene	98-06-6	1.0	1.0	50
	Carbon Tetrachloride	56-23-5	1.0	1.0	50
	Chlorobenzene	108-90-7	1.0	1.0	50
	Chlorodibromomethane	124-48-1	1.0	1.0	50
	Chloroethane	75-00-3	1.0	1.0	50
	Chloroform	67-66-3	1.0	1.0	50
	Chloromethane	74-87-3	1.0	1.0	50
	2-Chlorotoluene	95-49-8	1.0	1.0	50
	4-Chlorotoluene	106-43-4	1.0	1.0	50
	1,2-Dibromo-3-Chloropropane(DBCP)	96-12-8	1.0	1.0	50
	1,2-Dibromoethane(EDB)	106-93-4	1.0	1.0	50
	Dibromomethane	74-95-3	1.0	1.0	50
	1,2-Dichlorobenzene	95-50-1	1.0	1.0	50
	1,3-Dichlorobenzene	541-73-1	1.0	1.0	50
	1,4-Dichlorobenzene	106-46-7	1.0	1.0	50
	Dichlorodifluoromethane	75-71-8	1.0	1.0	50
	1,1-Dichloroethane	75-34-3	1.0	1.0	50
	1,2-Dichloroethane	107-06-2	1.0	1.0	50
	1,1-Dichloroethene	75-35-4	1.0	1.0	50
	cis-1,2-Dichloroethene	156-59-4	1.0	1.0	50
	trans-1,2-Dichloroethene	156-60-5	1.0	1.0	50
	1,2-Dichloropropane	78-87-5	1.0	1.0	50
	1,3-Dichloropropane	142-28-9	1.0	1.0	50
	2,2-Dichloropropane	590-20-7	1.0	1.0	50
	1,1-Dichloropropene	563-58-6	1.0	1.0	50

Table A-1
Standard Analyte List

Test	Compound	CAS number	Reporting Limit, µg/L or µg/kg		
			Aqueous	Low Soil	Medium Soil
	cis-1,3-Dichloropropene	10061-01-5	1.0	1.0	50
	trans-1,3-Dichloropropene	10061-02-6	1.0	1.0	50
	Ethylbenzene	100-41-4	1.0	1.0	50
	Hexachlorobutadiene	87-68-3	1.0	1.0	50
	Isopropylbenzene	98-82-8	1.0	1.0	50
	p-Isopropyltoluene	99-87-6	1.0	1.0	50
	Methylene Chloride	75-09-2	5.0	5.0	250
	Naphthalene	91-20-3	2.0	2.0	250
	n-Propylbenzene	10306501	1.0	1.0	50
	Styrene	100-42-5	1.0	1.0	50
	1,1,1,2-Tetrachloroethane	630-20-6	1.0	1.0	50
	1,1,2,2-Tetrachloroethane	79-34-5	1.0	1.0	50
	Tetrachloroethene	127-18-4	1.0	1.0	50
	Toluene	108-88-3	1.0	1.0	50
	1,2,3-Trichlorobenzene	87-61-6	1.0	1.0	50
	1,2,4-Trichlorobenzene	120-82-1	1.0	1.0	50
	1,1,1-Trichloroethane	71-55-6	1.0	1.0	50
	1,1,2-Trichloroethane	79-00-5	1.0	1.0	50
	Trichloroethene	79-01-6	1.0	1.0	50
	Trichlorofluoromethane	75-69-4	1.0	1.0	50
	1,2,3-Trichloropropane	96-18-4	1.0	1.0	50
	1,2,4-Trimethylbenzene	95-63-6	1.0	1.0	50
	1,3,5-Trimethylbenzene	108-67-8	1.0	1.0	50
	Vinyl Chloride	75-01-4	1.0	1.0	50
	Xylenes (total)	1330-20-7	1.0	1.0	50

Table A-2
Recommended Conditions for Aromatic Volatiles

Parameter	Recommended Conditions
Temperature program	50°C, 1min, 10°C/min to 200°C,1min
Column 1	Rtx-502.2 or DB-502.2 60m x 0.53mm 3.0um
Column 2	Rtx-1 or DB-1 60m x 0.53mm 3.0 um
Carrier gas	Helium or hydrogen
Purge Flow / time	40 mL/min, 11 minutes
Desorb Temp / time	180°C, 2 minutes (220°C for Vocarb 3000)
Bake Time / temp	200°C, 12 minutes (230°C for Vocarb 3000)
Transfer line / valve temp	115°C

Table A-3
Recommended Conditions for Method Halogenated Volatiles

Parameter	Recommended Conditions
Temperature program	35°C, 12 min, then 4°C/min to 200°C, hold for 5 min
Column 1	DB-VRX or RTX-502.2 105m x 0.53 mm id df = 3.0um
Column 2	DB-1 or RTX-1 105m x 0.53 mm ID df = 3.0um
Column 3	Rtx - Volatiles 120m x 0.53mm ID df=2.0um
Carrier gas	Helium
Purge Flow / time	40 mL/min, 11 minutes
Desorb Temp / time	180°C, 2 minutes (220°C for Vocarb 3000)
Bake Time / temp	200°C, 12 minutes (230°C for Vocarb 3000)
Transfer line / valve temp	115°C

Table A-4
Recommended Conditions for Method Combined Aromatic and Halogenated Volatiles

Parameter	Recommended Conditions
Temperature program	35°C, 12 min, then 4°C/min to 200°C, hold for 5 min
Column 1	DB-VRX or RTX-502.2 105m x 0.53 mm id df = 3.0um
Column 2	DB-1 or RTX-1 105m x 0.53 mm ID df = 3.0um
Column 3	Rtx - Volatiles 120m x 0.53mm ID df=2.0um
Carrier gas	Helium
Purge Flow / time	40 mL/min, 11 minutes
Desorb Temp / time	180°C, 2 minutes (220°C for Vocarb 3000)
Bake Time / temp	200°C, 12 minutes (230°C for Vocarb 3000)
Transfer line / valve temp	115°C

Table A-5
Surrogate and Internal Standard Concentrations

Standard	Components	Working Solution µg/mL	Spike amount µL (for 5 mL purge)	Final concentration µg/L (µg/kg)
Aromatic volatiles IS/SS	4-Chlorotoluene (SS) 1-Chloro-4-fluorobenzene (IS)	20 40	5	20 40
Halogenated volatiles IS/SS	4-chlorotoluene (SS) 1-Chloro-4-fluorobenzene (IS)	20 40	5	20 40
Combined Aromatic and halogenated volatiles IS/SS	Fluorobenzene (SS)	20	5	20
	1,4-Dichlorobutane (SS)	20	5	20
	1-Chloro-4-fluorobenzene (IS)	40	10	40

It may be necessary to select different surrogates in order to minimize sample interferences. 1-chloro-4-fluorobenzene and 4-chlorotoluene are fairly well resolved from analytes listed in this SOP. However 4-chlorotoluene may sometimes be requested as a target analyte. Other surrogates that may be considered, and issues associated with their use are:

Bromochloromethane: Elutes very close to chloroform and cis-1,2-dichloroethene on the 502.2 column. May be a target analyte.

1,2-Bromochloroethane:

1-Chloro-2-fluorobenzene: Elutes close to ethylbenzene on DB-1 or Rtx-1 and close to m,p-xylene on 502.2

a,a,a-Trifluorotoluene: Good for aromatic volatiles, coelutes or very close to trichloroethene

Bromofluorobenzene: Close to 1,1,2,2-trichloroethane and 1,2,3-trichloropropane on the 502.2 column. Good on DB-1 or Rtx-1.

2-Bromo-1-chloropropane: May coelute with 1,1,2-trichloroethane

Table A-6
Concentrations for LCS and MS/MSD compounds

Standard	Components	Working Solution µg/mL	Spike amount µL (5 mL purge)	Final concentration µg/L (µg/kg)
Aromatic	Benzene	20	5	20
	Toluene	20		20
	Chlorobenzene	20		20
Halogenated	Chlorobenzene	20	5	20
	1,1-Dichloroethene	20		20
	Trichloroethene	20		20
Combination aromatic / halogenated	Benzene	20	5	20
	Toluene	20		20
	Chlorobenzene	20		20
	1,1-Dichloroethene	20		20
	Trichloroethene	20		20

1. SCOPE AND APPLICATION

This SOP Appendix describes procedures to be used when SW-846 Method 8081A is applied to the analysis of organochlorine pesticides by GC/ECD. This Appendix may also to be applied when discontinued SW-846 Method 8080A is requested, and is applicable to extracts derived from any matrix which are prepared according to the appropriate Quanterra sample extraction SOPs. (CORP-OP-0001)

Table B-1 lists compounds which are routinely determined by this method and gives the Reporting Limits (RL) for each matrix. RLs given are based on the low level standard and the sample preparation concentration factors. Matrix interferences may result in higher RLs than those listed.

At client request, this method may also be used for the analysis of PCBs (Arochlors) in combination with pesticides, although these are normally analyzed following method 8082, as described in Appendix C of this SOP. In any event, if samples for PCB analysis do not need the acid clean up procedure, then the same injection may be used for method 8081B and 8082, assuming all calibration and QC requirements for both methods are met. Extracts that have been acid cleaned may not be analyzed for pesticides, since several of the pesticides will be degraded.

2. SUMMARY OF METHOD

This method presents conditions for the analysis of prepared extracts of organochlorine pesticides. The pesticides are injected onto the column and separated and detected by electron capture detection. Quantitation may be by internal or external standard methods.

3. DEFINITIONS

Refer to the QAMP for definitions of terms used in this document.

4. INTERFERENCES

4.1. Refer to the method 8000B section of this SOP for information regarding chromatographic interferences.

4.2. Interferences in the GC analysis arise from many compounds amenable to gas chromatography that give a measurable response on the electron capture detector. Phthalate esters, which are common plasticizers, can pose a major problem in the determinations. Interferences from phthalates are minimized by avoiding contact with any plastic materials.

4.3. Sulfur will interfere and can be removed using procedures described in SOP CORP-OP-0001.

4.4. Interferences co-extracted from samples will vary considerably from source to source. The presence of interferences may raise quantitation limits for individual samples. Specific cleanups may be performed on the sample extracts, including florisil cleanup (Method 3620), Gel Permeation Chromatography (Method 3640), and Sulfur cleanup (Method 3660). These cleanup procedures are included in SOP # CORP-OP-0001. Use or hexane / acetone as the extraction solvent (rather than hexane / methylene chloride) will reduce the amount of interferences extracted.

5. SAFETY

5.1. Refer to section 5 of the Method 8000B SOP for general safety requirements.

5.2. Aroclors have been classified as a potential carcinogen under OSHA. Concentrated solutions of Aroclors must be handled with extreme care to avoid excess exposure. Contaminated gloves and clothing must be removed immediately. Contaminated skin surfaces must be washed thoroughly.

6. EQUIPMENT AND SUPPLIES

6.1. Refer to Section 6 of the 8000B section of this SOP. A ^{63}Ni electron capture detector is required.

6.2. Refer to Table B-2 for analytical columns.

6.3. Microsyringes, various sizes, for standards preparation, sample injection, and extract dilution.

7. REAGENTS AND STANDARDS

7.1. Refer to the method 8000B section of this SOP for general requirements for reagents and supplies.

7.2. Refer to Table B-3 for details of calibration standards.

7.3. Surrogate Standards

Tetrachloro-m-xylene and decachlorobiphenyl are the surrogate standards. Refer to tables B-5 and B-6 for details of surrogate standards.

7.4. Column Degradation Evaluation Mix

A mid-level standard containing 4,4'-DDT and Endrin and not containing any of their breakdown products must be prepared for evaluation of degradation of these compounds by the GC column and injection port. This mix must be replaced after one year, or whenever corrective action to columns fails to eliminate the breakdown of the compounds, whichever is shorter. This solution also contains the surrogates. Refer to Table B-4 for details of the column degradation evaluation mix.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

Refer to Section 8 of the 8000B section of this SOP.

9. QUALITY CONTROL

Refer to Section 9 of the 8000B section of this SOP.

10. CALIBRATION AND STANDARDIZATION

10.1. Refer to Section 10 of the 8000B section of this SOP for general calibration requirements.

10.2. Refer to Table B-2 for details of GC operating conditions. The conditions listed should result in resolution of all analytes listed in Table B-1 on both columns. Closely eluting pairs are DDE and Dieldrin on the Rtx-5 or DB-5 column and Endosulfan II and DDD on the 1701 column.

10.3. Column Degradation Evaluation

Before any calibration runs, either initial or 12 hour, The column evaluation mix must be injected before each initial or daily calibration. The degradation of DDT and endrin must be calculated (see equations 9 and 10) and each shown to be less than 15% before calibration can proceed. This is only necessary if the target compound list includes DDT, Endrin, or any of their degradation products.

If the breakdown of DDT and/or endrin exceeds the limits given above, corrective action must be taken. This action may include:

- Replacement of the injection port liner or the glass wool.
- Cutting off a portion of the injection end of a capillary column.
- Replacing the GC column.

10.4. Initial Calibration

Refer to Section 10 of the 8000B section of this SOP for details of calibration procedures.

10.4.1. Refer to Table B-8 for the initial calibration analytical sequence.

10.4.2. The response for each single-peak analyte will be calculated by the procedures described in the general method for GC analysis.

10.4.3. The surrogate calibration curve is calculated from the Individual AB mix. Surrogates in the other calibration standards are used only as retention time markers. If there are resolution problems, then the A and B mixes may be analyzed separately.

10.4.4. For multi-component pesticides:

Single point calibration is used for multicomponent pesticides (typically toxaphene and technical chlordane). Two options are possible; the same quantitation option must be used for standards and samples. Refer to section 12.3 for guidance on which option to use.

10.4.5. For multicomponent analytes, the mid level standard must be analyzed as part of the initial calibration. This single point calibration is used to quantitate multicomponent analytes.

10.4.6. The analyst may include a full 5 point calibration for any of the multicomponent analytes with the initial calibration.

10.5. 12 hour Calibration Verification

The 12 hour calibration verification sequence must be analyzed within 12 hours of the start of the initial calibration and at least once every 12 hours thereafter if samples are being analyzed. If more than 12 hours have elapsed since the injection of the last sample in the analytical sequence, a new analytical sequence must be started with a 12 hour calibration. A mid level calibration standard is used for the 12 hour calibration. Refer to the 8000B section of this SOP for acceptance criteria.

10.5.1. At a minimum, the 12 hour calibration includes analysis of the breakdown mix followed by mid level standards of any single and multicomponent analytes.

10.5.2. The retention time windows for any analytes included in the 12 hour calibration are updated.

10.6. Continuing Calibration

The AB calibration mix is analyzed as the continuing calibration standard. At a minimum, this is analyzed after every 20 samples, including matrix spikes, LCS, and method blanks. If 12 hours elapse analyze the 12 hour standard sequence instead. The continuing calibration standard need not include multicomponent analytes. If instrument drift is expected due to sample matrix or other factors, it may be advisable to analyze the continuing calibration standard more frequently.

10.6.1. A mid level calibration standard is used for the continuing calibration.

11. PROCEDURE

11.1. Refer to the method 8000B section of this SOP for general procedural requirements.

11.2. Extraction

The extraction procedure is described in SOP No. CORP-OP-0001.

11.3. Cleanup

Cleanup procedures are described in SOP No. CORP-OP-0001.

11.4. Suggested gas chromatographic conditions are given in Table B-2.

11.5. Allow extracts to warm to ambient temperature before injection.

11.6. The suggested analytical sequence is given in Table B-8.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Refer to the 8000B section of this SOP for identification and quantitation of single component analytes.

12.2. Identification of Multicomponent Analytes

Retention time windows are also used for identification of multi-component analytes, but the "fingerprint" produced by major peaks of those compounds in the standard is used in tandem with the retention times to identify the compounds. The ratios of the areas of the major peaks are also taken into consideration. Identification of these compounds may be made even if the retention times of the peaks in the sample fall outside of the retention time windows of the standard, if in the analyst's judgment the fingerprint (retention time and peak ratios) resembles the standard chromatogram.

12.3. Quantitation of Multicomponent Analytes

Use 3-10 major peaks or total area for quantitation as described in section 10.4.4, initial calibration of multicomponent analytes.

12.3.1. If there are no interfering peaks within the envelope of the multicomponent analyte, the total area of the standards and samples may be used for quantitation. Any surrogate or extraneous peaks within the envelope must be subtracted from the total area.

12.3.1.1. Multiple peak option

This option is particularly valuable if toxaphene is identified but interferences make quantitation based on total area difficult. Select 3-10 major peaks in the analyte pattern. Calculate the response using the total area or total height of these peaks. Alternatively, find the response of each of the 3-10 peaks per multi-peak pesticide, and use these responses independently, averaging the resultant concentrations found in samples for a final concentration result. When using this option, it is appropriate to remove peaks that appear to be coeluting with contaminant peaks from the quantitation. (i.e. peaks which are significantly larger than would be expected from the rest of the pattern.)

Chlordane may be quantitated either using the multiple peak option (12.3.1.1) total area option (12.3.1.2.) or by quantitation of the major components, α-chlordane, γ-chlordane and heptachlor.

12.3.1.2. Total area option

The total area of the standards and samples may be used for quantitation of multicomponent analytes. Any surrogate or extraneous peaks within the envelope must be subtracted from the total area. This option should not be used if there are significant interference peaks within the multicomponent pattern in the samples. The retention time window for total area measurement must contain at least 90% of the area of the analyte.

12.4. Second column confirmation multi-component analytes will only be performed when requested by the client, because the appearance of the multiple peaks in the sample usually serves as a confirmation of analyte presence.

12.5. Surrogate recovery results are calculated and reported for decachlorobiphenyl (DCB) unless it is determined that sample interference has adversely affected the quantitation of that surrogate. Tetrachloro-m-xylene (TCMX) recovery is reported in lieu of DCB in samples having the interference,

and also may be used in samples in which DCB recovery is low. Corrective action is only necessary if DCB and TCMX are both outside of acceptance limits.

12.6. Calculation of Column Degradation/% Breakdown (%B)

$$DDT\ \%B = \frac{A_{DDD} + A_{DDE}}{A_{DDD} + A_{DDE} + A_{DDT}} \times 100$$

where:
A_{DDD}, A_{DDE}, and A_{DDT} = the response of the peaks for 4,4'-DDD, 4,4'-DDE, and 4,4'-DDT in the column degradation evaluation mix.

$$Endrin\ \%B = \frac{A_{EK} + A_{EA}}{A_{EK} + A_{EA} + A_{E}} \times 100$$

where:
A_{EK}, A_{EA}, and A_{E} = the response of endrin ketone, endrin aldehyde, and endrin in the column degradation evaluation mix.

13. METHOD PERFORMANCE

13.1. Performance limits for the four replicate initial demonstration of capability required under Section 13.1 of the main body of this SOP are presented in Table B-7. The spiking level should be equivalent to a mid level calibration.

14. POLLUTION PREVENTION

Refer to section 14 of the 8000B section of this SOP.

15. WASTE MANAGEMENT

15.1. Waste generated in this procedure will be segregated and disposed according to the facility hazardous waste procedures. The Environmental Health and Safety Director should be contacted if additional information is required.

16. REFERENCES

SW846, Update III, December 1996, Method 8081A

17. MISCELLANEOUS

17.1. Modifications from Reference Method

None

17.2. Modifications from Previous Revisions

17.3. Substantial revision were made to reflect the changes to method 8081 in Update III to SW-846.

17.4. Tables

Table B-1
Standard Analyte list and Reporting Limits

Compound	Reporting Limit, µg/L or µg/kg		
	water	soil	waste
Aldrin	0.05	1.7	50
α-BHC	0.05	1.7	50
β-BHC	0.05	1.7	50
δ-BHC	0.05	1.7	50
γ-BHC (Lindane)	0.05	1.7	50
α-Chlordane	0.05	1.7	50
γ-Chlordane	0.05	1.7	50
Chlordane (technical)	0.5	17	500
4,4'-DDD	0.05	1.7	50
4,4'-DDE	0.05	1.7	50
4,4'-DDT	0.05	1.7	50
Dieldrin	0.05	1.7	50
Endosulfan I	0.05	1.7	50
Endosulfan II	0.05	1.7	50
Endosulfan Sulfate	0.05	1.7	50
Endrin	0.05	1.7	50
Endrin Aldehyde	0.05	1.7	50
Heptachlor	0.05	1.7	50
Heptachlor Epoxide	0.05	1.7	50
Methoxychlor	0.1	3.3	100
Toxaphene	2.0	67	2000
APPENDIX IX ADD ONs			
Diallate	1.0	33	1000
Isodrin	0.1	3.3	100
Chlorobenzillate	0.1	3.3	100
Kepone[1]	1.0	33	1000

[1] Kepone is sometimes requested for analysis by method 8081A. However kepone may produce peaks with broad tails that elute later than the standard by up to a minute (presumably due to hemi-acetal formation). As a result kepone analysis by 8081A is unreliable and not recommended. Analysis by method 8270C is a possible alternative. Note: alpha chlordane, gamma chlordane, and endrin ketone are not required for some projects.

The following concentration factors are assumed in calculating the Reporting Limits:

	Extraction Vol.	Final Vol.
Ground water	1000 mL	10 mL
Low-level Soil	30 g	10 mL
High-level soil / waste	1 g	10 mL

Table B-2	
Parameter	**Recommended Conditions**
Injection port temp	220°C
Detector temp	325°C
Temperature program	120°C for 1 min, 8.5°C/min to 285°C, , 6 min hold
Column 1	Rtx-CLPesticides 30m x 0.32mm id, 0.5µm
Column 2	Rtx-35 30m x 0.32 mm id, 0.5µm
Column 3	DB-608, 30m X 0.32 mm, 0.25µm
Injection	2µL
Carrier gas	Helium or Hydrogen
Make up gas	Nitrogen
Y splitter	Restek or J&W or Supelco glass tee

Table B-3						
Calibration Levels ng/mL						
	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6[2]
Individual Mix AB[1]						
Aldrin	5	10	25	50	100	200
g-BHC (Lindane)	5	10	25	50	100	200
Heptachlor	5	10	25	50	100	200
Methoxychlor	10	20	50	100	200	400
Dieldrin	5	10	25	50	100	200
Endosulfan I	5	10	25	50	100	200
Endosulfan II	5	10	25	50	100	200
4,4'-DDT	5	10	25	50	100	200
Endrin Aldehyde	5	10	25	50	100	200
Endrin Ketone	5	10	25	50	100	200
β-BHC	5	10	25	50	100	200
δ-BHC	5	10	25	50	100	200
α-BHC	5	10	25	50	100	200
4,4'-DDD	5	10	25	50	100	200
4,4'-DDE	5	10	25	50	100	200
Endosulfan Sulfate	5	10	25	50	100	200
Endrin	5	10	25	50	100	200
α-Chlordane [3]	5	10	25	50	100	200
γ-Chlordane [3]	5	10	25	50	100	200
Multicomponent Standards						
Chlordane (Technical)			250[4]			
Toxaphene			1000[5]			
Surrogates are included with all the calibration mixes at the following levels:						
Tetrachloro-m-xylene	5	10	25	50	100	200
Decachlorobiphenyl	5	10	25	50	100	200

[1] Standards may be split into an A and B mix if resolution of all compounds on both columns is not obtained.
[2] Level 6 is optional and should only be used if linearity can be maintained on the instrument to this level.
[3] Compounds may be used in lieu of running a daily technical Chlordane standard for samples that are non-detect for technical Chlordane.
[4] This standard may be used for quantitation of technical chlordane between 50 and 1000 ng/mL. If the chlordane is more concentrated, the extract must be diluted and reanalyzed.
[5] This standard may be used for quantitation of toxaphene between 200 and 4000 ng/mL. If the toxaphene is more concentrated, the extract must be diluted and reanalyzed.

Table B-4	
Column Degradation Evaluation Mix ng/mL	
Component	Concentration
4,4'-DDT	25
Endrin	25
Tetrachloro-m-xylene (Surrogate)	20
Decachlorobiphenyl (Surrogate)	20

Table B-5			
LCS/Matrix Spike and Surrogate Spike levels µg/L or µg/kg			
	Aqueous	Soil	Waste
gamma BHC (Lindane)	0.20	6.67	200
Aldrin	0.20	6.67	200
Heptachlor	0.20	6.67	200
Dieldrin	0.50	16.7	500
Endrin	0.50	16.7	500
4,4'DDT	0.50	16.7	500
Tetrachloro-m-xylene (Surrogate)	0.20	6.67	200
Decachlorobiphenyl (Surrogate)	0.20	6.67	200

Table B-6		
LCS/Matrix Spike and Surrogate Spike levels for TCLP µg/L or µg/kg		
	Aqueous	Waste
Heptachlor	5	500
Heptachlor epoxide	5	500
Lindane	5	500
Endrin	5	500
Methoxychlor	10	1000

Table B-7
Suggested Analytical Sequence

Initial Calibration

Solvent blank (optional)	
Breakdown Mix	
Individual mix AB	All levels
Technical Chlordane	Level 3[1]
Toxaphene	Level 3[1]
Solvent blank	
Up to 20 samples unless 12 hours comes first)	
Solvent blank (optional)	
Individual mix AB	Mid level (Continuing calibration)
Samples	
After 12 hours:	
Breakdown mix	
Individual mix AB	
Any other single component analytes	
Any multicomponent analytes	

[1] A five point curve for any of the multicomponent analytes may be included
If Arochlors are included, a 5 point calibration for Arochlor 1016/1260 should be included with the initial calibration and a single point for the other Arochlors. The mid point 1016/1260 mix is included with the daily calibration (every 12 hours).

12 hour Calibration

At least every 12 hours, counting from the start of the initial calibration, or from the start of the last daily calibration, the retention time windows must be updated using the Individual mix AB, and the breakdown mix must be run before the continuing calibration.

Table B-8		
Performance limits, four replicate initial demonstration of capability		
Compound	Initial demonstration, mean recovery limits	Initial demonstration, RSD limits
Aldrin	46-112	21
alpha-BHC	51-122	24
beta-BHC	61-120	32
delta-BHC	49.5-118.5	36
gamma-BHC	57-116	23
Chlordane	44.8-108.6	20
4,4'-DDD	52-126	28
4,4'-DDE	46-120	27.5
4,4'-DDT	54-137	36
Dieldrin	42.5-124.5	38
Endosulfan I	43-141	24.5
Endosulfan II	78-171	61
Endosulfan Sulfate	62-132	27
Endrin	49-126	37
Heptachlor	57-100	20
Heptachlor Epoxide	43.5-131.5	25.4
Toxaphene	44.4-111.2	20

1. SCOPE AND APPLICATION

1.1. This SOP Appendix describes procedures to be used when SW-846 Method 8000B is applied to the analysis of polychlorinated biphenyls (PCB) by GC/ECD. This Appendix is to be applied when SW-846 Method 8082 is requested, and is applicable to extracts derived from any matrix which are prepared according to the appropriate Quanterra sample extraction SOPs. (CORP-OP-0001). The PCBs are determined and quantitated as Arochlor mixes.

Table C-1 lists compounds which are routinely determined by this method and gives the Reporting Limits (RL) for each matrix. RLs given are based on the low level standard and the sample preparation concentration factors. Matrix interferences may result in higher RLs than those listed.

Note: SW-846 method 8082 provides incomplete guidance for determination of individual PCB congeners. This SOP does not include directions for congener specific analysis.

2. SUMMARY OF METHOD

This method presents conditions for the analysis of prepared extracts of PCBs. The PCBs are injected onto the column and separated and detected by electron capture detection. Quantitation is by the external standard method.

3. DEFINITIONS

Refer to the QAMP for definitions of terms used in this document.

4. INTERFERENCES

4.1. Refer to the method 8000B section of this SOP for information regarding chromatographic interferences.

4.2. Interferences in the GC analysis arise from many compounds amenable to gas chromatography that give a measurable response on the electron capture detector. Phthalate esters, which are common plasticizers, can pose a major problem in the determinations. Interferences from phthalates are minimized by avoiding contact with any plastic materials.

4.3. Sulfur will interfere and can be removed using procedures described in SOP CORP-OP-0001.

4.4. Interferences co-extracted from samples will vary considerably from source to source. The presence of interferences may raise quantitation limits for individual samples. Specific cleanups may be performed on the sample extracts, including florisil cleanup (Method 3620), Gel Permeation Chromatography (Method 3640), and Sulfur cleanup (Method 3660). These cleanup procedures are included in SOP # CORP-OP-0001.

5. SAFETY

5.1. Refer to section 5 of the Method 8000B SOP for general safety requirements.

5.2. Aroclors have been classified as a potential carcinogen under OSHA. Concentrated solutions of Aroclors must be handled with extreme care to avoid excess exposure. Contaminated gloves and clothing must be removed immediately. Contaminated skin surfaces must be washed thoroughly.

6. EQUIPMENT AND SUPPLIES

6.1. Refer to Section 6 of the 8000B section of this SOP. A ^{63}Ni electron capture detector is required.

6.2. Refer to Table C-2 for analytical columns.

6.3. Microsyringes, various sizes, for standards preparation, sample injection, and extract dilution.

7. REAGENTS AND STANDARDS

7.1. Refer to the method 8000B section of this SOP for general requirements for reagents and supplies. All standards for this method must be replaced

7.2. Refer to Table C-3 for details of calibration standards.

7.3. Surrogate Standards

Tetrachloro-m-xylene and decachlorobiphenyl are the surrogate standards. Other surrogates may be used at client request. Refer to Table C-4 for details of surrogate standards.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

Refer to Section 8 of the 8000B section of this SOP.

9. QUALITY CONTROL

Refer to Section 9 of the 8000B section of this SOP.

10. CALIBRATION AND STANDARDIZATION

10.1. Refer to Section 10 of the 8000B section of this SOP for general calibration requirements.

10.2. Initial Calibration

10.2.1. Refer to Table C-5 for the initial calibration analytical sequence.

10.2.2. The response for each Arochlor will be calculated by the procedures described in the general method for GC analysis, with the following modifications.

10.2.3. A five point calibration of the Arochlor 1016/1260 mix is generated with at least mid level single points for the other Aroclor mixes. The average response factor is used to quantitate Arochlors 1260 and 1016, other Arochlors are quantitated from the mid level single point.

10.2.4. The analyst may include a full 5 point calibration for any of the Arochlors with the initial calibration.

10.2.5. The high and low standards for the initial 5 point calibration of 1016 / 1260 define the acceptable quantitation range for the other Arochlors. If any Arochlor is determined above this concentration the extract must be diluted and reanalyzed.

10.2.6. If the analyst knows that a specific Arochlor is of interest for a particular project, that Arochlor may be used for the five point calibration rather than the 1016 / 1260 mix.

10.2.7. The surrogate calibration curve is calculated from the Aroclor 1016/1260 mix. Surrogates in the other calibration standards are used only as retention time markers.

10.2.8. Two options are possible for quantitation of Aroclors. The same quantitation option must be used for standards and samples.

10.2.8.1. Multiple peak option

Select 3-10 major peaks in the analyte pattern. Calculate the response using the total area or total height of these peaks. Alternatively, find the response of each of the 3-10 peaks per Aroclor, and use these responses

independently, averaging the resultant concentrations found in samples for a final concentration result. When using this option, it is appropriate to remove peaks that appear to be coeluting with contaminant peaks from the quantitation. (i.e. peaks which are significantly larger than would be expected from the rest of the pattern.)

10.2.8.2. Total area option

The total area of the standards and samples may be used for quantitation of multicomponent analytes. Any surrogate or extraneous peaks within the envelope must be subtracted from the total area. This option should not be used if there are significant interference peaks within the multicomponent pattern in the samples. The retention time window for total area measurement must contain at least 90% of the area of the analyte.

10.3. 12 hour Calibration

The 12 hour calibration verification must be analyzed within 12 hours of the start of the initial calibration and at least once every 12 hours thereafter if samples are being analyzed. If there is a break in the analytical sequence of greater than 12 hours, then a new continuing calibration run must be analyzed before proceeding with the sequence. If more than 12 hours have elapsed since the injection of the last sample in the analytical sequence, a new analytical sequence must be started with a 12 hour calibration.

10.3.1. At a minimum, the 12 hour calibration includes analysis of the Arochlor 1260 / 1016 mix.

10.3.2. It is adequate to verify calibration with a mixture of Aroclors 1016 and 1260. If a specific Aroclor is expected, it should be included in the daily calibration check.

10.3.3. The retention time windows for any analytes included in the daily calibration are updated.

10.3.4. For this method samples must be bracketed with successful calibration verification runs.

10.4. Calibration verification

The Arochlor 1260/1016 calibration mix is analyzed as the calibration verification standard. This is analyzed after every 20 samples, including matrix spikes, LCS, and method blanks. (Depending on the type of samples, it may be advisable to analyze verifications more frequently in order to minimize reruns.).

10.4.1. A mid level standard is used for the calibration verification.

11. PROCEDURE

11.1. Refer to the method 8000B section of this SOP for general procedural requirements.

11.2. Extraction

The extraction procedure is described in SOP No. CORP-OP-0001.

11.3. Cleanup

Cleanup procedures are described in SOP No. CORP-OP-0001.

11.4. Suggested gas chromatographic conditions are given in Table C-2.

11.5. Allow extracts to warm to ambient temperature before injection.

11.6. The suggested analytical sequence is given in Table C-5.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Identification of Arochlors

Retention time windows are used for identification of Arochlors, but the "fingerprint" produced by major peaks of those analytes in the standard is used in tandem with the retention times for identification. The ratios of the areas of the major peaks are also taken into consideration. Identification may be made even if the retention times of the peaks in the sample fall outside of the retention time windows of the standard, if in the analyst's judgment the fingerprint (retention time and peak ratios) resembles the standard chromatogram.

A clearly identifiable Arochlor pattern serves as confirmation of single column GC analysis. However, if the pattern is not clear, or if no historical data for the site is available, then second column confirmation must be performed.

12.2. Quantitation of Arochlors

Use 3-10 major peaks or total area for quantitation

If the analyst believes that a combination of Aroclor 1254 and 1260, or a combination of 1242, 1248 and 1232 is present, then only the predominant Arochlor is quantitated and reported, but the suspicion of multiple Aroclors is discussed in the narrative. If well separated Aroclor patterns are present, then both Aroclors are quantitated and reported.

12.3. If there are no interfering peaks within the envelope of the Arochlor, the total area of the standards and samples may be used for quantitation. Any surrogate or extraneous peaks within the envelope must be subtracted from the total area.

12.4. Second column confirmation of Aroclors will only be performed when requested by the client, or if the pattern is not clear or there is no historical data leading to a suspicion that Arochlors may be present. The appearance of the multiple peaks in the sample usually serves as a confirmation of Aroclor presence.

12.5. Surrogate recovery results are calculated and reported for decachlorobiphenyl (DCB) unless it is determined that sample interference has adversely affected the quantitation of that surrogate. Tetrachloro-m-xylene (TCMX) recovery is reported in lieu of DCB in samples having the interference, and also may be used in samples in which DCB recovery is low. Corrective action is only necessary if DCB and TCMX are both outside of acceptance limits.

13. METHOD PERFORMANCE

13.1. Performance limits for the four replicate initial demonstration of capability required under Section 13.1 of the main body of this SOP are recovery of 70-130%. The spiking level should be equivalent to a mid level calibration.

13.2. Method detection limits (MDL) are determined for Arochlor 1016 and 1260. Arochlor 1016 represents an Arochlor consisting primarily of low chlorinated congeners while Arochlor 1260 represents an Arochlor consisting primarily of high chlorinated congeners. The same reporting limit is applied to all Arochlors, and must be supported by both MDLs.

14. POLLUTION PREVENTION

Refer to section 14 of the 8000B section of this SOP.

15. WASTE MANAGEMENT

15.1. Waste generated in this procedure will be segregated and disposed according to the facility hazardous waste procedures. The Environmental Health and Safety Director should be contacted if additional information is required.

16. REFERENCES

SW846, Update III, December 1996, Method 8082

17. MISCELLANEOUS

17.1. Modifications from Reference Method

17.1.1. Method 8082 includes limited direction for congener specific quantitation. This is outside the scope of this SOP.

17.2. Modifications from Previous Revisions

The 8082 procedure was added to this revision.

17.3.

Tables

Table C-1
Standard Analyte list and Reporting Limits

Compound	Reporting Limit, µg/L or µg/kg		
	water	soil	waste
Aroclor-1016	1.0	33	1000
Aroclor-1221	1.0	33	1000
Aroclor-1232	1.0	33	1000
Aroclor 1242	1.0	33	1000
Aroclor-1248	1.0	33	1000
Aroclor-1254	1.0	33	1000
Aroclor-1260	1.0	33	1000

The following concentration factors are assumed in calculating the Reporting Limits:

	Extraction Vol.	Final Vol.
Ground water	1000 mL	10 mL
Low-level Soil	30 g	10 mL
High-level soil / waste	1 g	10 mL

Table C-2

Parameter	Recommended Conditions
Injection port temp	220°C
Detector temp	325°C
Temperature program	70°C for 0.5min, 30°C/min to 190°C, 2.5°C/min to 225, 18°C/min to 280°C, 3 min hold
Column 1	DB-5 or Rtx-5 30m x 0.32mm id, 0.5µm
Column 2	DB-1701 or Rtx 1701 30m x 0.32 mm id, 0.25µm
Column 3	DB-608, 30m X 0.32 mm, 0.25µm
Injection	1-2µL
Carrier gas	Helium or Hydrogen
Make up gas	Nitrogen
Y splitter	Restek or J&W or Supelco glass tee

Table C-3						
Calibration Levels ng/mL						
	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6[1]
Aroclor 1016/1260	100	200	500	1000	2000	4000
Aroclor 1242[2]			500			
Aroclor 1221 +1254[2]			500			
Aroclor 1232[2]			500			
Aroclor 1248[2]			500			
Surrogates are included with all the calibration mixes at the following levels:						
Tetrachloro-m-xylene	5	10	25	50	100	200
Decachlorobiphenyl	5	10	25	50	100	200

[1] Level 6 is optional and should only be used if linearity can be maintained on the instrument to this level.
[2] Arochlors may be quantitated within the range 100 to 2000 ng/mL (4000ng/mL if the level 6 1016/1260 standard is included). If the Arochlor is more concentrated, it must be reanalyzed at a dilution.

Table C-4			
LCS/Matrix Spike and Surrogate Spike levels for Aroclor analysis with Acid Cleanup µg/L or µg/kg			
	Aqueous	Soil	Waste
Aroclor 1016/1260	10	333	10,000
Tetrachloro-m-xylene (Surrogate)	0.20	6.67	200
Decachlorobiphenyl (Surrogate)	0.20	6.67	200

Table C-5
Suggested Analytical Sequence

Initial Calibration

Injection #		
1	Solvent blank (optional)	
2	Aroclor 1016/1260	Level 1
3	Aroclor 1016/1260	Level 2
4	Aroclor 1016/1260	Level 3
5	Aroclor 1016/1260	Level 4
6	Aroclor 1016/1260	Level 5
7	Aroclor 1232	Level 3
8	Aroclor 1242	Level 3
9	Aroclor 1248	Level 3
10	Aroclor 1221/1254	Level 3
11	Solvent blank	
12-31	Sample 1-20 (or as many samples as can be analyzed in 12 hours	
	Solvent blank (optional)	
32	Aroclor 1016/1260	Level 3

etc

12 hour Calibration

At least every 12 hours, counting from the start of the initial calibration, or from the start of the last daily calibration, the retention time windows must be updated using the Arochlor 1260 / 1016 mix. Mid level standards of any other Arochlors expected to be present in the samples are also injected.

1. SCOPE AND APPLICATION

This method is applicable to the gas chromatographic determination of Chlorinated phenoxy acid herbicides in extracts prepared by SOP CORP-OP-0001. The herbicides listed in Table D1 are routinely analyzed. Other chlorinated acids may be analyzed by this method if the quality control criteria in section 9 and the initial demonstration of method performance in section 13 are met.

2. SUMMARY OF METHOD

This method presents conditions for the analysis of prepared extracts of phenoxy acid herbicides by gas chromatography. The herbicides, as their methyl esters, are injected onto the column, separated, and detected by electron capture detectors. Quantitation is by the external standard method.

3. DEFINITIONS

Refer to the QAMP for definitions of terms used in this document.

4. INTERFERENCES

4.1. Refer to the method 8000B section of this SOP for general information regarding chromatographic interferences.

4.2. Chlorinated acids and phenols cause the most direct interference with this method.

4.3. Sulfur may interfere and may be removed by the procedure described in SOP#CORP-OP-0001.

5. SAFETY

5.1. Refer to section 5 of the Method 8000B SOP for general safety requirements.

6. EQUIPMENT AND SUPPLIES

6.1. Refer to Section 6 of the 8000B section of this SOP. A Ni_{63} electron capture detector is required.

6.2. Refer to Table D2 for analytical columns.

6.3. Microsyringes, various sizes, for standards preparation, sample injection, and extract dilution.

7. REAGENTS AND STANDARDS

7.1. Refer to section 7 of the 8000B section of this SOP for general information on reagents and standards.

7.2. Refer to Table D-3 and D-4 for details of calibration and other standards.

8. SAMPLE PREPARATION, PRESERVATION AND STORAGE

Refer to section 8 of the 8000B section of this SOP.

9. QUALITY CONTROL

9.1. Refer to Section 9 of the 8000B section of this SOP for quality control requirements, including the initial demonstration of capability, definition of a batch, surrogate limits, method blanks, laboratory control samples (LCS), and matrix spikes (MS).

9.2. Refer to Table D-5 for minimum performance criteria for the initial demonstration of capability.

9.3. Refer to Table D-4 for the components and levels of the LCS and MS mixes.

10. CALIBRATION AND STANDARDIZATION

10.1. Refer to Section 10 of the 8000B section of this SOP for general calibration requirements.

10.2. External standard calibration is used for this method.

10.3. A mid level standard is used for the continuing calibration.

10.4. Refer to Table D-2, for details of GC operating conditions.

11. PROCEDURE

11.1. Refer to the method 8000B section of this SOP for procedural requirements.

11.2. Extraction

The extraction procedure is described in SOP #CORP-OP-0001.

11.3. Cleanup

The alkaline hydrolysis and subsequent extraction of the basic solution described in the extraction procedure provides an effective cleanup.

11.4. Analytical Sequence

The analytical sequence starts with an initial calibration of at least five points, or a daily calibration that meets % difference criteria from an existing initial calibration.

11.4.1. The daily calibration must be analyzed at least once every 24 hours when samples are being analyzed. If there is a break in the analytical sequence of greater than 12 hours, then a new continuing calibration run must be analyzed before proceeding with the sequence. If more than 24 hours have elapsed since the injection of the last sample in the analytical sequence, a new analytical sequence must be started with a daily calibration.

11.4.2. The daily calibration consists of mid level standards of all analytes of interest. Retention time windows must be updated with the daily calibration.

11.4.3. After every 12 hours a continuing calibration is analyzed. The continuing calibration consists of mid level standards of all analytes of interest. Retention time windows are updated with continuing calibrations.

11.5. Gas Chromatography

Chromatographic conditions are listed in Table D-2.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Refer to the 8000B section of this SOP for identification and quantitation of single component analytes.

12.2. The herbicides are analyzed as their methyl esters, but reported as the free acid. For this reason it is necessary to correct the results for the molecular weight of the ester versus the free acid. This is achieved through the concentrations of the calibration standards. For example the 20µg/L calibration standard for 2,4-D contains 21.3 µg/L of the methyl ester. No further correction is necessary.

13. METHOD PERFORMANCE

13.1. Multiple laboratory performance data has not been published by the EPA for this method. Table D-5 lists minimum performance standards required by Quanterra for the four replicate initial demonstration or capability (required by Section 13.2 of the 8000B part of this SOP) for this method. The spiking level should be equivalent to a mid level calibration.

14. POLLUTION PREVENTION

This method does not contain any specific modifications that serve to minimize or prevent pollution.

15. WASTE MANAGEMENT

Waste generated in this procedure will be segregated and disposed according to the facility hazardous waste procedures. The Environmental Health and Safety Director should be contacted if additional information is required.

16. REFERENCES

Method 8151A, SW-846, Update III, December 1996

17. MISCELLANEOUS

17.1. Modifications from Reference Method

Refer to the method 8000B section of this SOP for modifications from the reference method.

17.2. Modifications from Previous Revision

Modifications to the calibration directions were made in order to make the method consistent with method 8000B.

17.3. Tables

Table D-1				
Standard Analyte list				
Compound	CAS Number	Reporting Limit, µg/L or µg/kg		
		Aqueous	Soil	Waste
2,4-D	94-75-7	4	80	4000
2,4-DB	94-82-6	4	80	4000
2,4,5-TP (Silvex)	93-72-1	1	20	1000
2,4,5-T	93-76-5	1	20	1000
Dalapon	75-99-0	2	40	2000
Dicamba	1918-00-9	2	40	2000
Dichloroprop	120-36-5	4	80	4000
Dinoseb	88-85-7	0.6	12	600
MCPA	94-74-6	400	8000	400,000
MCPP	93-65-2	400	8000	400,000

The following concentration factors are assumed in calculating the Reporting Limits:

	Extraction Vol.	Final Vol.	Dilution Factor
Ground water	1000 mL	10 mL	20
Low-level Soil without GPC	50 g	10 mL	20
High-level soil / waste	1 g	10 mL	20

Specific reporting limits are highly matrix dependent. The reporting limits listed above are provided for guidance only and may not always be achievable. For special projects, the extracts may be analyzed without any dilution, resulting in reporting limits 20 times lower than those in Table D-1.

Table D-2	
Instrumental Conditions	
PARAMETER	Recommended conditions
Injection port temp	220°C
Detector temp	325°C
Temperature program	80,2/30/170,0/1/180,1
Column 1	DB-5MS or RTX 5 30x0.32, 0.5um
Column 2	DB-1701 or Rtx-1701
Injection	1-2µL
Carrier gas	Helium / Hydrogen
Make up gas	Nitrogen

Recommended conditions should result in resolution of all analytes listed in Table D-1.

Table D-3						
Calibration Standards, free acid equivalent (methyl ester)[1] ng/mL						
Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6[2]
2,4-D	20 (21.3)	40 (42.5)	80 (85.1)	160 (170)	320 (340)	640 (681)
2,4-DB	20 (21.1)	40 (42.2)	80 (84.5)	160 (169)	320 (338)	640 (676)
2,4,5-TP [Silvex]	5 (5.25)	10 (10.5)	20 (21.1)	40 (42.1)	80 (84)	160 (168)
2,4,5-T	5 (5.27)	10 (10.5)	20 (21.1)	40 (42.2)	80 (84.4)	160 (169)
Dalapon	10 (11.0)	20 (22.0)	40 (43.9)	80 (87.8)	160 (176)	320 (351)
Dicamba	10 (10.6)	20 (21.3)	40 (42.5)	80 (85.1)	160 (170)	320 (340)
Dichloroprop	20 (21.2)	40 (42.4)	80 (84.8)	160 (170)	320 (339)	640 (678)
Dinoseb	3 (3.17)	6 (6.35)	12 (12.7)	24 (25.4)	48 (50.8)	96 (102)
MCPA	2,000 (2,140)	4,000 (4,280)	8,000 (8,560)	16,000 (17,100)	32,000 (34000)	64,000 (68,000)
MCPP	2,000 (2,120)	4,000 (4,260)	8,000 (8,520)	16,000 (17,000)	32,000 (34,100)	64,000 (68,200)
2,4-DCAA [Surrogate]	20	40	80	160	320	640

[1]The calibration standard solutions contain the herbicides as their methyl esters at the levels in parentheses above. These levels are equivalent to the free acid levels also listed above. The herbicides are reported as the free acids.

[2] Level 6 is optional

The reporting limits listed in Table D-1 will be achieved with these calibration levels and a 20 fold dilution of the sample extract. Lower reporting limits can be achieved with lesser dilutions of the sample extract.

Table D-4			
LCS/Matrix Spike and Surrogate Spike levels µg/L or µg/kg[1]			
	Aqueous	Soil	Waste
2,4-D	16	800	16000
Silvex	4	200	4000
2,4,5-T	4	200	4000
2,4-DB	16	800	16000
Dalapon	8	400	8000
DCAA (surrogate)	16	800	16000

[1] LCS, MS and SS spikes are as the free acid.

Table D-5		
Performance limits, four replicate initial demonstration of capability		
Compound	Initial demonstration, mean recovery limits	Initial demonstration, RSD limits
2,4-D	50-150	25
2,4-DB	50-150	25
2,4,5-TP (Silvex)	50-150	25
2,4,5-T	50-150	25
Dalapon	50-150	25
Dicamba	50-150	25
Dichloroprop	50-150	25
Dinoseb	25-120	40
MCPA	50-150	25
MCPP	50-150	25

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OPERATION-SPECIFIC STANDARD OPERATING PROCEDURE

TITLE: AMMONIA AS N (LACHAT AUTOANALYZER)

(SUPERSEDES: LM-CAL-1105, REVISION 2.0, MARCH 6, 1990)

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CU-561

1. SCOPE AND APPLICATION

1.1. This method is applicable to the analysis of Ammonia using a LACHAT QUIKCHEM AE autoanalyzer system by EPA Method 350.1.

1.2. CAS Number: 7664-41-7.

1.3. The detection limit is 0.1 mg/L as N.

1.4. This method is applicable to drinking, surface and saline water, domestic and industrial wastes. Non-aqueous samples may also be analyzed if first extracted with deionized water.

1.5. Instrument response is linear to at least 20 mg/L under the conditions stated in the method. However, the instrument is usually only calibrated over the range 0.1 to 5 mg/L in order to improve sensitivity and avoid a large number of calibration points.

1.6. Approximate analytical time is 2 minutes per sample.

2. SUMMARY OF METHOD

2.1. Samples are analyzed on the LACHAT QUIKCHEM AE system. The method is based on the Berthelot reaction: ammonia reacts with alkaline phenol and sodium hypochlorite to form indophenol blue. The blue color is intensified using sodium nitroprusside. The LACHAT measures absorbance at 630 nm. According to Beer's Law, the absorbance of the reaction product is directly proportional to ammonia concentration.

3. DEFINITIONS

3.1. Deionized water

3.1.1. Distilled water pumped through the Millipore system to ensure that it is ammonia-free water.

3.2. Definitions of terms used in this SOP may be found in the glossary of the Quality Assurance Management Plan (QAMP).

4. INTERFERENCES

4.1. Suspended matter may clog the system. Particulates may be removed by manual filtration.

4.2. High concentrations of calcium and magnesium may cause a precipitate to form. To prevent precipitation, use EDTA for river and industrial water and tartrate for sea water.

4.3. Turbidity may cause interferences in the samples. Turbidity is removed by manual filtration.

4.4. Color may cause interference in the samples. Color may be prevented by running the samples through the manifold as all the reagents are pumping through with deionized water. Non colored samples are run through the manifold as all the reagents are pumping through with *sodium hypochlorite*. The results from the colored samples are then subtracted from the non colored samples to obtain the corrected result.

4.4.1. NOTE: Highly colored samples must be diluted prior to analysis.

4.5. Ammonium hydroxide is a frequently used laboratory reagent so care must be taken when handling to prevent contamination of samples, reagents, and standards. Other commonly used cleaning compounds may also contain ammonia and contribute to contamination.

4.6. Since the method uses a heating coil, the carrier and buffer reagents must be degassed with helium. Bubble helium vigorously through solution for one minute to degas reagents.

5. SAFETY

5.1. Procedures shall be carried out in a manner that protects the health and safety of all Quanterra associates.

5.2. Eye protection that satisfied ANSI Z87.1 (as per the Chemical Hygiene Plan), laboratory coat, and chemically resistant gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.2.1. Neoprene, natural rubber, and butyl gloves provide varying degrees of protection against those chemicals listed. Refer to permeation/degradation charts for the actual data.

5.3. The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the Material Safety Data Sheets (MSDS) maintained in the laboratory.

5.3.1. The following materials are known to be **corrosive**: sodium phenolate, sodium hypochlorite, ammonia, sulfuric acid.

5.3.2. Phenol is highly toxic and can be readily absorbed through the skin. Wear gloves when handling. Avoid breathing the fumes by handling phenol in a fume hood. In case of skin contact, flush with water for at least 15 minutes. Notify your project manager or team leader or safety officer of any spills or exposures.

5.3.3. Sodium nitroprusside can release cyanide. Avoid contact with acids.

5.3.4. Exposure to chemicals must be maintained as low as reasonably achievable, therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.3.5. The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operations will permit.

5.3.6. All work must be stopped in the event of a known or potential compromise to the health and safety of a Quanterra associate. The situation must be reported immediately to a laboratory supervisor.

5.3.7. Wear gloves and apron when handling concentrated acid and bases. Transport only in approved carriers. Avoid breathing fumes and vapors; handle in a fume hood. Neutralize and clean up any spills immediately. In case of skin contact, flush affected area with water for at least 15 minutes. Notify your project manager or team leader or safety officer of any spills or exposures.

6. EQUIPMENT AND SUPPLIES

6.1. LACHAT QUIKCHEM AE Automated Flow Injection Ion Analyzer, consisting of:

6.1.1. Automatic sampler

6.1.2. Proportioning pump

6.1.3. Injection Module equipped with microloop

6.1.4. Colorimeter with 630 nm filter and 10 mm flow cell

6.1.5. Reaction module #10-107-06-1-A

6.1.6. Heating coil (60°C)

6.1.7. QuikCalc II Software System

6.1.8. Disposable autosampler vials

6.1.9. Miscellaneous laboratory apparatus and glassware.

7. REAGENTS AND STANDARDS

7.1. Sulfuric acid for Trace Element Analysis

7.1.1. Malinkrodt AR Select Sulfuric acid.

7.2. Sodium Phenolate:

7.2.1. In a hood, weigh and dissolve 83 g crystalline phenol (or 88 mL 88% liquefied phenol) in about 600 mL deionized water. While mixing, cautiously add 32 g sodium hydroxide and allow to dissolve. Cool to room temperature, dilute to 1 liter with deionized water, and mix well.

7.3. Sodium Hypochlorite:

7.3.1. Dilute 250 mL household bleach (5.25% NaOCl) to 500 mL with deionized water.

7.4. EDTA Buffer (5%):

7.4.1. Dissolve 50.0 g disodium ethylenediamine tetraacetate and 5.5 g sodium hydroxide in about 900 mL deionized water. Cool to room temperature, dilute to 1 liter, and mix well. Degas this reagent with helium just before use.

7.5. Sodium Nitroprusside:

7.5.1. Dissolve 3.5 g sodium nitroprusside in 1000 mL deionized water and mix.

7.6. Ammonia Stock Standard, 1000 mg/L as N:

7.6.1. Dry ammonium chloride at 105°C for one hour and cool in a desiccator. Dissolve 3.819 g in deionized water and dilute to 1 liter.

7.7. Intermediate Standard, 100 mg/L as N:

7.7.1. Dilute 10 mL 1000 mg/L Stock Standard to 100 mL with deionized water.

7.8. Working Standards:

7.8.1. Dilute the intermediate standard (10 mg/L) with deionized water as follows:

Std.	Aliquot (mL)	Final Volume (mL)	Conc. (mg/L as N)
A	5.0	100	5.0
B	2.0	100	2.0
C	1.0	100	1.0
D	0.5	100	0.5
F	0.1	100	0.1
G	0	---	0 (Blank)

7.9. DCS Solution: The DCS solution is a 2.0 mg/L mid-level standard.

7.10. A reference standard prepared from an independent source is also run.

7.10.1.1.NOTE: Store all reagents at room temperature.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Samples are to be collected in plastic or glass containers, acidified with sulfuric acid to pH < 2, and refrigerated to 4°C.

8.1.1. If 0.2 mL of sulfuric acid is added to the sample, then 0.2 mL of sulfuric acid must be added to the wash water and standards.

8.2. The holding time for Ammonia is 28 days.

9. QUALITY CONTROL

9.1. One method blank must be extracted with every process batch of similar matrix, not to exceed twenty (20) samples. The method blank is an aliquot of laboratory reagent water processed in the same manner and at the same time as the associated samples. Corrective actions must be documented on a Non-Conformance memo, then implemented when target analytes are detected in the method blank above the reporting limit or when surrogate recoveries are outside control limits. Re-extraction of the blank, other batch QC, and the affected samples are required when the method blank is deemed unacceptable. See Quanterra Inc. QA Policy 003 for specific acceptance criteria.

9.2. A Laboratory Control Sample (LCS) must be extracted with every process batch of similar matrix, not to exceed twenty (20) samples. The LCS is an aliquot of laboratory matrix (e.g. water, Ottawa sand, sodium sulfate, etc.) spiked with analytes of known identity and concentration. The LCS must be processed in the same manner and at the same time as the associated samples. Corrective actions must be documented on a Non-Conformance memo, then implemented when recoveries of any spiked analyte is outside control limits provided on the LIMS or by the client. Re-extraction of the blank, other batch QC and all associated samples are required if the LCS is deemed unacceptable. See Quanterra Inc. QA Policy 003 for specific acceptance criteria.

9.3. A Matrix Spike/Matrix Spike Duplicate (MS/MSD or MS/SD) pair must be extracted with every process batch of similar matrix, not to exceed twenty (20) samples. An MS/MSD pair are aliquots of a selected field sample spiked with analytes of known identity and concentration. The MS/MSD pair must be processed in the same manner and at the same time as the associated samples. Spiked analytes with recoveries or precision outside control limits must be within control limits in the LCS. Corrective actions must be documented on a Non-Conformance memo, then implemented when recoveries of any spike analyte is outside control limits provided on the LIMS or by the client. Re-extraction of the blank, an LCS, the selected field sample, the MS/MSD may be required after evaluation and review.

9.4. A duplicate control sample (LCSD or DCS) must be substituted when insufficient sample volume is provided to process an MS/MSD pair. The LCSD is evaluated in the same manner as the LCS. See Quanterra Inc. QA Policy 003 for specific acceptance criteria.

9.5. QC Samples

9.5.1. Aqueous samples require a reagent blank. For soil extractions, a prep blank is required with every batch of 20 or less samples.

9.5.2. Two DCS samples are required per 20 samples.

9.5.3. Duplicates may be required for project specific QC.

9.5.4. Matrix Spike and Matrix Spike Duplicate are prepared at a frequency of an MS/MSD pair per twenty samples as required by the California DOHS. Client requested QC may be used to satisfy this requirement.

9.6. Blanks must be less than the detection limit.

9.7. DCS/SCS recovery and DCS relative percent difference (RPD) must be within control limits. Current limits are in LIMS.

10. CALIBRATION AND STANDARDIZATION

10.1. Standard checks must be within 10% of the expected value.

10.2. The calibration curve must have a correlation coefficient of 0.995 or greater and must consist of a minimum of 5 standards in addition to the blank.

10.3. A reagent blank and an Initial Calibration Verification Standard (ICV) must be analyzed following initial calibration and prior to sample analysis.

10.4. A reagent blank and a Continuing Calibration Standard (CCV) are to be placed on the sampler after every 10 or less samples and also at the end of all samples. The CCV standard may be made from the same source as the initial calibration standards.

11. PROCEDURE

11.1. One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and is approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

11.2. Any unauthorized deviations from this procedure must also be documents as a nonconformance, with a cause and corrective action described.

11.3. Sample Preparation

11.3.1. Soils must be taken through the D.I. Leach procedure before analysis. See SOP # LM-CAL-1012.

11.3.2. Check the pH of all samples to ensure that they have been properly preserved. If necessary, add sulfuric acid to produce a pH of less than 2.

11.4. Instrument Start-up

11.4.1. Set up and operate the instrument in accordance to manufacturer's instructions. Install the Ammonia manifold according to manufacturer's instructions. Use deionized water as the carrier.

11.4.2. Inspect manifold for proper connections.

11.4.3. Prepare working standards. Refer to section 7.

11.4.4. Turn on the power to the system unit, pump, autosampler, and the software system. Allow the heating coil to warm up to 60°C. The control switch is located on the back of the system unit under the back cover.

11.4.5. Place reagent lines into proper containers. Fasten down the pump tube cassettes and tighten the tension one notch. Make sure the reagent tubes are in acceptable condition.

11.4.6. Pump reagents through all lines until all air bubbles have been pumped out.

11.4.7. Load the AMMONIA method onto the computer. The Ammonia background will appear on the slaved computer screen.

11.4.8. If the flow system is working properly, then the average baselines should be stable in approximately two minutes. No adjustment for the baseline locations or peak heights is necessary. The baseline locations and peak heights are already set in the software based on the highest standard solution. Depending on the age of the color reagent, peak heights might vary slightly.

11.5. Analysis

11.5.1. Fill the culture tubes with the working standards and place in the autosampler positions indicated by the letters corresponding to each standard in decreasing concentrations.

11.5.2. Place the ICV (DCS) in position 1 of the sampler, followed by the ICB and reference standard in positions 2 and 3 respectively. Additional blanks and DCS are to be analyzed at the beginning of every set of 20 or less samples. Reagent blanks and CCV are to be analyzed every 10 samples or less and at the end of all samples. If applicable, prep blank and DCS are run and counted as regular samples.

11.5.3. Load the autosampler with all samples, recording the location of each sample number, blank, DCS, etc. on the bench sheet. While the sampler is loading and is preparing to run, enter the recorded information into the computer. Be sure to record and enter any dilution or prep factors.

11.5.4. Start the analysis.

11.5.5. The instrument will analyze the standards and proceed with the samples if the correlation coefficient > 0.995. If the calibration curve is not acceptable, locate and correct the problem and then re-calibrate. The instrument should be monitored periodically to make sure that standard checks are in control and no other problems arise. The calibration statistics and raw data prints out at the end of the run.

11.5.6. Check the printout for off scale samples and rerun if necessary.

11.5.7. If additional samples need to be analyzed, reload the autosampler and enter the new tray information. Since the instrument is already calibrated, continue direct analysis of the new samples.

11.6. Instrument Shut-Down

11.6.1. Place all reagent lines in deionized water and pump for five minutes. Then remove the lines from the water and pump the system dry.

11.6.2. Drain the water out of the wash vessel.

11.6.3. Turn off the units and unfasten the pump tube cassettes from the pump. Release the tension levers.

11.6.4. Clean up the work area and replace any reagents which have been depleted.

12. METHOD PERFORMANCE

12.1. The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required expertise.

13. POLLUTION PREVENTION

13.1. No solvents of any kind or in any amount are to be disposed of in the sinks or evaporated in the hoods.

14. WASTE MANAGEMENT

14.1. Waste management in the procedure must be segregated and disposed according to the facility hazardous waste procedures. The Environmental Health and Safety Coordinator or Hazardous Materials Technician should be contacted if additional information is required.

14.2. All waste must be disposed of according to the facility hazardous waste management procedures, Attachment C of the Chemical Hygiene Plan, Section WS002, Table 1.

14.3. Samples and other solutions containing high concentrations of toxic materials must be disposed of according to the facility hazardous waste management procedures, Attachment C of the Chemical Hygiene Plan, Section WS003, Disposal of Samples After Analysis.

15. REFERENCES

15.1. Method source: Lachat Method 10-107-06-1-A (equivalent to EPA Method 350.1)

16. MISCELLANEOUS

16.1. Deviations from reference method.

16.1.1. The range was modified from 0.1-20 mg/L to 0.1-5.0 mg/L.

16.2. Summary of modifications to SOP from previous revisions.

16.2.1. This SOP was modified to be consistent with the Lachat AE system.

16.3. List of other SOPs cross-referenced in SOP.

16.3.1. SAC-QA-0041 Calibration and Calibration Check of Balances, Revision 1.1.

16.3.2. QA Policy 003 Quanterra Quality Control Program, Revision 1.

16.3.3. QA Policy 008 Data Recording Requirements, Rev. 1.

16.3.4. Chemical Hygiene Plan, Attachment C, Hazardous Waste Management Procedures, Revision 0.

16.3.5. LP-CAL-0001 Laboratory Documentation Practices, Revision 3.0.

16.3.6. LM-CAL-1012 Deionized Water Leach Extraction for Wet Chemistry Analyses, Revision Original.

16.4. Facility-specific information required to implement SOP.

16.5. Tables or figures referenced in body of SOP.

16.6. Method appendices.

OPERATION-SPECIFIC STANDARD OPERATING PROCEDURE

TITLE: DETERMINATION OF NITRATE/NITRITE BY AUTOMATED COLORIMETRY-CADMIUM REDUCTION METHOD (MCAWW 353.2, SM4500-NO3 F)

(SUPERSEDES: LM-CAL-1036, REV. 2)

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1. SCOPE AND APPLICATION

1.1. This method determines nitrate and/or nitrite in surface and saline waters, domestic and industrial wastes. This SOP is based on EPA MCAWW 353.2 and Standard Method 4500-NO3 F.

1.2. Non-aqueous samples can be analyzed by this method following extraction using deionized water. See the SOP "Deionized Water Leaching Procedure for Non-aqueous Samples".

1.3. Nitrite is determined concurrently with nitrate + nitrite by the use of a separate manifold configured without the cadmium reduction column. Nitrate is calculated by subtracting the nitrite concentration from the combined nitrate + nitrite.

1.4. The analytical range of this method is 0.025 to 1.0 mg/L for nitrite and 0.05 to 2.0 mg/L for combined nitrate + nitrite. Samples that are over the linear range must be diluted and reanalyzed.

1.5. The reporting limit for each analyte is 0.05 mg/L as N.

1.6. This method is restricted to use by, or under the supervision of, analysts experience in the operation of Lachat Auto-Analyzer and the interpretation of its results.

2. SUMMARY OF METHOD

2.1. Samples are analyzed on the Lachat QuikChem AE, an automated colorimetric system configured with a cadmium reduction column. A filtered (optional) sample is passed through a column comprised of granulated copperized cadmium to reduce nitrate to nitrite. The reduced nitrate and any nitrite originally present in the sample are diazotized with the sulfanilamide, then coupled with N-(1-naphthyl)-ethylenediamine dihydrochloride to form a highly colored azo dye that is measured colorimetrically at 520 nm. The absorbance of this dye is directly proportional to the concentration of nitrate + nitrite as N.

3. DEFINITIONS

3.1. Definitions of terms used in this SOP may be found in the glossary of the Quality Assurance Management Plan (QAMP).

4. INTERFERENCES

4.1. Suspended matter builds up in the column and restricts samples flow. Filter the sample using 0.45 um filters to remove particulates.

4.2. Concentrations of iron, copper, or other metals above several milligrams per liter lower the reduction efficiency thus causing low results. EDTA is added to samples to eliminate this interference.

4.3. Oil and grease will coat the cadmium surface causing low results. This interference can be removed from samples by pre-extraction with organic solvent such as chloroform, methylene chloride, or another suitable solvent.

4.4. Samples preserved with mercuric chloride or sodium thiosulfate will degrade the reduction column and should not be analyzed by this method.

4.5. Physical interferences such as color and turbidity in the samples will cause high results and can be minimized by filtration or dilution.

4.6. Samples may contain interferences that cause the blue-colored azo-complex to dissipate upon extended exposure to air. It is recommended that the sample be loaded onto the autosampler near or just before injection to minimize exposure to air.

5. SAFETY

5.1. Procedures shall be carried out in a manner that protects the health and safety of all Quanterra associates.

5.2. Eye protection that satisfied ANSI Z87.1 (as per the Chemical Hygiene Plan), laboratory coat, and chemically resistant gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.2.1. Neoprene, natural rubber, and butyl gloves provide varying degrees of protection against those chemicals listed. Refer to permeation/degradation charts for the actual data.

5.3. The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the Material Safety Data Sheets (MSDS) maintained in the laboratory.

5.3.1. Chemicals that have been classified as **carcinogens**, or **potential carcinogens**, under OSHA include: cadmium.

5.3.2. The following materials are known to be **corrosive**: sodium hydroxide, hydrochloric acid, copper sulfate, and sulfanilamide reagent.

5.3.3. The following material is known to be **flammable**: acetone.

5.4. Exposure to chemicals must be maintained **as low as reasonably achievable**, therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.5. The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operation will permit.

5.6. All work must be stopped in the event of a known or potential compromise to the health and safety of a Quanterra associate. The situation must be reported **immediately** to a laboratory supervisor.

6. EQUIPMENT AND SUPPLIES

6.1. Lachat QuikChem AE Automated Flow Injection Ion Analyzer, consisting of:

6.1.1. Automatic sampler

6.1.2. Proportioning pump

6.1.3. Injection Module equipped with microloop

6.1.4. Colorimeter with 520 nm filter and 10 mm flow cell

6.1.5. Reaction module #10-107-04-1-C with Cd column

6.1.6. QuikChem II Software System

6.2. Disposable autosampler vials or culture tubes, 12 x 75 mm for samples and standards.

6.3. Acrodisk filters, 0.45 micron.

6.4. Miscellaneous laboratory apparatus and glassware.

7. REAGENTS AND STANDARDS

7.1. Sodium Hydroxide, 15 N: Add 150 g of sodium hydroxide pellets into a 250 mL volumetric flask containing at least 200 mL of deionized water and mix until dissolved. Cool to room temperature, bring to final volume of 250 mL. Store in a plastic bottle, cap tightly. This solution is stable for 1 year.

CAUTION: This mixture will get extremely hot and give off irritating fumes. Prepare in a hood.

7.2. Ammonium Chloride-EDTA Solution (Buffer solution): Dissolve 85.0 g ammonium chloride and 1.0 g disodium ethylenediamine tetraacetic acid dihydrate in about 800 mL deionized water in a 1 liter volumetric flask. Adjust the pH up to 8.5 by adding 15 N sodium hydroxide solution dropwise. Use pH meter and dilute to final volume of 1 liter with deionized water. This solution is stable up to one year.

7.3. Sulfanilamide Reagent: In a 1 liter flask containing at least 600 mL of deionized water, carefully add 100 mL of 85% phosphoric acid (H_3PO_4) and mix gently. Add 40.0 g sulfanilamide and 1.0 g N-1-naphylethylenediamine dihydrochloride (NED) and mix until dissolved. Bring the final volume to 1 liter. This solution is light sensitive, store this solution in a dark bottle. This solution is stable for at least one month. Discard if severe discoloration occurs prior to the one month shelf life.

7.4. Cadmium-Copper Reduction Column Preparation:

7.4.1. Hydrochloric Acid, 1 M: Carefully add 8 mL of concentrated hydrochloric acid (HCl) into a 100 mL volumetric flask or 100 mL graduated cylinder containing at least 90 mL of deionized water. Cap flask or cover the graduated cylinder with parafilm and mix solution gently. This solution is stable for at least 1 year.

7.4.2. Copper Sulfate, 2%: Dissolve 20 g $CuSO_4 \bullet 5\ H_2O$ in deionized water and dilute to 1000 mL.

7.4.3. Cadmium Preparation:

7.4.3.1. Place 10 to 20 g of cadmium granules (0.3 - 1.5 mm mesh diameter) into a 250 mL Erlenmeyer flask.

7.4.3.2. Wash with two 50 mL portions of acetone, then rinse with 50 mL deionized water for at least 3 times, followed by two 50 mL portions of 1 M hydrochloric acid.

7.4.3.3. Rinse several times with deionized water. Dispose of all washings and rinses in a suitable waste container.

7.4.4. Copperization of the cadmium

7.4.4.1. Pour about 50 mL 2% copper sulfate into the flask containing the prepared cadmium granules from 7.4.3.3 and swirl for 5 minutes.

7.4.4.2. Decant the liquid and repeat with another 50 mL portion of 2% copper sulfate. Continue this process until the blue color persists in the liquid.

7.4.4.3. Decant and wash with at least 5 portions of Ammonium Chloride-EDTA Solution (See Section 7.2). At this point, the cadmium granules should be black or dark gray. The copperized cadmium granules may be stored in a tightly capped bottle under Ammonium Chloride-EDTA Solution. Do not expose them to air. **Waste containing copper sulfate should not be poured down the drain, but placed in a suitable waste container for proper disposal.**

7.4.5. Packing Column: **Wear gloves!** Be careful not to chip or break the glass threads on the column.

7.4.5.1. Clamp the empty column in a upright position and unscrew the upper fitting.

7.4.5.2. Place a foam plug in the bottom end of the column. Fasten the fitting attached to the bottom end of the column with a disposable syringe and fill the column, tubing, and fittings with Ammonium Chloride-EDTA buffer solution.

7.4.5.3. Scoop the copperized cadmium granules with a spatula into the top end of the column so that they settle to the bottom of the column. Continue filling with cadmium granules, tapping the column with a

screwdriver handle to dislodge any air bubbles and prevent any gaps in the column.

7.4.5.4. When the column is filled to within about 2 mm of the top, cap the upper end of the column with the foam plug and reattach the fitting. Connect the two ends of the column to prevent air from getting inside.

7.4.5.5. Rinse the outside of the column with deionized water. If air remains in the column or is introduced accidentally, push the air out by using the disposable syringe filled with buffer solution and tapping the column gently with the screwdriver handle until all air bubbles are dislodged.

WARNING: Air bubbles interfere with sample flow. Do not let any air bubbles get into the reduction column. Cadmium granules should be kept in buffer solution at all times. The column must be taken off line through the blue valve while buffer is flowing through it.

7.5. Nitrate Stock Standard, 1000 mg/L as N: Dissolve 7.218 g KNO_3 in deionized water and dilute to 1 liter in a volumetric flask. This solution is stable for 6 months.

7.6. Nitrite Stock Standard, 1000 mg/L as N: Dissolve 4.925 g $NaNO_2$ in deionized water and dilute to 1 liter in a volumetric flask. This solution is stable for 6 months.

7.7. Nitrate + Nitrite Intermediate Standard, 10 mg/L NO_2/NO_3: Dilute 1.0 mL of each of the 1000 mg/L Stock Standards in Sections 7.5 and 7.6 to 100 mL with deionized water. Prepare fresh monthly.

7.8. Stock reference standards for Nitrate, Nitrite, Nitrate + Nitrite (1000 mg/L): Prepare as in sections 7.5, 7.6, 7.7 but use neat material from a second source (vendor or lot number). An ERA Nutrients reference standard or other premade standard can also be use as a second source verification standard.

7.9. Working Standards: Dilute the two intermediate standards (10 mg/L of NO_2+NO_3) with DI water into each volumetric flask as follows:

Std	Aliquot (mL)	Final Volume (mL)	Conc. (mg/L as N)*
A	10	100	1.0
B	5	100	0.5

C	2.5	100	0.25
D	1	100	0.10
E	0.5	100	0.05
F	0.25	100	0.025
G	0	---	BLANK

7.9.1. Working standards must be prepared monthly.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Samples are collected in plastic bottles and stored refrigerated at 4°C ± 2.

8.2. For nitrate <u>or</u> nitrite determination only, samples are unpreserved and must be analyzed within 2 days (48 hours) from the collection date.

8.3. For determination of nitrate + nitrite combined, samples are preserved with 2 mL of concentrated H_2SO_4 per liter of sample to pH $\leq$2. When preserved, the holding time is extended to 28 days from the collection date.

8.4. For leachates and extracts, analysis must be performed within 48 hours from completion of the extraction/preparation, including filtration.

9. QUALITY CONTROL

9.1. One method blank must be extracted with every process batch of similar matrix, not to exceed twenty (20) samples. The method blank is an aliquot of laboratory reagent water processed in the same manner and at the same time as the associated samples. Corrective actions must be documented on a Non-Conformance memo, then implemented when target analytes are detected in the method blank above the reporting limit. Re-extraction of the blank, other batch QC, and the affected samples are required when the method blank is deemed unacceptable. See Quanterra Inc. QA Policy 003 for specific acceptance criteria.

9.1.1. When no independant leaching or extraction procedure is performed, the ICB/CCB will be used and evaluated as the method blank.

9.2. A Laboratory Control Sample (LCS) must be extracted with every process batch of similar matrix, not to exceed twenty (20) samples. The LCS is an aliquot of laboratory matrix (e.g. water, Ottawa sand) spiked with analytes of known identity and concentration. The LCS must be processed in the same manner and at the

same time as the associated samples. Corrective actions must be documented on a Non-Conformance memo, then implemented when recoveries of any spiked analyte is outside control limits provided on the LIMS or by the client. Reextraction and/or reanalysis of the blank, other batch QC and all associated samples are required if the LCS is deemed unacceptable. See Quanterra Inc. QA Policy 003 for specific acceptance criteria.

9.2.1. When no independant leaching or extraction procedure is performed, the ICV/CCV will be used, evaluated, and reported as the LCS. ICV/CCV control limits will apply (i.e. ±10% from the true value) and corrective actions will be applied to affected samples, i.e. the associated and bracketed samples reanalyzed.

9.3. When solid samples are assayed by this procedure, a duplicate aliquot of a solid sample will be leached as part of the process batch, not to exceed 20 samples. The leachates will be the be spiked and then processed as MS/MSDs. The pair must be processed in the same manner and at the same time as the associated samples. Spiked analytes with recoveries or precision outside control limits must be within control limits in the LCS. Corrective actions must be documented on a Non-Conformance memo, then implemented when recoveries of any spike analyte is outside control limits provided on the LIMS or by the client. Re-extraction of the blank, an LCS, the selected field sample, the MS/MSD may be required after evaluation and review.

9.4. A duplicate control sample (LCSD or DCS) must be substituted when insufficient sample volume or matrix prevents processing of an MS/MSD pair. The LCSD is evaluated in the same manner as the LCS. See Quanterra Inc. QA Policy 003 for specific acceptance criteria.

9.4.1. When no independant extraction or leaching is performed, consecutive CCVs will be used, evaluated, and reported as the LCS/LCSD pair. The ICV/CCV acceptance criteria and corrective actions will be applied, i.e. ±10% from the true value, reanalyze associated and bracketed samples.

10. CALIBRATION AND STANDARDIZATION

10.1. The instrument maximization is part of the instrument set-up and pre-programmed internally.

10.2. Instrument calibration is done daily before any samples are analyzed. The calibration curve consists of five (5) standards and a blank. The correlation coefficient of the curve must be at least 0.995 or greater.

10.3. An Initial Calibration Verification Standard (ICV) and a reagent blank (ICB) must be analyzed immediately following initial calibration and prior to sample analysis.

10.3.1. The ICV/CCV standard concentration is the mid-point concentration of the calibration curve and must be within 10% of the expected value. If not, correct the problem and reanalyze any affected samples back to the last CCV. Perform a new multipoint curve if the ICV fails upon reanalysis.

10.3.2. The ICB/CCB value must be less than the reporting limit.

10.4. A Continuing Calibration Verification (CCV) and reagent blank (CCB) are to be placed on the autosampler after every ten or less samples and at the end of the run.

10.5. Corrective actions and suggestions for repeated failures of calibration verifications and calibration blanks.

10.5.1. Check for contamination in the reagents and standards.

10.5.2. Be sure all reagents were made correctly and have not exceeded their expiration dates.

10.5.3. Check system for obvious problems such as plugs, leaks, and pump tubes.

10.5.4. Identify samples may deactivate the reduction column. Samples may need to be diluted and reprepared. Consult with the Area Leader before continuing.

10.5.5. Analyze a 2 mg/L Nitrite-N standard and compare to a 2 mg/L Nitrate-N standard. If the results differ by more than 15%, the column has deteriorated and needs to be repacked.

10.5.6. If you are unable to locate and solve the problem, consult your supervisor for further corrective actions.

11. PROCEDURE

11.1. One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and approved by a Technical Specialist and QA Manager. If contractually required,

the client shall be notified. The Nonconformance Memo shall be filed in the project file.

11.2. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

11.3. This SOP was written explicitly for the Lachat QuikChem AE system and must be followed in order to work with the existing programs in the instrument. Any deviations from this SOP may necessitate modifications to the program. Also, any changes in the program may require alterations in this SOP.

11.4. Sample Preparation

11.4.1. Soils are leached following the SOP "Deionized Water Leaching Procedure for Non-Aqueous Samples".

11.5. Instrument Start-up

11.5.1. The instrument is to be set up and operated in accordance to the manufacturer's instructions. Install the nitrite and nitrate+nitrite manifolds according to manufacturer's instructions. Nitrite may be analyzed alone if the reduction column is bypassed. The carrier will be deionized water for unpreserved samples.

11.5.2. Inspect manifold for proper connections. Make sure that there is sufficient volume for all reagents for the entire run. All reagents and standards must be within the expiration dates.

11.5.3. Prepare working standards as stated in Section 7.

11.5.4. Turn on the power to the system unit, pump, autosampler, and the software system. The heating coil is not needed and must be turned off.

11.5.5. Place all lines into an Erlenmeyer flask containing deionized water for rinsing purposes only (approximately 5 min.). This step is recommended to ensure that the system does not retain any contaminants prior to initiation of the analysis.

11.5.6. Place reagent lines into proper containers. Fasten down the pump tube cassettes and tighten the tension one notch. Make sure the reagent tubes are in good working condition.

11.5.7. Pump reagents through all lines until all air bubbles have been pumped out.

11.5.8. Bring the cadmium column on line by turning on the blue valve on the NO_2+NO_3 manifold. Make sure there are no air bubbles inside the flow system.

11.5.9. Load the NITRATE+NITRITE method in the computer. You will see the NO_2+NO_3 and NO_2 backgrounds on the computer screen.

11.5.10. If the flow system is working properly, the average baselines should be stable in about two minutes. No adjustment for the baseline locations or peak heights is necessary. The baseline locations and peak heights are already set in the software based on the highest standard solution. Depending on the age of the color reagent, peak heights can be slightly different from previous runs.

11.6. Analysis

11.6.1. Check the pH of samples by using pH paper. If the sample pH is below 5 or above 9, adjust to between 5 and 9 with either conc. H_2SO_4 or conc. NaOH. Document the pH adjustment on the associated sample paperwork.

NOTE: If pH adjustment is not performed, then standards must be matrix-matched to the acid-preserved NO3 + NO2 samples. Adjust standards with dropwise additions of conc. H_2SO_4 to pH 2.

11.6.2. Fill the sample tubes with the working standards in **decreasing concentration** and place in the autosampler positions indicated by the letters corresponding to each standard. (Example: spot A should have the highest standard).

11.6.3. Place the ICV (LCS) in position 1 of the sampler, followed by the ICB and reference standard in positions 2 and 3 respectively. Additional blanks and LCSs are to be analyzed at the beginning of every 20 or less samples. If applicable, prep blanks and additional LCSs are run and counted as regular samples.

11.6.4. Load the autosampler with the samples, recording the location of each sample number, blank, LCS, etc., in the instrument run logbook. When the sampler is loaded and ready to run, enter this information into the computer. Be sure to record and enter any dilution or prep factors.

11.6.5. Start the analysis. The instrument will analyze the standards and proceed with the samples if the correlation coefficient > 0.995. If the calibration curve is not acceptable, locate and correct the problem and then recalibrate. The instrument should be monitored periodically to make sure that standard checks are in control and no other problems arise. The calibration statistics and raw data should be printed at the end of the run.

11.6.6. Check the printout for off scale samples and rerun if needed.

11.6.7. If additional samples are to be analyzed, reload the autosampler and enter the new tray information. Since the instrument is already calibrated, you may proceed directly to analysis of the new samples using a different batch identification.

11.7. Instrument Shut-Down

11.7.1. Take the cadmium column off line by turning the blue valve off. If the column is still in acceptable condition, you may leave it connected on the manifold for the next use.

11.7.2. Place all reagent lines in deionized water and pump at normal speed for at least 5 minutes. Then remove the lines from the water and pump the system dry.

11.7.3. Drain the water out of the wash vessel.

11.7.4. Turn off the units and unfasten the pump tube cassettes from the pump. Release the tension levers.

11.7.5. Clean up the work area and replace any reagents which have been depleted.

11.8. Calculations:

11.8.1. Nitrate is calculated manually by subtracting the Nitrite value from the Nitrate + Nitrite value.

11.8.2. Nitrocellulose (NC) calculation (see appropriate SOP):

$$\text{Conc. In sample extract (mg N / L)} = \frac{\text{Instrument Response} - \text{Y Intercept}}{\text{Slope}}$$

11.8.2.1.The final result is calculated by the following equation:

$$\text{Final Results (mg NC/g)} = A \times B \times C$$

Where:

A = Concentration in sample extract (mg N/L)

Conversion from mg/L to µg/L : mg/L × 1000 µg/mg = 1000 µg/L

B = Concentration Factor

$$= \frac{\text{Volume of Extract}}{\text{Weight of Sample taken for Analysis (g)}} = \frac{0.1\ \text{L}}{0.5\ \text{g}} = 0.2\ \text{L / g}$$

C = Conversion Factor

$$= \frac{\text{ug NC/ g}}{\text{ug N/ g}} = \frac{1}{0.1325} = 7.547$$

Note: Conversion Factor is based on the assumption that Nitrocellulose (NC) is 13.25% N.

11.8.3. For % recovery and %RPD calculations, see Policy QA-003.

12. METHOD PERFORMANCE

12.1. The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required expertise.

13. POLLUTION PREVENTION

13.1. No solvents of any kind or in any amount are to be disposed of in the sinks or evaporated in the hoods.

14. WASTE MANAGEMENT

14.1. Waste management in the procedure must be segregated and disposed according to the facility hazardous waste procedures. The Environmental Health and Safety Coordinator or Hazardous Materials Technician should be contacted if additional information is required.

14.2. All waste must be disposed of according to the facility hazardous waste management procedures, Attachment C of the Chemical Hygiene Plan, Section WS002, Table 1.

14.3. Samples and other solutions containing high concentrations of toxic materials must be disposed of according to the facility hazardous waste management procedures, Attachment C of the Chemical Hygiene Plan, Section WS003, Disposal of Samples After Analysis.

15. REFERENCES

15.1. EPA MCAWW 353.2 Nitrogen, Nitrate-Nitrite (Colorimetric, Automated, Cadmium Reduction).

15.2. Standard Method 4500-NO3 F. Automated Cadmium Reduction Method, 18th Edition 1992.

15.3. Method source: Lachat Method 10-107-04-1-C equivalent to EPA Method 353.2

15.4. The Determination of Nitrocellulose in water by Colorimetric Autoanalyzer. DataChem Laboratories, Version 1, January 15, 1991.

16. MISCELLANEOUS (TABLES, APPENDICES, ETC.)

16.1. Deviations from the referenced methods.

16.1.1. Instrumentation : Lachat Quickem AE instead of Technicon Autoanalyzer. Both instruments are compatible and designed for this method.

16.1.2. Linear range is designed based on the manufacturer's recommendation of 0.05 to 2.0 mg/L instead of 0.05 to 10.0 mg/L.

16.1.3. Absorbance is 520 nm instead of 540 nm.

16.1.4. Sodium Hydroxide is used in place of Ammonium Hydroxide for pH adjustment.

16.1.5. Sulfuric Acid is used in place of HCl for pH adjustment.

16.1.6. Reagent preparation are performed according to the manufacturer's instructions and may differ from the method.

16.1.7. Shelf life for standard curve of daily is changed to monthly and was based from the in-house stability study performed for these analytes.

16.1.8. Preservation with Chloroform is omitted. Shelf life on stock standards lasts for at least 1 year without preserving with chloroform.

16.1.9. From the reference material pertaining analysis of Nitrocellulose, the standard preparation, calibration and criteria is not clearly defined and therefore the analytical protocols for Nitrate are adopted for this analyte. Dedicated section for the conversion of Nitrate as N to Nitrocellulose (NC) is included in this SOP.

16.2. Summary of modifications to SOP from previous revisions.

16.2.1. The SOP was modified to be consistent with the use of the LACHAT AE Autoanalyzer.

16.2.2. The new QA-003 policy is incorporated.

16.2.3. Quanterra SOP format adopted.

16.3. List of other SOPs cross-referenced in SOP.

16.3.1. LP-CAL-0001 Laboratory Documentation Practices, Revision 3.0.

16.3.2. QA Policy 003 Quanterra Quality Control Program, Revision 1.

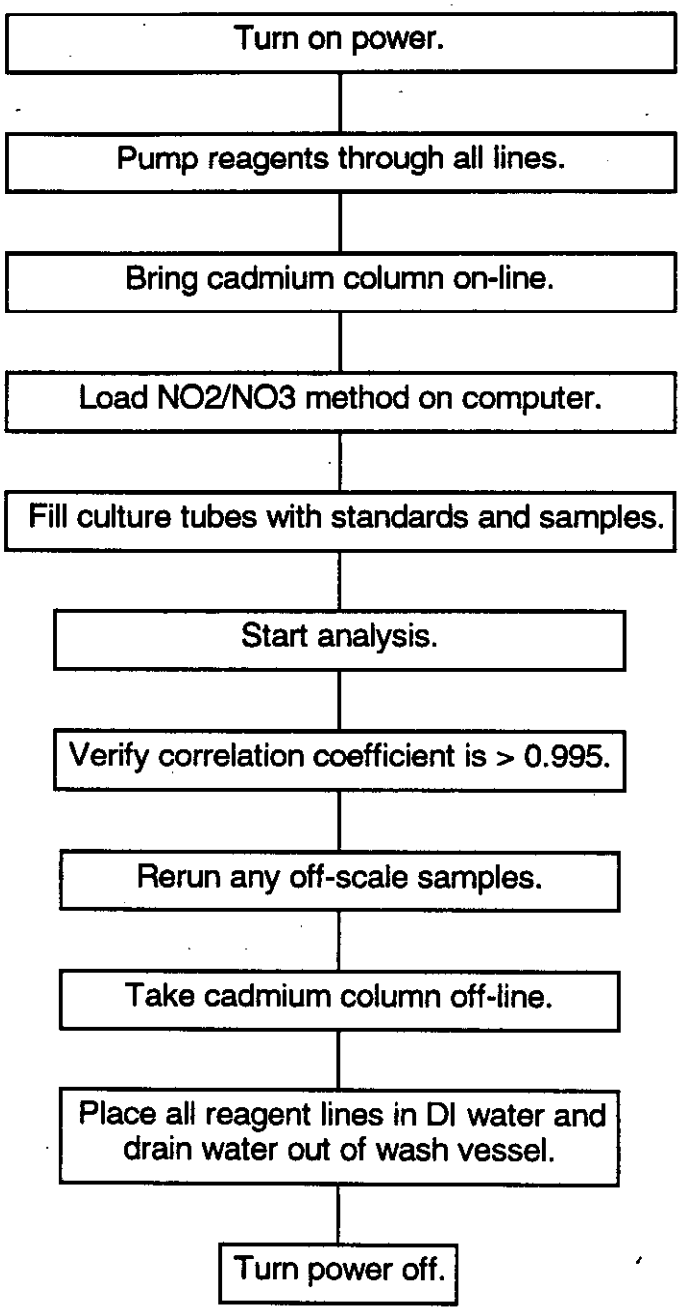
16.3.3. QA Policy 008 Data Recording Requirements, Rev. 0.

16.3.4. Chemical Hygiene Plan, Attachment C, Hazardous Waste Management Procedures, Revision 0.

16.3.5. Deionized Water Leaching Procedure for Non-Aqueous Samples.

16.3.6. Extraction of Nitrocellulose from water and soils samples.

Nitrate/Nitrite by Lachat AE Autoanalyzer
SAC-WC-0036



Appendix 1

Example Forms

(Reviewer Checklist)
(Example Lachat Report)
(Instrument Run Log)
(Non-Metals Extraction Log)

UNCONTROLLED COPY

STANDARD OPERATING PROCEDURE

Subject of Title: **OIL AND GREASE, GRAVIMETRIC (EPA 413.1 AND EPA 9070)**

SOP No.:	Revision:	Effective Date:
LM-CRL-1026	0	February 1, 1992
	Supersedes: none	

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Prepared by: David C. Dawes	Date: 3/11/92
Management Approval: [signature]	Date: 3/23/92
QA Officer Approval: Paul Christi	Date: 3/27/92

C4-591

1. Scope and Application

1.1 This method includes the measurement for fluorocarbon-113 (freon) extractable matter from surface and saline waters, industrial and domestic wastes. It is applicable to the determination of relatively non-volatile hydrocarbons, vegetable oils, animal fats, waxes, soaps, greases and related matter. It is NOT applicable to the measurement of light hydrocarbons that volatilize at temperatures below 70 degrees Centigrade.

1.2 Reporting limits: 5 mg/L for liquids
100 mg/Kg for solids

1.3 Applicable matrices: Water, soil, solids, sludge and waste.

1.4 Dynamic range: This method covers the range from 5 to 1000 mg/L of extractable material for liquids. The range for solids is from 100 to 100,000+ mg/Kg.

1.5 Analysis time: Total hands-on time for analysis of samples is approximately 30 minutes per sample (15 samples + 3 QC per shift). Because of drying, total time for analysis is approximately 24 hours for 1 to 15 samples.

2. Method Summary

2.1 Oil and grease is determined differently depending on whether sample matrix is liquid or solid.

2.1.1 For liquids, an acidified sample (pH<2) is serially extracted with freon in a separatory funnel. The solvent is then evaporated from the extract and the residue weighed.

2.1.2 For solids, a representative aliquot of the sample is placed in a glass container with sodium sulfate and serially extracted with freon. The solvent is then evaporated from the extract and the residue weighed.

3. Comments

3.1 Interferences

3.1.1 Matrix interference is likely if samples contain any non-oil substances that dissolve in freon.

3.1.2 Scrupulous care must be taken to avoid any water being extracted which will lead to erroneously high results.

4. Safety Issues

4.1 All employees are expected to be familiar with and follow the procedures outlined in the Enseco safety plan. Lab coats and safety glasses are required in all laboratory areas at all times. Consult your supervisor or safety officer with any specific questions or safety concerns.

4.2 Wear gloves when handling concentrated acids and solvents. Transport them only in approved containers. Avoid breathing fumes and vapors of freon by venting separatory funnels and evaporating extracts inside a fume hood.

4.3 The toxicity or carcinogeneity of each chemical used in this procedure has not been precisely defined. Each chemical and sample should be treated as a potential health hazard.

4.4 A Material Safety Data Sheet (MSDS) is available for each laboratory reagent used in this analysis. These should be reviewed by the analyst before handling the chemicals.

5. Sample Collection and Preservation

5.1 Liquid samples are collected in 1 liter glass bottles. If analysis is to be delayed for more than a few hours, the sample is preserved by the addition of 1 mL concentrated sulfuric acid at the time of collection and refrigerated at 4° C.

5.2 Solid samples are to be sealed and refrigerated at 4° C.

5.3 The holding time for liquids is 28 days from the date of sampling. There is no defined holding time for solid samples.

6. Apparatus

6.1 Separatory funnel, 2000 mL with Teflon stopcock.

6.2 Boiling flask, 125 mL with ground glass joint.

6.3 Distilling arm to fit boiling flask and collection bottle.

6.4 Collection bottle, 1000 mL capacity.

6.5 Filter paper, Whatman 1/PS 11 cm diameter.

6.6 Glass funnel for filter paper.

6.7 Water bath, 70° C.

6.8 Glass bottles, 6 ounce, with screw cap (for solids).

6.9 pH indicator paper, 0-14 range.

6.10 Graduated cylinder, 1000 mL

7. Reagents and Standards

7.1 Sulfuric acid, concentrated.

7.2 Freon-113, 1,1,2-trichloro-1,2,2-trifluoroethane(freon), b.p.=48° C. NOTE: Each new batch of freon must be analyzed by NDIR (EPA 413.2) to verify that it contains less than 5mg/L oil and grease.

7.3 Sodium sulfate, anhydrous crystal.

7.4 DCS solution. Weight 1 gram 3-in-1 oil to the nearest 0.1 mg into a 100mL volumetric flask and dilute to mark with freon. Calculate spike amount based on 5mLs in 1000mL DI water. Refrigerate at 4° C. EXPIRES IN 3 MONTHS.

8. Procedure

8.1 For liquid samples:

8.1.1 Mark the sample bottle at the water meniscus for later determination of sample volume.

8.1.2 Measure sample pH with indicator paper. If pH is not less than 2 then add 1mL concentrated sulfuric acid to sample and mix. Check pH again. If not less than 2, add subsequent mLs of acid until it is less than pH 2. Record on benchsheet any sample that is unpreserved.

8.1.3 Pour the sample into a separatory funnel.

8.1.4 Tare a boiling flask that has been pre-dried at 103° C. and desiccated until cool to the nearest 0.1 mg.

8.1.5 Add 30mL freon to the empty sample bottle and rotate to rinse the sides. Transfer the solvent into the separatory funnel. Extract by shaking vigorously for 2 minutes. Allow the layers to separate then filter the lower layer (freon) through 1 gram sodium sulfate salt placed in the filter paper cone. Both filter paper and salt should be moistened with solvent before sample is added.
Note 1: If an emulsion forms, drain it slowly through the sodium sulfate. Add more salt as required.
Note 2: To avoid having water penetrate the filter paper, leave a small amount of freon behind in the funnel after each extraction.

8.1.6 Repeat 8.1.5 twice more with additional portions of fresh solvent. (Do not rinse container further.) Combine all three extracts in the flask.

8.1.7 Rinse the separatory funnel tip, the filter paper and sodium sulfate with 10-20mLs freon. Add this to the extract already in the flask

8.1.8 Connect the boiling flask to the distilling arm and evaporate the solvent by immersing the lower half of the flask in water at 70° C.

8.1.9 When the flask appears dry, remove it from the water bath. Carefully wipe the flask down to remove excess water and then place it in a drying oven set at 60° C for 1 hour. Desiccate flask until cool.

8.1.10 Weigh the flask to the nearest 0.1 mg and record on benchsheet. If weight is not stable then return flask to desiccator for further cooling.

8.1.11 Determine volume of sample used by filling sample bottle to meniscus mark (8.1.1) with water and then transferring to a 1000mL graduated cylinder.

8.2 For soil samples:

8.2.1 Weigh out 10 grams sample to the nearest 0.1 gram. Place sample in a 6 ounce glass bottle.

8.2.2 Add 30mL freon to bottle and shake vigorously for 10-15 minutes (mechanical shaker can be used).

8.2.3 Pour extract through moistened sodium sulfate salt and filter paper into a tared flask as described in 8.1.5. Add more salt as needed to prevent water from passing through the filter.

8.2.4 Repeat 8.2.2 and 8.2.3 twice, combining all three extracts in the tared flask.

8.2.5 Rinse the salt and filter paper with 10-20 mLs of additional freon and combine with extracts.

8.2.6 Evaporate solvent and dry flask as described in 8.1.8 through 8.1.10.

9. Calculation

9.1

$$\text{mg/L or mg/Kg oil and grease} = \frac{R - T}{A} \times 1{,}000{,}000$$

where:

R = residue plus flask weight in grams
T = tare weight of flask in grams
A = aliquot of sample, liquids in mLs, solids in grams.

10. QA/QC Requirements

10.1 QC Samples

10.1.1 One blank is required with every batch of 20 or less samples. A blank consists of 1000mL acidified DI water which is taken through all steps of the analysis.

10.1.2 A pair of LCS samples (DCS) is required with every 20 or less samples. An LCS consists of 5mL DCS solution (7.4) added to 1000 mL DI water, acidified, and taken through all steps of the analysis.

10.2 Acceptance Criteria

10.2.1 Recovery and RPD on DCS samples must be within laboratory control limits which are updated periodically.

10.2.2 Blank result must be less than the reporting limit.

10.3 Corrective Action

10.3.1 If the blank reads greater than the reporting limit but is less than twice its value then the data can be reported with a notation. If the blank is greater than twice the reporting limit then the data is rejected. In either case, the solvent should be re-checked by NDIR and discarded if found to be greater than 5 mg/L. If the solvent is "clean" then all transfer containers should be checked for contamination and cleaned.

C4-597

10.3.2 If the DCS average percent recovery and/or the RPD are outside of control limits then the data is rejected. The DCS solution should be discarded.

11. Reporting

11.1 Report all results as either mg/L or mg/Kg Oil and Grease.

11.2 Results below the detection limit are reported as ND.

11.3 Significant figures to report are 2 for all results less than 10 ppm and 3 for all results greater than or equal to 10 ppm.

12. References

12.1 Method Source:

12.1.1 EPA-600, 1974 Edition. Method 413.1.

12.1.2 SW-846, Third Edition 9/86. Method 9070.

12.2 Deviations from Source Method

12.2.1 1 mL concentrated sulfuric acid is used to acidify samples instead of 5 mLs 1:1 hydrochloric.

12.2.2 Whatman PS/1 filter paper used instead of No. 40.

12.2.3 Flasks with residue are dried in a 60° C oven for 1 hour and desiccated until cool instead of sweeping out with vacuum and desiccating for 30 minutes.

12.2.4 Samples are not blank subtracted.

12.2.5 Extraction procedure for soils is a modified addition to the method.

Subject or Title:
Cyanide, Total: Manual Micro Distillation

SOP No.:	Revision No.:	Effective Date:
LM-CAL-1053	Original	June 28, 1990

Supersedes: None

1. SCOPE AND APPLICATION

1.1 This method is applicable to the determination of total cyanide in drinking, surface, and saline waters, domestic and industrial wastes, sludges, and soil samples. The method detects cyanide compounds that are present, as either simple soluble salts or complex cyanides. This is used to determine values for total cyanide.

1.2 The detection limit is 0.010 mg/L for aqueous samples and 0.5 mg/kg for non-aqueous samples, using the automated colorimetric method of analysis. The range extends to 0.5 mg/L for aqueous samples and to 25 mg/kg for non-aqueous samples.

1.3 A batch of 10 samples is processed through distillation in about 3 hours.

2. SUMMARY OF METHOD

2.1 The cyanide as hydrocyanic acid (HCN) is released from acidified cyanide complexes by means of a reflux distillation operation and is absorbed in a scrubber containing sodium hydroxide (NaOH) solution. The cyanide ion in the absorbing solution is then determined colorimetrically.

3. COMMENTS

3.1 Interferences

3.1.1 Physical interferences are eliminated or reduced during the distillation process.

Prepared By:	Date:
John Barnett	May 25, 1990
Management Approval:	Date:
[illegible]	June 28, 1990
QA Officer Approval:	Date:
B McNeill	June 28, 1990

3.1.2 Sulfides can seriously affect the colorimetric analysis. Sulfide presence is indicated by lead acetate paper turning blue when moistened with a drop of sample. It can be eliminated by adding powdered cadmium carbonate to approximately 75 mL of sample. Yellow cadmium sulfide will precipitate. Add the cadmium carbonate until the sample no longer darkens the lead acetate paper. Filter the solution through filter paper into a graduated cylinder. Avoid a large excess of cadmium and a long contact time in order to minimize a loss by complexation or occlusion of cyanide (CN) on the precipitated material.

3.1.3 Oxidizing agents such as chlorine can destroy cyanide. Chlorine presence is indicated by potassium iodide-starch test paper turning blue when moistened with a drop of sample. Chlorine can be eliminated by adding ascorbic acid, a few crystals at a time until a drop of sample produces no color on the indicator paper. Then add an additional amount (approximately 0.06 g) of ascorbic acid to the sample. Maintain pH of sample at >12 during treatment by adding NaOH pellets if needed.

3.1.4 Carbonate in high concentration may affect the distillation by causing excessive gassing when acid is added. The carbon dioxide (CO2) released may significantly reduce the NaOH content of the scrubber. Bubbles formed in the flow cell may interfere with the automated colorimetric analysis.

3.1.5 The presence of surfactants may cause the sample to foam during refluxing. If this occurs, the addition of an agent such as Dow Corning 544 Antifoam Agent will prevent the foam from collecting in the condenser. This can be prevented in certain samples by first adding $MgCl_2$ and then Conc. H_2SO_4. The foaming can be eliminated or minimized.

3.1.6 Nitrate and/or nitrite may react with organic compounds during distillation and ultimately form cyanide. They may be removed prior to distillation with sulfamic acid.

4. SAFETY ISSUES

4.1 Follow Enseco-Cal Lab safety regulations (e.g., gloves, glasses, and lab coats (GGC), safety sheet, and Emergency Preparedness Plan).

4.2 Cyanide is highly toxic. Handle it only in a fume hood. Clean up spills immediately. If it comes into contact with acid the extremely toxic gas hydrocyanic acid forms.

4.3 In case of skin contact with cyanide, bases, or acids, flush area with water for 15 minutes.

4.4 Work with concentrated acids only in a fume hood; avoid breathing fumes. Neutralize and clean up spills immediately. Transport concentrated acid bottles only in impact-proof containers.

4.5 Conduct distillation only in a fume hood. Build up of pressure in the apparatus can cause hot acidic solution to spray out of the thistle tube. Keep the sash of the hood down while samples boil.

5. SAMPLE COLLECTION, PRESERVATION, CONTAINERS, AND HOLDING TIMES

5.1 Liquid samples should be collected in 1000 mL plastic or glass bottles with 2 mL of 10% N NaOH as preservative.

5.2 Solid samples can be collected in an 8 oz. jar, or a metal core tube.

5.3 All water and wastewater samples must be distilled as soon as possible and analyzed within 14 days after sampling.

5.4 Contract Laboratory Program (CLP) soil/sediment samples must be distilled and analyzed within 14 days of sample receipt.

5.5 Store at 4°C.

6. APPARATUS

6.1 Microdistillation set-up.

6.2 Heat controller.

6.3 Glassware: 100-mL and 1-L volumetric flasks
100-mL graduated cylinders.

6.4 Top pan balance.

6.5 125 mL Nalgene bottles

7. REAGENTS AND STANDARDS

7.1 Sodium hydroxide solution, 1.25 N: Dissolve 50 g of NaOH in approximately 300 mL of deionized water in a 1-liter volumetric flask. Dilute to the mark.

7.2 Sulfuric acid, concentrated.

7.3 Magnesium chloride solution: Dissolve 510 g of $MgCl_2$-$6H_2O$ in approximately 500 mL of deionized water into a 1-liter volumetric flask. Dilute to the mark.

7.4 Stock cyanide standard, 1000 ppm: Dissolve 2.51 g of KCN and 2.0 g of NaOH in about 300 mL deionized water in a 1-liter volumetric flask. Dilute to mark.

7.5 Working cyanide standard, 10 ppm: Add 1.0 mL of 1000ppm stock standard to 20 mL of 1.25 N NaOH in a 100-mL volumetric flask. Dilute to the mark with deionized water.

7.6 pH test strips.

7.7 Lead acetate paper.

7.8 KI-starch test paper.

7.9 Cadmium carbonate, powdered.

7.10 Ascorbic acid crystals.

7.11 Sulfamic acid solution, 0.4N: Dissolve 40g of sulfamic acid crystals in 500 mL of deionized water in 1 liter volumetric flask. Dilute to mark.

7.12 Dow Corning 544 antifoaming agent.

8. PROCEDURE

8.1 For aqueous samples:

8.1.1 Measure pH of samples with pH test strips. If >12 indicate so on log sheet. If <12 notify supervisor and indicate approximate pH on log sheet.

8.1.2 If samples are known or suspected to contain sulfide, test with lead acetate paper. If positive, proceed as in 3.1.2.

8.1.3 If samples are known or suspected to contain chlorine, proceed as in 3.1.3.

8.1.4 Pour 50 grams, or portion of the sample diluted to 50 grams, in the 100 mL microdistillation flask. Add a few boiling stones.

8.1.5 If samples are known or suspected to contain nitrate and or nitrite, add 5 mL of 0.4 N sulfamic acid solution.

8.2 For solid samples:

8.2.1 Measure 2.0 g of well-mixed sample into the microdistillation flask. Add a few boiling stones. Add 50 g deionized water.

8.2.2 If samples are known or suspected to contain sulfide, test with lead acetate paper. If positive, proceed as in 3.1.2.

8.2.3 If samples are known or suspected to contain chlorine, proceed as in 3.1.3.

8.2.4 If samples are known or suspected to contain nitrate and or nitrite, add 5 mL of 0.4 N sulfamic acid solution.

8.3 Connect the flask to the condenser. Fill the absorbing tube with 20 mL of 1.25 N NaOH and connect it to the scrubber. Connect air line and be sure all connections are airtight.

8.4 Turn on condenser water at a sufficient rate to cool condenser. Flow of water must be checked when solution starts refluxing. Be sure that condenser stays cool by increasing water flow.

8.5 Start a stream of air entering the boiling flask by adjusting the vacuum to approximately two bubbles per second. The air flow must be monitored throughout the analysis. The bubble rate will not remain constant after reagents are added and while heat is applied to the flask.

8.6 Slowly and carefully add 3.0 of concentrated H_2SO_4 to the inlet tube.

8.7 Slowly add 2.0 mL of magnesium chloride solution and follow with a distilled water rinse of the inlet tube. Swirl gently.

8.8 Heat the sample to boiling and reflux for one hour after condensation appears. Turn off heat and continue bubbling for 15 minutes.

8.9 Remove the absorption tube from the distillation apparatus. Rinse twice the inside and outside of the bubbler into the tube with dHOH. Pour the contents of the tube (1.25N NaOH) into a 125 mL Nalgene bottle. Rinse twice. Bring to a total volume of 100 grams.

8.10 Label the bottle with the original sample volume or weight, the final distillate volume (100g), and the sample number. If it is a redistillation, put that on the bottle along with the date. If it is a spike, label it as such and include the spike concentration as it applies to the sample. If it won't be analyzed that day, store in a refrigerator.

8.11 After distillation wash the inlet tube, flask, and absorbing tube with soap and water. Then rinse absorbing tube with HCl rinsewater to ensure complete removal of CN.

8.11.1 The rest of the apparatus can be rinsed with deionized water between distillations.

9. QUALITY ASSURANCE/QUALITY CONTROL (QA/QC) REQUIREMENTS

9.1 Definition

9.1.1 A batch is a group of samples processed at the same time.

9.2 QC Samples for Contract Laboratory Program (CLP)

9.2.1 One preparation blank per batch or per 20 samples, whichever is more frequent. Use deionized water.

9.2.2 The ICV solution must be distilled and analyzed with samples.

9.2.3 For each matrix and each concentration level, one sample duplicate per batch or per 20 samples, whichever is more frequent. Sample may be designated by EPA.

9.2.4 For each matrix and each concentration level, one sample spike (add 0.5 mL of 10 ppm CN to 50 g of aqueous sample or 2 g of solid sample) per batch or per 20 samples, whichever is more frequent. Sample may be designated by EPA.

9.3 QC Samples for Commercial Work (Non-EPA)

9.3.1 One preparation blank per batch or per 20 samples, whichever is more frequent. Use 50 g deionized water.

9.3.2 Duplicate Control Samples (DCS) per 20 samples. Add 0.5 mL of 10 ppm CN to 50 g deionized water.

9.3.3 A Single Control Sample (SCS) per analytical lot (here defined as all samples processed in a 24-hour period). Add 0.5 mL of 10 ppm CN to 50 g deionized water.

9.3.4 One sample duplicate - only when requested by client.

9.3.5 One sample spike (add 0.5 mL of 10 ppm CN to 50 g of aqueous sample or 2 g of solid sample) - only when requested by client.

10. REFERENCES

10.1 Method Sources

10.1.1 Method 335.2, Methods for Chemical Analysis of Waters and Wastes (EPA).

10.1.2 Standard Methods for the Examination of Water and Wastewater, 16th edition.

10.1.3 Method 9010, SW-846, 2nd edition.

10.1.4 CLP-M, Method 335.2, modified for the Contract Laboratory Program.

10.2 Deviations to Method Sources and Rationale

10.2.1 This method can be used for CLP-M, Method 335.2; MCAWW 335.2; SW846 9010; and Standard Methods. The method variances are minor.

10.2.2 This method uses a micro-distillation set-up so that smaller amounts of sample are used than in the quoted methods. This has been approved by EPA as an acceptable variance.

10.2.3 SW846 Method 9010 included both total cyanide and amenable cyanide. If only total cyanide is requested by 9010, we perform only the total cyanide test and quote Method 9010.

10.2.4 To reduce sulfide interferences when a sample contains sulfide, this method uses cadmium carbonate powder to precipitate the sulfide. This is from CLP-M Method 335.2. Method 9010 uses bismuth nitrate and Method MCAWW 335.2 uses lead acetate. The cadmium carbonate is an acceptable alternative for all methods.

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STANDARD OPERATING PROCEDURE

Subject or Title: Cyanide, Total: Automated Analysis		Page 1 of 11
SOP No.: LM-CAL-1034	Revision No.: 2.0	Effective Date: October 12, 1993
Supersedes: June 15, 1989		

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1. SCOPE AND APPLICATION

1.1 This method is applicable to the determination of:

Total cyanide
Cyanide amenable to chlorination
Weak acid dissociable cyanide

1.2 Sample preparation for the above parameters is described in separate SOP's, i.e., SOP No. LM-CAL-1099 (total), LM-CAL-1102 (amenable to chlorination), LM-CAL-1101 (weak acid dissociable).

Prepared By: Don Fredrickson	Date: October 12, 1993
Management Approval: Lori Ann Upton	Date: 11-8-94
QA Officer Approval: [illegible]	Date: 11/8/94

C4-607

1.3 Detection Limits:

Parameter	Waters	Soil/Waste
Total cyanide	0.01 mg/L	0.5 mg/kg
Amenable to chlorination	0.01 mg/L	0.5 mg/kg
Weak acid dissociable	0.01 mg/L	0.5 mg/kg

1.4 Applicable matrices

1.4.1 This method is applicable to drinking, surface, and saline waters, domestic and industrial wastes, soil and other non-aqueous matrices.

1.5 Dynamic range

1.5.1 The range extends to 0.5 mg/L (aqueous samples) and to 25 mg/kg (non-aqueous samples). Higher concentrations are analyzed by dilution of the sample.

1.6 Approximate analytical time is 1 hour for a group of 20 samples, not including time for instrument set-up.

2. SUMMARY OF METHOD

2.1 Samples for total cyanide are prepared by distillation of the cyanide from an acidic medium.

2.2 Cyanide amenable to chlorination is prepared by chlorinating an aliquot of the sample and then distilling for total cyanide. A second aliquot is distilled without chlorination for total cyanide. Cyanide amenable to chlorination is defined as the difference between the two results.

2.3 Weak acid dissociable cyanide is prepared by distillation from a slightly acidified solution.

2.4 Samples prepared by the above methods are analyzed colorimetrically on a Autoanalyzer System. The sample is buffered and reacted with Choramine-T to produce cyanogen chloride. This then reacts with a pyridine-barbituric acid reagent to produce a red colored complex.

3. COMMENTS

3.1 Interferences

3.1.1 Oxidizing agents such as chlorine will destroy cyanide.

3.1.2 Some unidentified organic compounds may oxidize or form decomposition products during chlorination, giving higher results for cyanide after chlorination than before chlorination; this gives a negative value for cyanide amenable to chlorination. Examples include samples from petroleum refineries, the steel industry, and pulp and paper processing. The weak acid dissociable method should be used for these samples.

3.1.3 Sulfide may interfere with the analysis. Sulfide is tested for and removed during sample prep.

3.1.4 Chlorine added to the sample for amenable cyanide must be completely destroyed before distillation. Otherwise, it may distill over and destroy the non-amenable cyanide.

3.1.5 Nitrate and/or nitrite may react with organic compounds during distillation to form cyanide.

3.1.6 Samples containing surfactants may foam excessively during distillation.

3.1.7 The extent to which some metal-cyanide complexes are destroyed by chlorination depends on the amount of chlorine present and the time allowed for reaction.

3.1.8 High carbonate concentrations may react violently when sulfuric acid is added to the samples during distillation. It may also interfere with the automated colorimetric analysis by forming bubbles in the flow cell.

3.1.9 Aldehydes, glucose, and other sugars may convert cyanide to cyanohydrin during distillation.

3.1.10 Amino acids may distill with the cyanide and interfere with the analysis.

3.1.11 Fatty acids may interfere by forming soaps in the absorption solution.

3.2 Helpful hints

3.2.1 Cyanide amenable to chlorination is sometimes referred to as "Free Cyanide." The weak acid dissociable method also determines "Free Cyanide" and it is important to identify the method due to the different sample preparation.

4. SAFETY ISSUES

4.1 Follow Enseco-Cal Lab safety regulations (e.g., Gloves, Glasses, and Lab Coats (GGC), Safety Sheet, and Emergency Preparedness Plan).

4.2 Cyanide is highly toxic. Prepare standards in a hood. Clean up spills immediately. Do not allow acids to come into contact with cyanide salts or concentrated solutions - extremely toxic HCN gas will form from acid and cyanide.

4.3 Pyridine and chloramine-T are eye/skin/membrane irritants. The pyridine-barbituric acid and chloramine-T reagents should be prepared in a fume hood and kept covered as much as possible.

4.4 In case of skin contact with samples, standards, or reagents, flush area with water for at least 15 minutes.

4.5 The waste from the analysis is slightly acidic. Add sodium hydroxide to the waste container to keep any unreacted cyanide from escaping into the air.

5. SAMPLE COLLECTION, PRESERVATION, CONTAINERS, AND HOLDING TIMES

5.1 Water samples are to be collected in plastic bottles and preserved with 2 ml of 10 N sodium hydroxide per liter of sample. Refrigerate at 4°C.

5.2 Soil and waste samples should be collected in wide mouth jars and refrigerated at 4°C.

5.3 The holding time for water and soil samples is 14 days from collection. For CLP samples, the holding time is 12 days from receipt of samples.

6. APPARATUS

6.1 Lachat autoanalyzer consisting of auto-sampler, pump, injection module, colorimeter, and computer.

6.2 570 nm filter and 10 mm flow cell for colorimeter.

6.3 Miscellaneous laboratory apparatus and glassware.

7. REAGENTS AND STANDARDS

7.1 Pyridine-Barbituric Acid Reagent

7.1.1 Place 15g barbituric acid in a 1000 mL volumetric flask and add about 100 mL distilled water. Add 75 mL pyridine and mix. Carefully add 15 mL concentrated hydrochloric acid and mix. Dilute to volume with distilled water and store at 4°C.

7.2 Phosphate Buffer Solution

7.2.1 Dissolve 97g potassium dihydrogen phosphate momnohydrate in distilled water and dilute to 1000 mL.

7.3 Chloramine-T

7.3.1 Dissolve 4.0g chloramine-T in distilled water and dilute to 1000 mL. Prepare fresh daily and keep covered.

7.4 Sampler Wash--Sodium Hydroxide, 1%

7.4.1 Dissolve 10g sodium hydroxide in distilled water. Cool to room temperature, dilute to 1000 mL, and mix well. Store in a plastic bottle.

7.5 Calibration Standard Stock Solution, 1000 mg/L

7.5.1 Dissolve 2.51g potassium cyanide and 2g sodium hydroxide in distilled water and dilute to 1000 mL. Standardize using 0.0192 N AgNO3. The titrated value should be within 5% of expected.

7.6 10 mg/L Intermediate Standard.

7.6.1 Pipette 1.0 mL Stock Standard (1000 mg/L) into a 100 mL volumetric flask. Add 20 mL 1.25 N sodium hydroxide, dilute to volume with distilled water and mix. Prepare fresh bi-weekly.

7.7 Calibration Standards

7.7.1 Using 100 mL volumetric flasks add 20 mL 1.25 N NaOH, add amount of intermediate standard as below, and dilute to volume with distilled water. Prepare these standards biweekly.

Aliquot, mL	Final Vol., mL	Concentration, mg/L
0	100	Blank
0.1	100	0.01
0.5	100	0.05
2.0	100	0.20
4.0	100	0.40
5.0	100	0.50

7.7.2 The blank also serves as the Continuing Calibration Blank (CCB).

7.7.3 A mid-range standard (typically the 0.2 mg/L) serves as the Continuing Calibration Verification (CCV).

7.8 Initial Calibration Verification (ICV) Standard

7.8.1 A second source is used for both CLP and non-CLP samples.

8. PROCEDURE

8.1 Set up the manifold. Pump reagents through the system until a steady baseline is obtained.

8.2 While the system is pumping, prepare the calibration standards.

8.3 When a steady baseline is obtained, adjust it to the bottom of the chart with the baseline control.

8.4 Start the sampler and adjust the peaks for the high standard to full scale with the standard cal control. Readjust the baseline if necessary.

8.5 Analyze the calibration standards in order of increasing concentration, followed by the ICV, ICB, Reference Standard, prep blank, DCS/SCS, and billable samples. A CCV and a CCB must be analyzed at least every 10 samples (distilled items), and again at the end of the run.

8.6 Each QC lot should be analyzed together as a group. Dilute any samples that are greater than the high standard and reanalyze. All dilutions must be performed with 1% sodium hydroxide. The sequence of reference, samples, etc. should be recorded on the bench sheet.

8.7 At the end of the run, flush the system with distilled water and turn off the instrument.

9. QA/QC REQUIREMENTS

9.1 QC for CLP Samples (Contract Laboratory Program)

9.1.1 A prep blank must be analyzed after the ICV.

9.1.2 As a control sample, an ICV, a 0.1 mg/L standard (mid-range) must be distilled and analyzed with every analytical run. This should be run immediately after the calibration.

9.1.3 After every 10 samples and at the end of the run a CCV and a CCB must be run.

9.1.4 A duplicate and a sample spike (0.10 ppm) must be run for each batch or for every 20 samples, whichever is more frequent.

9.2 QC for Non-CLP Samples

9.2.1 An ICV is run immediately after the calibration standards. The ICV must be distilled with the samples.

9.2.2 An ICB must be run after the ICV.

9.2.3 A prep blank, distilled with the samples, must be analyzed after the Reference Standard.

9.2.4 Duplicate Control Samples (DCS) and Single Control Samples (SCS), are spiked with CN at 0.10 ppm and prepared with the samples. DCS (two samples) are required for every 20 samples. An SCS is required with every analytical lot (here defined as all samples processed in a 24-hour period).

9.2.5 Duplicates may be required for project specific QC. Duplicates are to be taken through the entire prep and analysis together.

9.2.6 Sample spikes may be required for project specific QC. Spikes are prepared with the samples.

9.2.7 A CCV and CCB are required after every 10 samples and at the end of the run.

9.3 Acceptance Criteria

9.3.1 ICB's and CCB's must be less than the reporting limit.

9.3.2 The ICV must be within control limits of 85-115% for CLP-like projects. Alternatively, non-CLP projects will be controlled using historical control limits provided in LIMS.

9.3.3 DCS/SCS recovery and Relative Percent Difference (RPD) for DCS must be within control limits established according to the Laboratory Performance QC SOP, M-EQA-002. Current control limits are in LIMS.

9.3.4 Control limits for CLP sample spike recoveries and duplicate RPD's are, with certain exceptions described in the CLP Statement of Work, 75-125% and 20% respectively.

9.3.5 The standard curve must have a correlation coefficient of 0.995 or greater.

9.3.6 CCV's must be within 10% of the expected value.

9.4 Corrective action required

9.4.1 Check the dilutions made when preparing standards and redilute if necessary.

9.4.2 Reprep samples if DCS/SCS results are out of the control limits or if the blank shows excessive contamination.

9.4.3 If the sample spike recovery is not within the control limits a post-distillation spike must be run. This is done by spiking the unspiked sample distillate at twice the sample concentration or twice the reporting limit whichever is greater.

9.4.4 For cyanide amenable to chlorination, if the chlorinated aliquot shows more cyanide than the unchlorinated aliquot, the sample must be reprepped using the Weak and Dissociable method.

10. CALCULATIONS

10.1 Cyanide amenable to chlorination:

10.1.1 Subtract the result for the chlorinated aliquot from the unchlorinated aliquot. This difference is cyanide amendable to chlorination.

11. REPORTING

11.1 Reporting units are ug/L for waters and mg/kg for nonaqueous samples.

11.2 Reporting limits are 10 ug/L for waters and 0.5 mg/kg for nonaqueous samples. Samples less than the reporting limit are reported as ND.

11.3 Significant Figures:

	0.01-0.09	0.10-0.99	1.0-9.9	10-99	100-999	1000+
Waters	0.0X	0.XX	X.X	XX	XXX	XXX0
Soils	--	0.X	X.X	XX	XXX	XXX0
Reactive	--	0.X	X.X	XX	XXX	XXX0

12. REFERENCES

12.1 Method sources: EPA Method 335.3 and EPA Method 335.2 CLP-modified.

12.1.1 Additional information was obtained from Standards Methods and ASTM.

12.2 Deviations from source method and rationale

12.2.1 The automated method is used following manual distillation. Automatic distillation is not performed.

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QUANTERRA® STANDARD OPERATING PROCEDURE

TITLE: NONCONFORMANCE AND CORRECTIVE ACTION

(SUPERSEDES: REVISION NO. 0 12/11/95)

Prepared by: Quanterra® QA Managers

Approved by: _____
Director of Quality Assurance

Approved by: _____
Director of Environmental Health and Safety

Approved by: _____
Vice President Operation Services

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C4-618

TABLE OF CONTENTS

1. PURPOSE 4

2. RESPONSIBILITIES 4

2.1. Laboratory Associate 4

2.2. Group Leader/Team Leader/Supervisor 4

2.3. Project Manager (PM 5

2.4. QA Manager 5

2.5. Operations/Systems Manager 5

2.6. Laboratory Manager 5

2.7. Corporate QA Director 5

3. SAFETY 6

4. PROCEDURE 6

4.1. When to Initiate a Nonconformance Memo 6

4.2. How to Process the NCM Form 7

4.3. Supervisory Review, Approval, and Signature 8

4.4. Project Manager Review, Client Notification, and Project Documentation 9

4.5. Quality Assurance Review and Tracking 10

4.6. Externally Generated Corrective Actions 11

5. DEFINITIONS 11

5.1. Nonconformance 11

5.2. Corrective Action 12

C4-619

6. MISCELLANEOUS 12

6.1. Associated Documents 12

6.2. Appendices 13

6.3. Flow Chart for Internal NCM 14

1. PURPOSE

1.1. The purpose of this document is to establish procedures for the identification and documentation of nonconformances and the corrective actions taken as a result of these events. The Quanterra® Quality Assurance Management Plan (QAMP) requires documentation of instances of deviations from established control limits, approved SOPs, or client-specified requirements. The Nonconformance Memo (NCM) described in this procedure is used to document deviations from plan specifications together with their cause and correction. The NCM maybe a written document or a defined electronic formatted document transmitted via e-mail.

1.2. This document applies to procedures, services, data, reports, or materials purchased by the laboratory or supplied by the laboratory to its clients. Nonconformances related to incoming samples may be documented separately from the NCM process by use of the Condition Upon Receipt (CUR) Form as described in a facility-specific sample receiving/log-in standard operating procedure (SOP). Regardless of which system a lab chooses to use to handle these sample receipt client-related issues, including holding time violations (HTVs), the system must include and emphasize the immediate notification of the Project Manager (PM). This will allow the PM to initiate immediate client notification and resolution of how to proceed. See Section 5, Definitions, for further clarification of application.

1.3. Identification of suspected adverse conditions (deviations) can be made by laboratory employees in the course of their work or by individuals outside Quanterra's® laboratories through review of records, audit, or proficiency testing.

2. RESPONSIBILITIES

2.1. **Laboratory Associate:** During the course of their work, all employees are responsible for identifying and documenting problems, using a Nonconformance Memo, that might affect the quality of Quanterra's® product. They should also identify or attempt to seek out possible measures to correct the problem. By signing or initialing laboratory notebooks, forms, bench sheets, data reports, and other quality-related documents, associates are verifying that procedures have been followed. Any deviation that might render a measurement suspect shall be documented.

2.2. **Group Leader/Team Leader/Supervisor** (for purposes of this SOP, the term "Supervisor" will be used): Each supervisor is responsible for the review of NCMs to ensure that problems which might affect quality are adequately described and that personnel are assigned to correct them. Supervisors sign NCMs and forward them to a project manager. Together with project managers and Quality Assurance personnel,

supervisors are responsible for determining the appropriateness of planned corrective actions.

2.3. **Project Manager (PM):** The project manager is responsible for relaying project requirements to staff so that special project requirements are understood and nonconformances recognized. The project manager communicates conformance problems to clients and documents decisions made with clients. The project manager ensures that short-term corrective actions for routine analytical QC failures are completed. An example would be making sure that repreparation and analysis of a sample was done. The project manager can and must withhold final reports to clients until corrective actions agreed to with the client have been completed.

2.4. **QA Manager:** The Quality Assurance manager or his or her designee is responsible for assigning log numbers to the NCMs as received, reviewing all prepared NCMs to ensure that actions taken are appropriate, and assisting in resolving QA/QC discrepancies. The QA staff will maintain a nonconformance tracking system to guarantee that each nonconformance is brought to closure. The system will also be used to monitor for trends that might indicate long-term quality problems. Systematic problems are investigated, NCMs issued and reviewed, and audits conducted to ensure that long-term corrective actions have been successfully completed. If review of an area reveals a significant problem with data quality, the Quality Assurance manager has the authority and responsibility to stop production in that laboratory area.

2.5. **Operations/Systems Manager** : The operations/systems manager shall ensure that corrective actions are correct and have been implemented. The operations/systems manager shall document this review and concurrence by signing the NCM as the responsible manager, if QA-required, for a specific corrective action. Along with the laboratory manager, the operations/systems manager shall emphasize the importance of quality requirements and require all employees to report any problem that might adversely affect the quality of work.

2.6. **Laboratory Manager:** The laboratory manager shall emphasize the importance of quality requirements and require all employees to report any problem that might adversely affect the quality of work. The laboratory manager is also responsible for the implementation of the NCM system in the laboratory.

2.7. **Corporate QA Director**: The Quanterra® Quality Assurance Director should be notified of any continuing non-conformances that are not properly addressed by operations or where the root cause cannot be identified.

3. SAFETY

3.1. Normal office dependent safety precautions must be taken in performing this SOP. If personnel are required to perform any portion of the procedure in laboratory areas, appropriate personal protective equipment and precautions must be utilized.

3.2. Procedures shall be carried out in a manner that protects the health and safety of all Quanterra® associates.

3.3. All work must be stopped in the event of a known or potential compromise to the health and safety of a Quanterra® associate. The situation must be reported **immediately** to a laboratory supervisor.

4. PROCEDURE

4.1. When to Initiate a Nonconformance Memo

4.1.1. Lab associates are to prepare a nonconformance memo (NCM) whenever procedures, services, data, reports, electronic disk deliverables (EDDs), or materials deviate from established specifications. All nonconformances require an NCM (see definitions of nonconformance, anomalies, and deficiencies in Section 5 and Section 1.2 for exceptions).

4.1.2. All standard operating procedures (SOPs) shall be followed. By signing or initialing laboratory notebooks, forms, data reports, and other quality-related documents, employees are verifying that the SOPs have been followed with the exceptions of the pre-approved deviations (as described in QAPjPs or Quality Assurance Summaries). Any intentional deviation from an SOP must be pre-approved by the QA manager and technical specialist. Any deviation from a SOP or client requirement not previously approved must be documented on an NCM.

4.1.3. An NCM is to be completed for each instance of a nonconformance. A single NCM can be used for a single event affecting multiple project numbers and samples, but normally a separate NCM would be prepared for different nonconformance issues. If the nonconformance involves projects for multiple project managers, then the NCM will need to be communicated to each project manager. The laboratories may utilize any communication system they feel best satisfies this required communication and maintains the integrity of the NCM process.

4.2. How to Process the NCM Form

4.2.1. Enter the information required at the top of the form (see Example Form in Attachment A; printed forms are available from QA). Names or initials must be uniquely identified by using three initials or by spelling out the last name. Use specific test codes (when available) rather than generic codes such as "VOAs" or "Anions." At a minimum enter Project ID, QuantIMS batch number or LIMS QC Lot number if appropriate, in addition to sample numbers. The laboratory generating the NCM is identified as part of the log number (e.g., AUS-97-123). Therefore, separate laboratory location identification is not required on the form.

4.2.1.1. If the NCM is for a holding time violation, the project manager ***must*** be notified immediately.

4.2.1.2. An instrument tag-out does not require the completion of the NCM if there are no client samples involved (e.g., instrument out of service to perform routine maintenance.) See section 4.2.5.

4.2.2. Check appropriate NCM boxes. If box 31—"Other"—is checked, supply a brief description of the problem. Attach further information as necessary.

4.2.3. Complete the reverse side of the form concerning root cause and corrective actions. These sections may be filled out jointly with the supervisor. Consult the project manager or the operations/systems manager and QA manager if the supervisor and associate are uncertain of corrective actions. Be objective and specific but brief. Include enough information that decisions to approve the NCM can be made easily (include pertinent QC information).

4.2.3.1. Design corrective actions to correct the immediate problem (short-term corrective actions) and to minimize the possibility of its recurrence (long-term corrective actions). Examples of corrective actions are modifications to nonconforming procedures, repair or replacement of deficient equipment, training personnel, and reanalysis of any affected samples. Additional sheets documenting corrective action may be attached to the NCM. Reference the attachment on the NCM.

4.2.3.2. Where operational corrective actions are required, they shall be supported with reference to recovery data, control charts, or other documentation.

4.2.4. If the corrective action involved retraining, the training must be documented with the signatures of the trainees and submitted to the QA staff.

4.2.5. Instrument/Equipment Nonconformance Tag

4.2.5.1. Instruments and equipment which habitually fail to meet calibration criteria or are out of service due to needed repair or other reasons must be marked with a tag indicating the nonconforming condition. (See Example in Attachment B.)

4.2.5.2. If the reason for the nonconformance tag caused sample data to be impacted, initiate an NCM and mark box 30. Identify the instrument by name and/or identification number and briefly describe the problem. This information can be recorded on page 2 under "Problem Description/Root Cause". Submit the NCM to the group supervisor.

4.2.5.3. Upon receipt of the NCM, QA assigns the NCM number and provides the number to the analytical group so that the nonconformance tracking number can be recorded in the instrument's maintenance log.

4.2.5.4. The corrective action will be to either permanently remove the instrument from service or to have the instrument repaired. If an instrument is repaired, its reliability must be demonstrated through successful recalibration before the nonconformance can be closed. The nonconformance tag remains in effect during the demonstration period. Record this information in the instrument maintenance logbook. Reference the successful calibration on the tag and return the tag to the QA staff for closure of the NCM.

4.2.6. Submit the form to the supervisor responsible for the area indicated at the top of the form.

4.3. Supervisory Review, Approval, and Signature

4.3.1. Review the information provided. Complete or amend the descriptions of root cause and corrective action. If the corrective action has not been determined, the situation must be referred to the project manager and the operations/systems manager for resolution to ensure client requirements can be satisfied. The QA staff should be consulted if there are questions as to how to proceed. If the above input was not needed, the operations/systems manager does not need to have every NCM routed to him or her. If, upon receipt and review of the NCM by the QA staff, it is felt the operations/systems manager

needs to be made aware of the issues, the QA staff will notify and document the notification of the operations/systems manager on the form.

4.3.2. If the nonconformance is hardware/equipment related, the item shall be nonconformance tagged and segregated, if possible, to ensure that it is not used until repaired.

4.3.3. The supervisor will be responsible for the completion of the corrective action unless otherwise indicated. Enter the name of the person responsible for performing the corrective action if other than the supervisor. This is Operations' commitment to rectify the problem. This will be verified by the QA staff and/or the operations/systems managers.

4.3.4. The supervisor signs and dates the "Corrective Action Approved By" line and forwards the original form to the project manager. The project manager must receive the NCM in a timely manner, generally within 48 hours. If the NCM is for a holding time violation, a project manager ***must*** be notified ***immediately***.

4.4. Project Manager Review, Client Notification, and Project Documentation

4.4.1. The project manager shall determine if client notification is required to either assist in the definition of corrective action or to notify the client of problems related to sample analysis. The project manager shall indicate on the NCM whether client notification is required or not.

4.4.2. Record the result of the client contact or leave blank if client contact was not required. Sign and date the NCM form. This must be done whether client notification is completed or not. The signature documents the project manager as having read and reviewed the NCM.

4.4.3. Document any changes to the corrective action plan, and notify the supervisor. The project manager's signature on the front of the NCM serves as documentation that the PM has read and reviewed the NCM and is aware of the corrective action plan.

4.4.4. The project manager will forward the original to the QA office within 72 hours for review and closure. The PM may keep a copy for his or her own personal use, but the original must be forwarded to the QA office.

4.4.5. If the nonconformance involves analytical work in process, the **final** report cannot be released until a project manager has signed the NCM.

4.4.6. If it is found that erroneous analytical data (e.g., from data validation comments or phone requests, etc.; inaccurate chromatograms, spectra, calculations, or final reports) have been released by the laboratory, this fact must be documented on an NCM (items 25 and 26 on NCM form) and forwarded to the QA office. Prior to making the corrections, proper documentation shall be filled out and turned in to the QA staff if corrections are needed in the database (LIMS or QuantIMS). The original data shall be marked as unusable and maintained for historical purposes. The corrective action shall include prompt client notification and issuance of amended reports.

4.4.7. After the QA staff has closed the NCM, the original of the closed NCM will be filed either in the project file or QA files per laboratory procedure.

4.5. Quality Assurance Review and Tracking

4.5.1. The QA staff shall review all NCMs for conformance with standard laboratory practices.

4.5.2. NCMs will be reviewed to ensure that the corrective action was completed.

4.5.3. A tracking system will also be used to monitor for repetitive failures that might indicate systematic problems. Tracking records would (when applicable) include:

- NCM log number
- Date initiated
- Project number
- Lab sample ID numbers
- Method or parameter
- Nonconformance description and NCM box number
- Corrective action required
- Characterization as an anomaly or deficiency
- Closure of NCM

4.5.4. The QA staff shall identify repetitive quality issues that may be systematic in nature and may require corrective actions to prevent recurrence. Recurrent technical or Information Technology problems shall be referred to the appropriate technical group for corrective actions. Correction of systematic problems could take the form of modifications of nonconforming procedures, repair or replacement of deficient equipment, training or replacement of personnel. Findings and corrective actions from these investigations or audits shall also be documented. Resolution of corrective actions for systematic

problems must be documented by the responsible laboratory area along with supporting evidence.

4.5.5. The QA staff shall conduct follow-up assessments to confirm that correction of systematic problems is successful. These assessments can be done as part of the monthly spot assessment program at the laboratory.

4.5.6. The signature of the QA manager or designee is required for corrective actions to indicate that the issue has been closed.

4.5.7. The QA office shall maintain a central file/record of all NCMs.

4.6. Externally Generated Corrective Actions

4.6.1. Reports from external quality organizations, such as audit reports, data validation comments, and reports of proficiency tests, shall be considered externally generated. The QA staff is responsible for maintaining the files of externally generated quality reports. If the report is received by an associate other than the QA manager, the report (or copy) must be forwarded to the QA Manager.

4.6.2. Preformatted NCMs are not required to be used to document audit deficiencies or performance evaluation deficiencies. A memorandum format notification by the QA staff and responsible supervisor response with corrective action can be used to document the corrective action process.

4.6.3. The QA manager or designee shall review the reports to identify any deficiencies requiring action.

4.6.4. The QA manager or designee shall work with the responsible supervisor to formulate a corrective action plan. The plan shall be implemented and evidence of corrective action supplied to the QA office.

4.6.5. The QA staff shall verify that the problem has been corrected and prepare a written response to the external organization, if required. Data validation responses shall be coordinated through a project manager for the client.

5. DEFINITIONS

5.1. ***Nonconformance:*** an unplanned deviation from an established protocol or plan. The deviation may be the result of Quanterra's® actions, then termed a **deficiency**, or the result of events beyond the control of Quanterra®, then termed an **anomaly**.

A nonconformance exists when:

5.1.1. Any laboratory QC sample (e.g., method blank, laboratory control sample, duplicate laboratory control sample, matrix spike, matrix spike duplicate, and surrogate spike) component result is outside established control limits and demonstrate a **systematic** deficiency. Any matrix spike or matrix spike duplicate or sample related QC outside of established control limits attributed to matrix effects **must** be documented, but an NCM is not required.

5.1.2. A procedure is not performed as described in the applicable SOP or QA Policy, ***except*** in cases where the procedure has been performed according to a client-specified document Quanterra® has agreed to follow (e.g., EPA SOWs and QAPjPs).

5.1.3. A practice or procedure is not performed as described according to a client or project document that Quanterra® has agreed to follow.

5.1.4. Purchased materials or services are determined to be defective and their use would effect data quality.

5.1.5. Holding time violations occur regardless of what or whose actions caused them.

5.1.6. A formal NCM is not required for routine instrument maintenance, malfunctions, and power failures which can be documented in instrument maintenance logbooks.

5.2. ***Corrective action:*** Measures taken to rectify conditions adverse to quality and, where possible, to prevent their reoccurrence.

5.2.1. Corrective actions may vary from reporting the data as is—with appropriate documentation—to a complete reevaluation and restructure of a system.

5.2.2. Many corrective actions can be implemented immediately; however, some will take time to implement.

6. MISCELLANEOUS

6.1. Associated Documents

6.1.1. Quanterra® Quality Assurance Management Plan (QAMP), current revision.

6.1.2. ANSI/ASME NQA-1, Chapter II, Basic Requirement 15 "Control of Nonconforming Items." Supplement 15S-1 "Supplementary Requirements for the Control of Nonconforming Items."

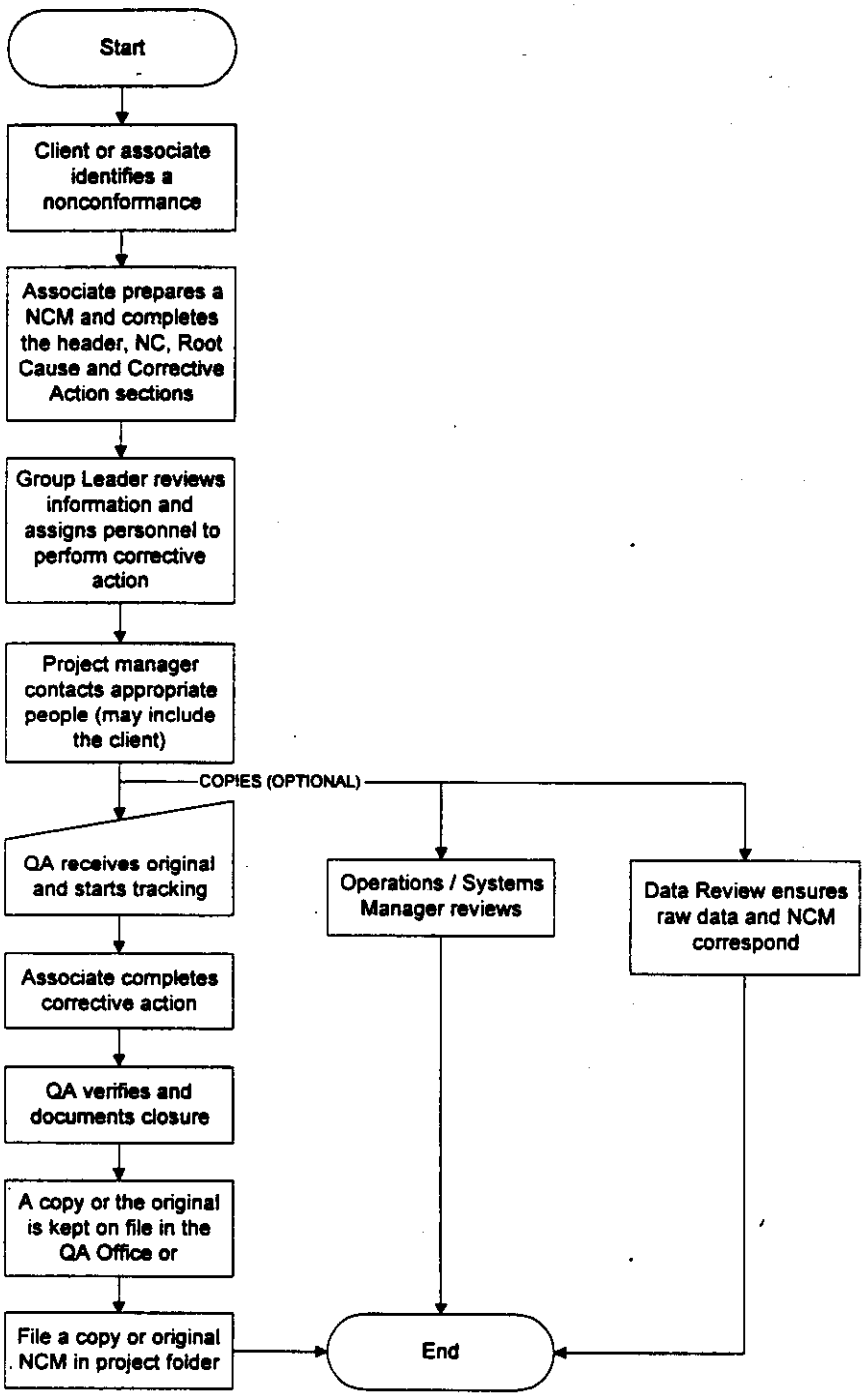
6.1.3. ANSI/ASQC Q94-1987, "Quality Management and Quality System Elements - Guidelines," Section 14.0 "Nonconformity" and Section 15.0 "Corrective Action."

6.2. Appendices

6.2.1. Attachment A: Laboratory Nonconformance Memo (NCM).

6.2.2. Attachment B: Instrument/Equipment Nonconformance Tag Form

6.3. Flow Chart for Internal NCM



ATTACHMENT A LOG #: _____

LABORATORY NONCONFORMANCE MEMO (NCM)

Quanterra® Incorporated

Project ID/Client: _____

NCM Initiated by/Date: _____ Project Manager: _____

Sample Numbers/QC batch or lot numbers _____

Tests: _____

Analytical Area (check appropriate area):

- ❑ Sample control
- ❑ Organic preparation
- ❑ Inorganic preparation
- ❑ GC
- ❑ HPLC
- ❑ GC/MS
- ❑ Wet chemistry
- ❑ Metals
- ❑ Reporting
- ❑ Data review
- ❑ Radiochemistry
- ❑ _____

Nonconformance (check appropriate area): *To be completed by analyst*

Holding Time Violations (exceeded by _____ days)

Category I: Laboratory Independent

- ❑ 1. Holding time expired in transit
- ❑ 2. Sample rec'd > 48 hrs after sampling. or ½ holding time has expired
- ❑ 3. Test added by client after expiration

Category II: Laboratory Dependent

- ❑ 4. Instrument failure
- ❑ 5. Analyst error
- ❑ 6. Log-in error
- ❑ 7. Miscommunication
- ❑ 8. Other (explanation required)_____

_____

Category III: Analysis Reruns (QA/QC)

- ❑ 9. Surrogates
- ❑ 10. Internal standards
- ❑ 11. Spike recoveries
- ❑ 12. Blank contamination

Category IV: Analysis Reruns (Confirmation)

- ❑ 13. Second column
- ❑ 14. Contamination check
- ❑ 15. Confirmation of matrix effects
- ❑ 16. Other (explanation required)_____

Category V: Analysis Reruns (Dilution)

- ❑ 17. Over calibration
- ❑ 18. Under calibration
- ❑ 19. Other (explanation required)_____

Quality Assurance/Quality Control

- ❑ 20. QC data reported outside of controls
- ❑ 21. Incorrect procedure used
- ❑ 22. SOP intentionally modified with QA and tech approval
- ❑ 23. Invalid instrument calibration
- ❑ 24. Received insufficient sample for proper analysis

Incorrect or Incomplete Client Deliverable

- ❑ 25. Hardcopy deliverable error
- ❑ 26. Electronic deliverable error

Reported Detection Limits Elevated Due to:

- ❑ 27. Sample matrix: Does not include high analyte content
- ❑ 28. Insufficient sample volume
- ❑ 29. Other (explanation required)

Miscellaneous

- ❑ 30. Instrument/equipment Tag-out
- ❑ 31. Other (explanation required)

_____

Notification (check appropriate area): ❑ **Required** ❑ **Not Required** *To be completed by project manager*

Client notified by (name and date): _____

- ❑ In writing
- ❑ By facsimile
- ❑ By telephone
- ❑ Other (explain)

Client's name and response: _____

- ❑ Process "as is"
- ❑ Re-sample
- ❑ On hold until _____
- ❑ Other (explain)

Project manager (signature and date): _____

C4-632

QUANTERRA® LABORATORY NONCONFORMANCE MEMO

LOG #: _____

Corrective Action: *To be completed and reviewed by all associates involved*

Problem Description/Root Cause Author's initials and date: _____

Corrective Actions (Short Term) Author's initials and date: _____

Corrective Actions to Prevent Reoccurrence (Long Term) _____

Corrective Action approved by (Supervisor/Group Leader) and date: _____

Additional Comments: _____

Corrective Action to be completed by (if other than Supervisor/Group Leader): _____

Date Corrective Action is to be completed: _____

Quality Assurance Review *To be completed by a QA associate*

❑ Anomaly ❑ Deficiency ❑ Notified Ops/Sys Manager (Initials) _____

❑ Further action required: _____

Further action assigned to: _____

QA signature: _____ Date: _____

Corrective Action Verification: *To be completed by a QA associate*

❑ Verification not required or requested

❑ Verified / CA completed on: _____ by _____

❑ Cannot verify (specify reason) _____

Verified by: _____ Date: _____

Nonconformance Memo Closure:

QA signature: _____ Date: _____

The Office of Quality Assurance maintains a copy or the original of this NCM indicating its final status.

ATTACHMENT B

EXAMPLE

INSTRUMENT/EQUIPMENT NONCONFORMANCE TAG FORM

QUANTERRA®

CAUTION

DO NOT USE

NONCONFORMING ITEM

NCM NUMBER _____

AFFECTED ITEM _____

_____

_____

_____ _____
ANALYST DATE

WORK MAY NOT PROCEED ON THIS ITEM UNTIL SUCCESSFUL CALIBRATION IS DOCUMENTED.

C4-634

APPENDIX D
LABORATORY CUSTODY PROCEDURES

TABLE OF CONTENTS

	PAGE
Custody Procedures for Southwest Laboratories	D-1
Custody Procedures for Quanterra Environmental Services	D1-1

Quanterra
Environmental Services

Controlled Copy
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SOP No. SAC-QA-0003
Revision No. 5
Revision Date: 7/3/96
Page: 1 of 42

OPERATION-SPECIFIC STANDARD OPERATING PROCEDURE

TITLE: SAMPLE RECEIPT AND PROCEDURES (SUPERSEDES: LP-CAL-0007, REVISION 4)

Prepared by: Peter Isaeff

Reviewed by: Technical Specialist, Roger Freize

Approved by: Quality Assurance Manager, Gail Cho Celaschi

Approved by: Environmental Health and Safety Coordinator, Cheryl Ann Stith

Approved by: Laboratory Director, Bruce Warden

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1. PURPOSE

1.1. This SOP describes the procedures for laboratory chain-of-custody, including receipt and acceptance of sample shipments, storage requirements, generation of computer records, and corrective actions for sample receipt anomalies.

2. RESPONSIBILITIES

2.1. Sample Administrator

2.1.1. Sample Administrators are responsible for following the procedures and policies set forth in this SOP.

2.1.2. Primary responsibilities include: sample receipt and acceptance, documentation of sample identity, preservation of custody chains, annotation of discrepancies and obvious anomalies, maintenance of proper sample storage conditions, and creation of computer records which integrate laboratory expectations with specifications from shipment documentation.

2.2. Project Manager (PM)

2.2.1. The PM is responsible for verifying the accuracy of all computer records entered at project creation.

2.2.2. The PM is responsible for providing essential information in order for acceptance to proceed.

2.2.3. The PM is responsible for resolving discrepancies with client, Sample Management Office (EPA), or Contracting Officer in order for work to proceed.

2.3. Laboratory Analysts

2.3.1. Analysts are responsible for ensuring the integrity of the internal chain of custody of each sample they have signed out of or returned to sample control. Refer to Section 4.12.

3. SAFETY

3.1. Procedures shall be carried out in a manner that protects the health and safety of all Quanterra associates.

3.2. Eye protection that satisfied ANSI Z87.1 (as per the Chemical Hygiene Plan), laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

3.3. The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the Material Safety Data Sheets (MSDS) maintained in the laboratory.

3.4. Exposure to samples must be maintained **as low as reasonably achievable**, therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation.

3.5. All subsampling activities will be conducted in a fume hood with the sash closed as far as the operation will permit.

3.6. All work must be stopped in the event of a known or potential compromise to the health and safety of a Quanterra associate. The situation must be reported **immediately** to a laboratory supervisor.

3.7. Safety policies pertain to all Sample Administration visitors, including employees of clients who deliver samples.

3.8. All samples are to be considered hazardous and must be handled according to safety policies and any pertinent SOP.

4. PROCEDURE

4.1. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

4.2. Receiving Shipment

4.2.1. Verify accuracy of each container's delivery address.

4.2.2. Sign bills of lading, if present, including date, time, and custody seal condition. In the event of multiple package deliveries, the receipt time is the carrier's time of arrival, not the time when each package is handled.

4.2.3. Record each project received in the Project Receipt Logbook (PRL). A single shipping container may be listed as one or several projects. Record the client and/or shipper name(s), receipt date and time. If the project number is generated at this time, record the number in the far right column. If a single shipment is subdivided into multiple projects at a later time, all project numbers may be listed on the original logbook entry. The PRL (Example 1) is used to track the flow of incoming work and as a cross reference between shipments and computer records.

4.2.4. All packages received must be entered in the PRL, whether samples are present or not. If the shipment is not a project, enter a comment explaining why no other receipt records were generated (e.g. "empty returned cooler", "technical components", "standards", or "personal").

4.2.5. Initiate a Project Receipt Checklist (PRC) for each sample shipment (Example 2). Each person who completes an entry must also enter initials and date in the corresponding columns. For not applicable items, enter "N/A", with initials and date. The initiator must complete entries A, B, and F-K, unless responsibility for the shipment is immediately passed to another technician.

- Item A: Enter client/shipper name, as entered in PRL.
- Item B: Enter PRL number. PRL number consists of the logbook page number and line number (e.g. 1-1, 1-2, etc.).
- Item F: Enter date shipment is received.
- Item G: Enter time shipment is received.

- Item H: Enter carrier (method of delivery). "Over the counter" indicates a delivery made directly to the laboratory by a client.

- Item I: Enter condition of custody seal (if present). Enter pre-printed number of custody seal or "N/A". Custody seals are defined by the presence of the shipper's signature accompanied with date/time. Note the presence of custody tape (which will have no space for entries and crumbles when removed).

- Item J: Record ownership/disposition of shipping container. Non-company containers must be returned to client if possible.

4.3. Opening Shipment - Prioritizing Workload

4.3.1. Following safety policies, open the shipping container, remove paper work, and check interior condition. Shipments accompanied by MSDS documentation, shipments of concentrated product, or exhibiting strong/noxious odors must be treated with extra care. Open any suspect shipments only after placing the container in hood H1.

NOTE: If a shipment contains broken or leaking samples, place the container in hood H1 and notify the project manager. Proceed with disposal of broken samples in accordance with "Response to Samples Broken Upon Arrival", SOP LP-CAL-0005.

4.3.2. Immediately after opening, ascertain if ice or artificial coolants are present. Measure temperature and record results under Item: K of the PRC.

4.3.3. Note pre-printed Chain-of-Custody (COC) number if a COC is present and possesses a sequential document number.

4.3.4. Use the infra-red thermometer to determine temperature. Measure by directing the thermometer at sample containers. Do not measure the temperature of any cooling agents (ice, dry ice, artificial ice). Do not allow packing to interfere. One layer of plastic, such as a ziplock bag may cause a discrepancy of two or three degrees. [The IR thermometer is calibrated quarterly against a "NIST" thermometer. All filament thermometers are calibrated annually. The calibration documentation resides with the Quality

Assurance Department.] If the COC lists a temperature blank, locate the blank and measure the temperature.

4.3.5. Record result under Item K on the PRC.

4.3.5.1. An anomaly must be entered if the temperature reading is below 2°C or above 6°C. Use one of the anomaly selections available with the LABEL program (Example 6). This notifies the project manager and provides a hard copy to include with the project folder. Some sample matrices do not require cooling during transit. Anomalies need not be filed for these samples and "N/A" may be entered for Item: K. Situations where acceptable temperature range exceedence may be expected include, but are not limited to:

- samples delivered within six hours of close of daily sampling event; an anomaly must be filed in this case
- paperboard and dry pulp samples;
- dry incinerator ashes;
- filters, PUF and XAD cartridges;
- dry product samples.

4.3.6. If the shipment shows any sign of matrix degradation, an anomaly must be filed.

4.3.7. Read accompanying documentation. If documentation is absent and the shipment arrival was unexpected, contact the lead project manager for assistance.

4.3.8. Examine the analyses requested and the sampling dates/times. Be aware of any notation for expected Turn-Around-Time (TAT) or a due date which is less than 14 days.

4.3.9. Most aqueous organic analyses require extraction within seven days of sampling. If three days or less are remaining, consider the project to be a priority and file in an orange folder. [Methods 8280 and 8290 (dioxin/furan analyses) are an exception, requiring extraction within thirty days.]

4.3.10. Any analysis which has a standard holding time of forty-eight hours or less is considered a "Short-Holding Time" (SHT) analysis (Example 3). These are reported to the General Chemistry Department immediately. [Reporting SHT analyses does not require complete entry of project records on the computer.]

4.3.11. Any requested TAT of fourteen days or less is also considered a priority.

4.3.12. Prioritize the shipment according to the TAT, while ensuring extraction holding times have not been exceeded. "RUSH" projects are placed in a red folder, SHT analyses are placed in orange folders, while all other projects are placed in manila folders. If a project with a short TAT also includes SHT analyses, file in a red folder.

4.4. Sample Processing

Select the project with the highest priority:

1. orange folder;
2. red folder;
3. manila folder.

4.4.1. In circumstances where chemists have arranged to extract samples immediately, project TAT is forty-eight hours or less, or SHT analyses will be handled at an arranged time (so that holding times do not expire), some flexibility is allowed in processing the workload.

4.4.2. Read shipment documentation. Ideally, samples are to have possession documented on a Chain-of-Custody form. Chains-of-Custody will identify samples individually by alpha-numeric designators, list sampling dates/times for each sample, requested analyses and document possession. Signatures of possession qualify samples as having been "received under Chain-of-Custody". A Letter-of-Transmittal is also accepted as definitive documentation. Other forms of documentation include Request for Analysis, Shipping Order, Purchase Order, and various computer listings of sample information. If no documentation which lists sample identifications exists, complete a COC when accepting the samples.

4.4.3. Remove the samples from the shipping container. If the temperature has not been measured, do so and record under Item: K, as in Section 4.3.4. If a

temperature blank is discovered during unpacking, measure it and record under Item K also.

4.4.4. Align containers from left to right in rows, with additional containers for any sample placed behind the first in columns.

4.4.5. Check the samples against the COC for accuracy (e.g., sample ID, collection date/time, etc.). If the samples have been received in good condition, document this on the COC. All discrepancies must be noted as well, including the lack of relinquishing signature of the shipper.

NOTE: Good condition is loosely defined as all containers intact with no obvious discrepancies present. The shipment is estimated at this time as viable; what is being requested coincides with what has been received. Temperature exceedences and minor discrepancies become issues when so specified by contract or client instruction. The presence of bubbles in volatile containers is documented. Head space present in organic lead samples originating from U.S.Navy Program work is anomalous and must be documented on custody chains, the PRC (Item: Q), and by using the Anomaly option of the LABEL program.

4.4.6. Complete the bottle inventory on the back of the project receipt checklist (Example 5).

4.4.7. Compare the containers and their state of preservation with the list of analyses requested, if present on receipt documents. Although some exceptions are permitted regarding particular clients, container types must compare to the Quanterra QA (Quality Assurance) Plan. Discrepancies of this nature must also be annotated on the COC and an anomaly filed.

4.4.8. Assume custody of the samples by signing and dating the COC in the section marked "Received for Lab By" or "Received By". For documentation that is not very specific, specify what was received. [Example: Two solid product samples received in good condition.] Complete Item: P and/or Q.

4.4.9. Affix a color dot to the lid of each sample container. The color corresponds to the receipt week on the disposal calendar. The dot color aids in sample rotation.

4.4.10. For all work sampled at or concerning government property, or as specified in client QAPjPs, the pH of the preserved aqueous samples must be checked and recorded except for the VOA vials. The pH is noted as <2 or >12 with the preservative type following. [Example: pH<2 H2SO4).

4.4.10.1. Ascertain the pH by inverting the sample container three times, removing the cap and inserting a clean pipette into the sample container, removing a small amount. Release a droplet of the aliquot on a fresh piece of pH indicator paper. Compare against the pH Color Grid Table. Do not reuse the pipet. Use a clean pipet for each container. Recheck readings that indicate samples were unpreserved when they should have been. Note discrepancies on the COC. Notify the Project Manager and file an anomaly for the concerned sample.

4.4.10.2. Transport samples to the General Chemistry or Metals Departments when sample preservation is necessary as a corrective action. Complete Item: L

4.4.11. Referring to Section 4.7, obtain a project number.

4.4.12. Generate labels for the sample containers on the computer terminal next to the label printer. The user name and password is "labels". The LABELS program will automatically come on the screen (Example 6).

4.4.13. Some usage is necessary to understand LABELS. One must have pre-selected the storage unit location, and know the Project Manager and appropriate disposal codes before use.

4.4.14. To designate a storage location, use the following guide:

- VOA(V) - volatile containers awaiting transfer to the VOA group refrigerators and organic lead samples received under the U.S.Navy Program.
- W1 through W3 - environmental soil samples;
- W4 through W10, W17 through W26 - environmental aqueous samples;

- W11 through W13 - Advanced Tech aqueous and solid samples;
- W14 - incoming/outgoing shipments;
- W15 - product/known hazard/air toxics/oversized containers (Specialty) samples;
- W16 - extended storage, disposal codes exceeding thirty day retention time;
- F2 - any samples requiring freezing, including plant/animal tissues;
- R2 - any samples where secondary containment in ziplock bags is deemed necessary due to smells, spillage, degradation of container exterior;
- C1 - dry pulp/paper samples;
- EPA1 - soil samples received from the Environmental Protection Agency or clients requiring dioxin work under SOW DFLM01.1.
- WR1/WF1 - refrigerator/freezer storage dedicated to Explosives & Specialty Chemicals projects.
- MA - room temperature storage for aqueous samples (excluding Hg, CRVI, Organic), located in Metals Department and dedicated to U.S.Navy Program projects.

NOTE: Location selections (specify shelf by letter, Example: W4A).

4.4.15. Label sample containers. Be certain the labels adhere to containers. Attempt to leave all client label information exposed. However, when impossible to do so, affix label so that at least the client ID, sampling date/time, and preservative are showing. Preprinted laboratory names may be covered with no consequence. If label adhesive is insufficient, use cellophane tape to secure label. Place sample in ziplock bag when storage location is R2.

4.4.16. Initial/date the PRC (Item: M).

NOTE: U.S.Navy Program requires that each bottle for each sample be designated with an alpha character. The first container for the -001 will be "a", the second "b", etc. Sample -002 will repeat with "a","b",etc.

4.4.17. Project/sample labeling may be peer reviewed whenever the complexity of the project warrants a second check to ensure accuracy. A peer compares the order of the samples to the documentation and ensures that all containers of a sample have the same sample number and are sequential. The reviewer then initials/dates the PRC (Item: N). If no review is performed, the labeler completes line N.

4.4.17.1. Circumstances where a review is warranted include:

a) a large number of samples are present;
b) many containers per sample are present;
c) VOA containers are present;
d) client identifications are illegible or confusing:
e) a review is requested by a Quanterra employee

4.4.18. Anomalies can be filed using the LABELS program. Shipment discrepancies as well as temperature exceedences must be conveyed to Project Managers promptly. Holding-times that expire prior to receipt or result due to half the holding-time expiring prior to receipt require documentation using the ANOMALY Program (Example 7). Holding-time violations can only be entered after LIMS entry is complete.

4.5. Shipping Container Return

4.5.1. Containers in which sample containers have broken or in which sample volume has mixed with packing material or melted ice must be handled according to LP-CAL-0005, Response to Samples Broken Upon Arrival.

4.5.2. Except when discarded, shipping containers are cleaned in Sample Administration and either taken to Bottle Prep or sent to the client. Containers belonging to Quanterra Incorporated will be marked in felt pen with an easily identified tracking number. If the container belongs to a client, be certain the return address is recorded before stripping the container of used tape and labels.

4.5.3. Strip the container of tape and shipping labels. Older containers may be covered with aged tape and labels that are not easily removed. Do not spend excessive amounts of time with these. All hazardous materials labeling must be removed or defaced in some way. Courier/shipping companies consider intact hazard labels valid, and will refuse to carry containers displaying hazard labels unless accompanied by proper documentation.

4.5.4. Blue foam inserts can be placed in a hood or on a counter to dry when received wet. They may also be stored in the warehouse outside Bottle Prep if necessary. If the inserts are dry they may be returned to Bottle Prep in the container after it has been cleaned.

4.5.5. Dry the interior of the container if wet, replace packing and return to Bottle Prep. A cart is located in the room for transporting containers. Place the container on the cart. If the cart is full, take the entire cart load to Bottle Prep. Transfer load from cart to storage room.

4.5.6. Client containers are to be returned in as satisfactory condition as possible. Packing/artificial ice and temperature blanks are returned to clients. Packing which resembles trash or is ruined during unpacking is disposed as trash. Packing which is deemed re-usable may be returned also. Drain any water from the container before sealing it closed. Due to partially frozen artificial ice, client containers may not be perfectly dry when returned.

4.5.7. Complete a return mailing label. Secure it with cellophane tape and seal the container with tape also. Return packages are listed in the UPS Logbook and set near the door. Be certain to complete both address labels and the UPS shipping logbook, including weights and zone codes.

4.5.8. When ice is present, it is often enclosed in plastic bags. A basin used for dumping ice is located in Sample Administration. Open the bags and dump ice into the basin. Draining into a closed system, water may not be left running in the basin. When excessive amounts of ice are received, collect the ice in an ice chest and dump it over the storm drains along the sidewalk outside the building. **This is permissible only if the ice and coolers are uncontaminated and the amounts disposed in this way are recorded.** A

logbook located in Sample Administration records the date/time, person and amount of ice.

4.6. Client Deliveries ("Over the Counter")

4.6.1. Samples received over the counter are hand delivered by the client/sampler or by a courier directly employed by the client's company. The pivotal question in such deliveries is whether the person possessing the shipment is in custodial possession of the samples or merely transporting the shipment. If in custodial possession, the deliverer will wish to relinquish custody to you before departing and will probably retain a copy of the documentation. To determine the matter, simply ask the deliverer if he/she had signed any of the sample documents.

4.6.2. If the "client" does not wish to remain while the samples are unpacked and verified, sign the COC somewhere other than the "Received By" section. Include a statement that only the shipping container was received. [Example: One cooler received from Company X in good condition. MMDDYY/ Initials]

4.6.3. Generally, the client/sampler will wish to remain, have the samples verified and relinquish custody to this laboratory. Follow Sections 4.2 through 4.4 as applicable. [Over the counter receipts are a condensed, abbreviated form of standard delivery practices. As such, they require more skill and expertise to accomplish necessary tasks without prolonging the client's involvement beyond what is necessary.]

4.6.4. Mention any discrepancies and allow corrections to be made by the client/sampler. Also ask for any special instructions or important aspects of the project (i.e. rush turn-around times, impending hold-time violations, sample matrix specifics, etc.).

4.6.5. Solicit the client's wishes regarding shipping container and/or any other needs, such as bottle order pick-up or a need to speak with a project manager.

4.6.6. After the client's departure, complete any aspects of Sections 4.2 through 4.4 left unfinished. Prioritize the new project within the existing workload and proceed with the highest priority.

4.7. Project Generation

4.7.1. Log on to the VAX/VMS computer system and enter the Laboratory Information Management System (LIMS) by typing: LIMSMENU <R>. For the basic use of the LIMS system, refer to the LIMS user manual.

4.7.2. Projects can be created using a variety of resources. Minimal project login consists of the Project Screen (pages 1 and 2), Sample List and Group Codes (Example 8). Frameworks for new projects may be dictated using instructions from project managers, copying templates or existing projects.

4.7.3. If project information is not available and a project must be setup from scratch, determine the name and location of client. This information can be found on the COC, shipping labels, or obtained from the project manager.

4.7.4. Pull up the appropriate client screen.

4.7.5. If the client does not exist in the database, a new one must be created using the protocol in the LIMS user manual. New JDE IDs (for billing) need to be requested of corporate by the project manager and are not necessary to complete login at this time.

4.7.6. Search the programs listing of the client and choose the appropriate one for the project. If one does not exist, confer with the project manager whether a new program needs to be created or not.

4.7.7. Once the appropriate program screen is linked with the client, search the project listing for a template or similar project. If none is found a project must be created.

NOTE: Duplication of templates is the most desirable method of project set up as the template contains all necessary information needed to accurately create a project.

4.7.8. The necessary LIMS fields that must be filled in are as follows:

- Project name: follow the naming procedures used for previous projects belonging to the same client. If none exist, list the client's project name and number, and the date.
- Matrices and brief analytical list may also be added if room is available in the data block.
- P.O. number: if no P.O. number is available, list LIMS contact name (i.e. D.Client).
- Description: list the matrix type and analyses requested. Also note anomalies, special requests, and/or any hazards of which analyst(s) should be aware.
- Contact: enter LIMS ID of contact, the person who will receive the final report.
- JDE ID: J.D.Edwards ID of company and/or individual who meets credit requirements.
- Mail To: Address to which invoice is mailed. This may be identical to JDE ID, or completely different. Third party billing and larger companies often use separate address.
- Team ID:
  - 11 - Environmental Department
  - 40 - Advanced Tech, Dioxins
  - 70 - Energetics
  - 561- Air Toxics
- Calculate holding times from collection dates?: If the answer is Yes(Y), LIMS will calculate all analytical holding times from the collection (sampling) date. If the answer is No (N), then LIMS will calculate the holding times from the date of receipt to the laboratory. At present, only projects requiring CLP protocol will have a "No" answer.

- Primary PA: the project manager who is responsible for daily monitoring of project.

- Turnaround Class:
  - NORMAL - Standard Deliverable;
  - RUSH - Faxed Deliverable;
  - EXTENDED - Custom Deliverable (e.g., CLP, IRPMS, or Raw Data).
  - [If unknown, default is "NORMAL".]

- Promised Turnaround Time: enter number of calendar days in order to produce the report one day before "Due Date" specified by client on COC. Default is "20" for NORMAL TAT.

- Actual Start Date: date from which the project TAT is calculated. The project has met all sample delivery acceptance criteria as stated in the Quanterra terms and conditions.

- Receipt Date and Time: actual date and time the shipment arrived at the laboratory.

- Storage location: unit/shelf where samples are stored until rotation or disposal. See listing in Section 4.4.15.

- Comments: short description of the number of samples, matrices, condition upon receipt, delivery method and whether the samples were received under chain of custody. [Examples: Two aqueous samples received under Chain-of-Custody in good condition. Delivered by Federal Express. Soil samples (103) received in good condition, listed on purchase order. Delivered by client.]

4.8. Sample Login

4.8.1. Refer to the LIMS manual section for entering sample information into LIMS.

4.8.2. Creation of group codes depends upon information available when the samples arrive. Each group code lists analyses to be entered against a particular sample. Group codes are constructed to the best of the technician's ability, but final responsibility lies with the Project Manager.

D7 116

4.8.3. The Sample List is a series of Sample Detail screens. Each screen records the information for a particular sample. Each screen records client identification, sampling date/time, receipt date/time, due date and container types. This screen does draw information from other screens, but must be individualized to the sample it describes.

NOTE: Sample screens may also indicate a formal record of quality assurance aliquots extracted in order to monitor occurrence of contamination. Dioxin projects always have one method blank logged per matrix type (Example 8). U.S.Navy Program projects require a "VOA Holding Blank" assigned to every SDG (Sample Delivery Group) received.

4.9. Folder Assembly: Use an appropriately colored folder (Red - Rush, Manila -Normal) for project information and manila/any color for sample tracking and complete as follows:

4.9.1. Project Manager Folder

4.9.1.1. Staple the air bill, secondary documentation, the original COC and a ticket report in reverse order to the left hand side of the folder. Tab with project label is to be on right hand side.

4.9.1.2. In reverse order, place the following on the right-hand side of the folder:

a) Project Review Checklist #1 (completed);
b) Data Control Instruction Sheet (Example 9);
c) Ticket Report (for projects with method blank aliquots listed);
d) Sample List with Group Code List underneath (stapled together);
e) Duplicate copy of COC stapled underneath a Ticket Report;
f) Project Receipt Checklist (completed).

4.9.2. In-House COC Folder Assembly

4.9.2.1. Align folder as with the project folder. Tab is on right-hand side.

4.9.2.2. On the right-hand side of the folder, staple the In-House COC form QA188 (Example 10).

4.9.2.3. On the left-hand side, staple a Ticket Report. If the entire bottle inventory for any sample is not complete on the ticket report, include a copy of the bottle inventory (reverse side of PRC).

4.9.2.4. If an MSDS is provided in shipment, staple a copy beneath the ticket report on the right-hand side of the folder.

4.10. Final Tasks

4.10.1. Record project number in PRL on the line corresponding to the shipment's arrival.

4.10.2. Complete Project Review Checklist #1 QA-235 (Example 11).

4.10.3. File an HTV, if necessary, by entering "Anomaly" and the project number at your prompt. Answer the queries, and be certain to mail the HTV to the department supervisor and project manager involved. The HTV is filed by Sample Administration when a holding time is expired upon receipt. If the holding times have not completely expired for a project, the department handling the analysis in question files the HTV.

4.10.4. Place the Project Manager Folder in the project manager's tray and the Internal Tracking Folder in the check-out boxes (both located in Sample Administration).

4.11. Sample Storage

4.11.1. Refrigerator and freezer temperatures will be monitored and recorded once daily Monday through Saturday. Any temperature below 2°C or above 6°C for the refrigerators and above -10°C for the freezer will need corrective action. Refer to "Corrective Action for Refrigerators Out of Acceptable Temperature Range", SOP LP-CAL-0014.

4.11.2. Temperatures will be recorded on the appropriate charts (Example 12). Temperature charts will then be stored in the temperature logbook. All charts for a particular refrigeration unit will be stored together in chronological order.

4.12. Internal Sample Tracking

4.12.1. Analyst selects sample check-out folder for the project of interest and ascertains storage location.

4.12.2. Analyst removes appropriate containers from storage unit and organizes them by project/sample number.

4.12.3. Analyst completes check-out portion of Internal COC and has the record and container labels verified by another Quanterra employee (Example 9). The witness must initial the internal chain of custody. Any laboratory personnel may verify the transaction. If the containers are not to be returned and will enter the wastestream through an extraction lab, the analyst will also enter this information during check-out.

4.12.4. Analyst files the check-out folder before leaving the room.

4.12.5. At return, the analyst completes the check-in portion of the Internal COC.

4.12.6. Analyst returns samples to their storage location.

4.12.7. If subsampling or aliquoting is performed, whether outside the area or not, containers must be checked out by the analyst. The original containers' whereabouts and those of any other containers generated must be documented.

4.12.8. Transfer of volatile containers from VOA to RD and RF do not require verification.

4.12.9. Empty containers are not returned to Sample Administration.

5. DEFINITIONS

5.1. Refer to Glossary in the Quanterra QAMP.

5.2. Chain of Custody (COC): A system of documentation demonstrating the physical custody and traceability of samples.

5.3. A sample is considered in your custody if the following conditions are met:

5.3.1. The sample is in your possession.

5.3.2. The sample is in your view.

5.3.3. The sample was in your possession and was returned to a secured area.

5.3.4. The sample was placed in a secure area accessible to authorized personnel only.

5.4. HTV: Holding Time Violation

6. MISCELLANEOUS (TABLES, APPENDICES, ETC...)

6.1. Ensure client confidentiality at all times, giving special attention during facility audits conducted by clients and regulatory agencies.

6.2. Procedural Additions for Receipt/Acceptance of EPA sample shipments.

6.2.1. Shipment documentation should also include an official EPA Traffic Report, if it has not been incorporated with the Chain-of-Custody. The Traffic Report (TR) is the equivalent of the COC and is to be handled identically.

NOTE: The lowest alpha-numeric identification is used to designate the Sample Delivery Group (SDG). The SDG will be used to identify the twenty samples that are received during the sampling event. A single SDG may be received at one time, or several groups may arrive over a short period of time. If so, every twentieth sample will complete the on-going SDG.

6.2.2. Sample identification also consists of an EPA Sample Tag, which will be fastened to each sample container, either by string/wire ties or by tape. Verify the preprinted tag number against the COC and/or TR listing. Remove each tag and seal inside a ziplock bag. Note the Quanterra project/sample number on the bag as well as the SDG designation. The bags containing the tags are included within the Project Manager Folder.

6.2.3. Volatile holding blanks, if received with a shipment, are labeled with the project number of the applicable SDG and are stored with the samples. Holding blanks will record exposure of samples to contamination and must accompany them during storage. Do not separate holding blanks by assigning them to "V" (VOA).

6.2.4. Shipment anomalies must also be recorded on the DC-1 or EPA/CLP form (Example 13). The DC-1 is generated by the EPA, while the EPA/CLP is Quanterra specific. Each records receipt conditions and discrepancies with associated shipment documentation. Both forms coincide with the PRC, except that sequential airbill/waybill numbers are recorded and therefore must be verified. The completed form is included in the Project Manager Folder, stapled beneath the COC/TR on the left side.

6.2.5. Calculate holding times from collection dates? The answer is "No", according to protocol requirements.

6.2.6. Turnaround class is "EXTENDED" as EPA reports are due thirty-five days from the actual start date. A TAT of forty-five days applies under DFLM01.1 (dioxins).

6.2.7. Actual start date is entered with the completion of an SDG. The actual start date may be postponed pending the resolution of discrepancies between the laboratory and the Sample Management Office (SMO).

6.2.8. All EPA samples and empty containers are to be stored at the laboratory for no less than sixty days following the submission of the respective report. Insert the "DISP-60" status on the Group Code screen during LIMS records creation (Example 8).

EXAMPLE 1

QUANTERRA - West Sacramento
SAMPLE RECEIVING LOG

EXAMPLE 1

Init	CLIENT	DATE	TIME	PROJECT#	COMMENTS	
						1
						2
						3
						4
						5
						6
						7
						8
						9
						10
						11
						12
						13
						14
						15
						16
						17
						18
						19
						20
						21
						22
						23
						24
						25
						26
						27
						28
						29
						30
						31
						32
						33
						34

EXAMPLE 2

Quanterra Incorporated - West Sacramento
PROJECT RECEIPT CHECKLIST

EXAMPLE 2

Client Name: ____ Item: A ____ Log #: B ____

Project # (LIMS ID): ____ C ____ Project name: D ____

Lessee(s): ____ E ____ Initials ____ Date ____

Date Received: ____ F ____ Time Received: ____ G ____ ____ ____

Delivered by: H ☐ Federal Express ☐ Airborne
☐ Courier Express ☐ DHL
☐ White Cotton Delivery ☐ UPS
☐ Over the counter (OTC) ☐ Go-Getter
Other: ____

Custody Seal Status: I ☐ Intact ☐ Broken ☐ N/A ____ ____
Custody Seal Number(s): ____

Shipping Container(s): J ☐ Quanterra ☐ Client ☐ N/A ____ ____

Temperature Record (in °C): K
COC#: ____ ____ ____
Temp Blank: ____
Ambient Temp: ____

pH Measured: L ☐ Yes ☐ Assumed ☐ N/A ____ ____

Sample(s) Labeled By: M..... ____ ____

Sample Labeling Checked By: N..... ____ ____

Short Hold Time Notifications: Sample Receiving ☐ N/A ____ ____
O Wet Chem ☐ N/A ____ ____
Metals (Pb/Fm) ☐ N/A ____ ____

P ☐ Complete shipment received in good condition, with appropriate temperature, containers, and preservatives. ☐ N/A ____ ____

☐ Assumed (-ion)/exceptions: ☐ Temperature exceeded (>C-4°C) ____ ____
Q ____ ☐ PM notified ☐ N/A

Leave no space blank. Use "N/A" if not applicable. Initial and date all "N/A" entries. QA105 02/96 MCD

D723

EXAMPLE 3

SUBJECT - Short Holding Time Analysis EXAMPLE 3

INORGANICS - AQUEOUS SAMPLES

Parameter	Method Number	Holding Times From Collection
pH	150.1	ASAP
Coliform	909A/909C/SM9221	*ASAP
Sulfite	377.1	*ASAP
Chromium III/VI	7196 (water & soil)	24 hours
Chlorine, Residual	330.4	*24 hours
BOD	405.1	*48 hours
Color	110.2	48 hours
Nitrate	300.0/353.2 (unpreserved)	48 hours
Nitrite	300.0/353.2 (unpreserved)	48 hours
Odor	140.1	48 hours
Orthophosphate	300.0/365.3 (unpreserved)	48 hours
Settleable Solids	160.5	48 hours
Surfactants (MBAS)	425.1	*48 hours
Turbidity	180.1	48 hours
AOX	9562	3 days
Sulfide (Waters)	376.2/9030A	7 days
Total Dissolved Solids (TDS)	160.1	7 days
Total Suspended Solids (TSS)	160.2	7 days
Total Solids	160.3	7 days
Total Volatile Solids	160.4	7 days
Alkalinity (all types)	310.1	14 days
Cyanide	335.2/9010	14 days
Free Cyanide	412M(STD)	14 days
Sulfide (Soils)	9030A	14 days

* These analyses not performed at this facility.

ORGANICS - AQUEOUS SAMPLES (Except CLP samples)

Volatiles	8010/8020/8240/8260	7 days
OC Pesticides/PCB's	8080	7 days
Polyaromatic Hydrocarbons (PAH)	8310	7 days
OP Pesticides	8140	7 days
Phenoxy Acid Herbicides	8150	7 days
Semivolatiles by GC/MS	8270	7 days
Volatiles	8010/8020/8240/8260(preserved)	14 days

All organic analyses on soil samples (except CLP samples) require extraction within 14 days after collection. An exception is that soil samples for TCLP-8270 must be TCLP extracted within 7 days of sampling.

ORGANICS - CLP AQUEOUS SAMPLES (Holding Times calculated from receipt date)

OC Pesticides/PCB's	8080	5 days
Volatiles	8240/8260	10 days
Semivolatiles by GC/MS	8270	5 days

ALL CLP organic analyses on soil samples require extraction within 10 days after receipt.
Dissolved Metals - if unfiltered or unpreserved -- ASAP!
Remember to add the P-FILTM or P-FILTPRESM tests in LIMS if the samples need to filtered and/or preserved.

EXAMPLE 3 (cont.)

EXAMPLE 3

Quanterra Incorporated - West Sacramento
SHORT HOLDING TEST NOTIFICATION

CLIENT:_____ ☐ SHT ANALYSIS

PROJECT ID:_____ ☐ PENDING HTV

DATE:_____ ☐ RUSH (24-72 Hr TAT)

TIME:_____ ☐ SEE ATTACHED

CHEMIST NOTIFIED:_____ QAS#_____

GENCHEM ANALYSIS:	SAMPLE #:	ASSIGNED CHEMIST:
☐ pH ☐ ALK	_____	_____
☐ C/S ☐ EC	_____	_____
☐ TURB ☐ COLOR	_____	_____
☐ SS ☐ SO3	_____	_____
Lachat		
☐ NO2+NO3-A	_____	_____
☐ NO2-A	_____	_____
☐ NO3-A	_____	_____
Method 365.3		
☐ OPO4	_____	_____
Method 300.0		
☐ NO3 ☐ NO2 ☐ OPO4 ☐ BR ☐ CL ☐ SO4 ☐ F	_____	_____
☐ OTHER: _____	_____	_____
_____	_____	_____
_____	_____	_____

HOLDING TIME ALREADY VIOLATED? ☐ YES ☐ NO

HTV FILED? ☐ YES ☐ NO ☐ N/A

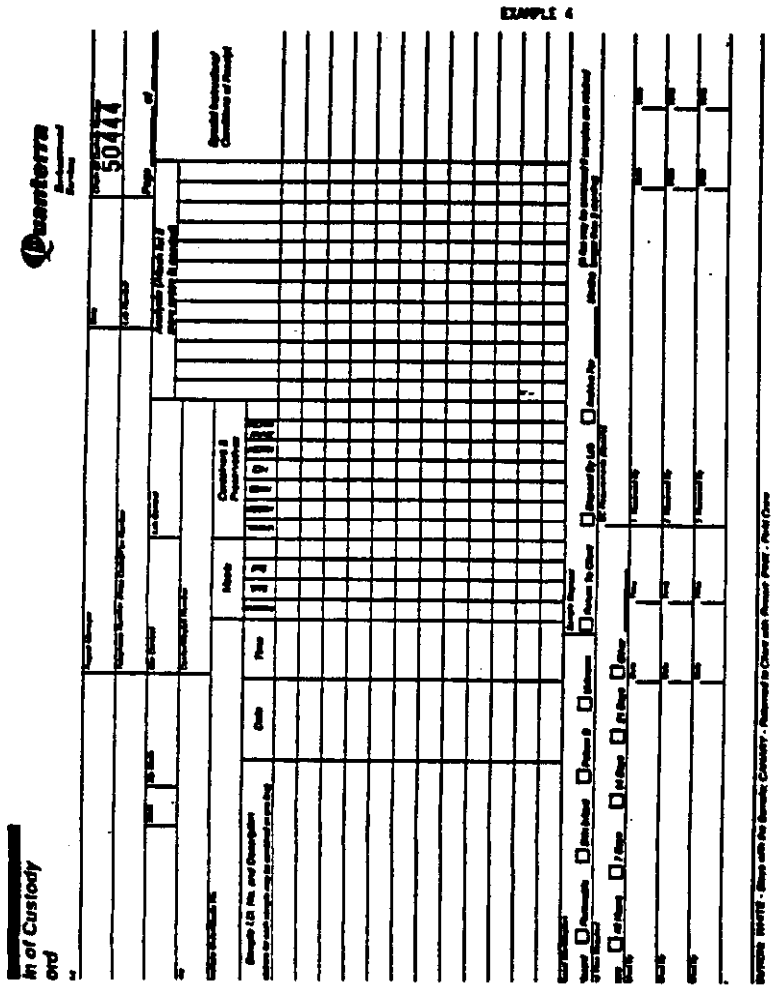
☐ MS/SD ☐ MS/DU ☐ CLIENT REQUESTED (Sample#_____) ☐ N/A

COMMENTS:_____

_____

9/96 MCD QA-300

EXAMPLE 4



EXAMPLE 5

EXAMPLE 5

	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20
VOA																				
VOAs																				
AGB																				
AGBe																				
250AGB																				
250AGBe																				
250AGBc																				
250AGBns																				
AGJ																				
500AGJ																				
250AGJ																				
CGJ																				
500CGJ																				
250CGJ																				
125CGJ																				
PB																				
PBe																				
PJe																				
500PB																				
500PBe																				
500PBns																				
500PBns/ns																				
250PB																				
250PBe																				
250PBns																				
250PBns/ns																				
CT																				
Folder/Filter																				
PUF																				
PETRI																				
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20

EXAMPLE 6

LABEL PROGRAM EXAMPLE 6

```
CTRL-Y to exit
Make you selection to print labels

Just the label ma'am!!

0.  Send Shipment Anomaly
1.  For series of samples
2.  For one sample
3.  For specified set of sample #s (up to 25)
4.  For specified starting and ending sample
5.  For set of Custody Seals

Enter your selection:
```

[The most common usages of the selections are explained below.
Most selections include shipment anomaly queries.]

0. Asks if shipment was received two and six degrees Celsius. Also provides an edit screen for messages. Sends E-mail to Project Manager and prints hardcopy on SAMPRINT.
1. Prints title labels for Project Manager and Internal Sample Tracking folders. Creates the same number of labels per sample. for each sample beginning with -001. "Notes" query may be used for disposal codes. hazard cautions or accelerated TAT.
2. Prints a number of labels per sample number selected.
3. Similar to selection 1. but allows the number of labels to vary per sample number selected. The link between queries is linear. If 1.5.6.9 and 2.3.4.5 are entered. two labels for sample -001. three labels for sample -005. etc are produced.
4. Allows for the same number of labels to be printed per sample. for sequential samples between the beginning and ending samples selected. (Example: four labels for each sample between -045 and -057).
5. Prints a number of labels which read "Custody Seal".

EXAMPLE 6 (cont.)

LABEL PROGRAM (continued) EXAMPLE 6

```
Selection 1:

Enter project#:  08726J
Enter location(s) of sample(s):  W1A
Enter Notes (ie. Hazard info. NS/SD. Rush):  HAZ-MAT
Enter # of samples:  4
Enter # of labels:  3
Enter the client (max 15 chrs):  Quanterra-Macinaw Textile
Any shipment anomalies? (y,n)  ["y" will initiate E-mail option]
Print Labels (y,n):
Can project be rotated?:       [refer to disposal code listed in LIMS]


Anomaly Option:

Enter PM:  KVerrue
Was temperature out of range (acceptable is 4+2 )? (y,n): y
Enter temperature(s): 12
Enter affected sample(s): -001.2.3.4

SHIPMENT RECEIPT ANOMALY(-IES)

            CLIENT:  Quanterra-Macinaw Textile
           PROJECT:  08726J
   PROJECT MANAGER:  KVERRUE
AFFECTED SAMPLE(S):  -001.2.3.4
    TEMPERATURE(S):  12

        RECEIPT ANOMALY(-IES)

[Any length message recording poor or unusual shipment
 conditions may be entered here.
```

EXAMPLE 7

```
ANOMALY PROGRAM                                          EXAMPLE 7

[Anomalies may be filed only after login is complete.  Samples must
 have analyses logged against them in order to operate this program.]

      +--086803_INITIAL_MENU-------+
      | A. Anomaly Report          |
      | H. HTV Report              |
      |                            |
      | X. EXIT Program            |
      +----------------------------+
      |  : Enter Selection         |
      +----------------------------+


+--086803_DEPT_MENU------+
| A. Metals              |
| B. Metals_Prep         |
| C. GCMS                |
| D. Special_Services    |L_MENU-------+
| E. Wetchem             |oft          |
| I. Data Control        |             |
|                        |             |
| X. Exit Program        |m            |
+------------------------+-------------+
|  : Enter Selection     |tion         |
+------------------------+-------------+

--086803_HTV_OPTIONS_MENU-----------------------------------+
|                                                           |
|    Choose HTV Category from List                          |
|                                                           |
| A. Holding Time Expired in Transit (field to lab).        |
|                                                           |
|    Original Analysis Performed within HT: reprepped or    |
|    reanalyzed outside of holding time because:            |
| B.      Internal QC Out.                                  |
| C.      Matrix QC out.                                    |
| D.      Other.                                            |
| E. PA or Log-In Error.                                    |
| F. Capital Resource / Priority Conflict.                  |
| G. Labor   Resource / Priority Conflict.                  |
| H. Instrument Failed.                                     |
| I. Analysis Error.                                        |
| J. Scheduling Error.                                      |
| K. Other.                                                 |
|                                                           |
| X. EXIT Program                                           |
+-----------------------------------------------------------+
|  : Enter Selection                                        |
+-----------------------------------------------------------+
```

EXAMPLE 7

ANOMALY PROGRAM (continued) EXAMPLE 7

[Selection "A - Holding Time Expired in Transit (field to lab)." is generally used. and requires completion of the following screen.]

```
PROJECT ID        : 086803
PA                : GILDERSLEEVE
CLIENT            : STONE CONTAINER CORPORATION
AREA DIRECTOR     : MORBANOSKY
AREA SUPERVISOR   : JJONES  (current supervisor)
REPORTED BY       : CDUNCAN
ANALYSES          : PH-A
DATE SAMPLED      : MMYYDD
SAMPLES AFFECTED  : ALL
DATE HT EXPIRED   :       (calculate from COC or LIMS)

DATE PREP/ANALYSES
ACTUALLY PERFORMED:

REASON:

1. Holding time expired in transit (field to lab).

EXPLANATION:

[Record any pertinent circumstances concerning this HTV.]
```

[Edit screens can be terminated by CTRL Z. Enter "exit" to store/send or "quit" to avoid saving.]

EXAMPLE 8

```
                                                                    EXAMPLE 8
Quanterra, Inc.                    Quanterra Environmental Services - Sacramento
Date: 15 APR 96          RBONALY                    Sample List Screen Print Report
                                                                          Page  1

                                 PROJECT (Page 1 of 2)
Client: CALLAB-QUECA01  Name: Quanterra, Inc. - Sacramento
Program: CALLAB-003450  Name: Advanced Tech (LR) - General Program
Project: CALLAB-08726]  Name: [redacted]ASH TCDDDF 041596
Type: NONE          Case:          SDG:          SAS:          Level:
PO #: 122623                                                Invoice #:
Description: Ash samples: TCDDDF and 8080(PCB).

             Composite containers of each sample prior to analysis.
             Results by dry weight.

             -003: sample containers cracked in transit, volume transferred.


  Contact: CALLAB-[redacted]  Dattz        (414)272-0209
Alternate:        -        -                 |   |   -
 CC: Copy:        -                          |   |   -
   JOE Id: CALLAB[redacted]  ACCT PAYABLE (414)272-0209
   Inv To: CALLAB[redacted]  ACCT PAYABLE (414)272-0209
Project Status: ACTIVE   Status History: AB                          QASF
Team Id: 0040  -CAL High Res        Market Segment: AT- Advanced Tech
  Results to client computer? N                     Deliverables:
     Phone results to client? N                 Client Project ID: MACINAW
Other Quanterra lab involved? N            Project Duplicated from:
      Subcontractor involved? N                       Sales Exec: EMAHSELL
Calculate holding times from collection dates? Y      Primary PM: KVERRUE
   Delivery Penalty: NONE                           Turnaround Class: NORMAL
 Last Followup Date:                          Promised Turnaround Days: 20
  Actual Start Date: 15 APR 96                     Next Followup Date:
   Project Due Date: 05 MAY 96                     Expected Start Date: 15 APR 96
       Receipt Date: 15 APR 96 Time: 10:00         Storage Location: WIA
Comments: Four incinerator ash samples received under Chain-of-Custody in good
          condition. Delivered by Express Western Delivery.
     Log Released:                                Invoiced:
 Preliminary Data:                                Reported:
Delivery Accepted: RBONALY      15 APR 96 10:30 Reviewed:
          Created: RBONALY      15 APR 96       Modified: RBONALY      15 APR 96

 Smf  Grp Alq  Rec Date  Due Date   Matrix  Status    Client Description   Inst
 0001 B * MB   15 APR 96 02 MAY 96  ASH     LOGGED    Method Blank          0
 0001 A * SA   15 APR 96 02 MAY 96  ASH     LOGGED    01NE                  0
 0002 A   SA   15 APR 96 02 MAY 96  ASH     LOGGED    02SE                  0
 0003 A   SA   15 APR 96 02 MAY 96  ASH     LOGGED    03SW                  0
 0004 A   SA   15 APR 96 02 MAY 96  ASH     LOGGED    04NW                  0
```

EXAMPLE 8 (cont.)

EXAMPLE 8

Quanterra, Inc. — Quanterra Environmental Services - Sacramento
Date: 16 APR 96 — RBONALY — Sample Detail Screen Print Report
Page 1

SAMPLE DETAIL

Client: CALLAB-QUECA01 Name: Quanterra, Inc. - Sacramento
Program: CALLAB-003458 Name: Advanced Tech (LR) - General Program
Project: CALLAB-087261 Name: [redacted]ASH TCDDDF 041596
Sample #: 0001 • Aliquot: MB Group Code: B
Sample Status: LOGGED
Status History: L

Client Description: Method Blank
Calculate holding times from collection dates? N
Collection Date: Time: Interval: 0.00
Receipt Date: 15 APR 96 Time: 10:00 Turnaround Class: NORMAL
Sample Due Date: 02 MAY 96 Matrix: ASH
Quanterra sample #: 414045 Permit #: Well ID:
Lab Description: Method Blank
Received By: RBONALY
Disposal Type: NO-DISP Last Instruction #: 0
Color Before: Clarity Before: Artifact:
Color After : Clarity After : Texture:
Delivery Accepted: RBONALY 15 APR 96 Disposed:
Logged: RBONALY 15 APR 96 Completed:
Created: RBONALY 15 APR 96 Modified:

Smf	Alq	Rp	Test Code	Ver	Matrix	Priority	Statdr	Due Date	M
0001	MB	0	TCDDDF-S	3	ASH	NORMAL	PREPWAIT	02 MAY 96	
0001	MB	0	P-DXNFUR-SOX-L	3	ASH	NORMAL	LOGGED	25 APR 96	

EXAMPLE 8 (cont.)

EXAMPLE 8

Quanterra, Inc. Quanterra Environmental Services - Sacramento
Date: 15 APR 96 RBONALY Sample Detail Screen Print Report
Page 1

SAMPLE DETAIL
Client: CALLAB-QUECAD1 Name: Quanterra, Inc. - Sacramento
Program: CALLAB-003458 Name: Advanced Tech (LR) - General Program
Project: CALLAB-087261 Name: [redacted] ASH TCDDDF 041596
Sample #: 0001* Aliquot: SA Group Code: A
Sample Status: LOGGED
Status History: L

Client Description: BINE
Calculate holding times from collection dates? Y
Collection Date: 14 APR 96 Time: 16:30 Interval: 0.00
Receipt Date: 15 APR 96 Time: 10:00 Turnaround Class: NORMAL
Sample Due Date: 02 MAY 96 Matrix: ASH
Quanterra sample #: 414046 Permit #: Well ID:
Lab Description: 3-500CEJ
Received By: RBONALY
Disposal Type: DISP-30 Last Instruction #: 0
Color Before: Clarity Before: Artifact:
Color After : Clarity After : Texture:
Delivery Accepted: RBONALY 15 APR 96 Disposed:
Logged: RBONALY 15 APR 96 Completed:
Created: RBONALY 15 APR 96 Modified:

Smf	Alq	Rp	Test Code	Ver	Matrix	Priority	Status	Due Date	M
0001	SA	0	TCDDDF-S	3	ASH	NORMAL	PREPWAIT	02 MAY 96	
0001	SA	0	P-DIXFUR-SOX-L	1	ASH	NORMAL	LOGGED	24 APR 96	
0001	SA	0	8085-PCB-S	2	ASH	NORMAL	PREPWAIT	02 MAY 96	
0001	SA	0	P-8080-PCB-S	1	ASH	NORMAL	LOGGED	28 APR 96	

EXAMPLE 8 (cont.)

EXAMPLE 8

Quanterra, Inc. — Quanterra Environmental Services - Sacramento
Date: 15 APR 96 — RBCNALT — Group Codes Screen Print Report
Page 1

GROUP CODE

Client: CALLAB-QUECA01 Name: Quanterra, Inc. - Sacramento
Program: CALLAB-003450 Name: Advanced Tech (LR) - General Program
Project: CALLAB-007261 Name: [redacted] ASH TCDDDF 041896

Grp	Smp/	Description	Matrix	Disposal	Inst
A	0	TCDDDF/BDBO(PCB)	ASH	DISP-30	0
B	0	TCDDDF(MB)	ASH	NO-DISP	0

Grp	Description	Matrix	Disposal	# Samples Expect
A	TCDDDF/BDBO(PCB)	ASH	DISP-30	0

Package/Project	Test Code	Yr	Rp	Test Description	Inst
	TCDDDF-S	3	0	2,3,7,8-TCDD/TCDF - by Low	0
	P-DIXFUR-SOX-L	1	0	Prep - Low Res. Soxhlet Ext	0
	BDBO-PCB-S	2	0	PCBs in Soil	0
	P-BDBO-PCB-S	1	0	Prep - PCBs by GC	0

Grp	Description	Matrix	Disposal	# Samples Expect
B	TCDDDF(MB)	ASH	NO-DISP	0

Package/Project	Test Code	Yr	Rp	Test Description	Inst
	TCDDDF-S	3	0	2,3,7,8-TCDD/TCDF - by Low	0
	P-DIXFUR-SOX-L	1	0	Prep - Low Res. Soxhlet Ext	0

EXAMPLE 8 (cont.)

EXAMPLE 8

Ticket Report
Quanterra Environmental Services, Sacramento -
880 Riverside Parkway

West Sacramento, California 95605
(916) 373-5600

CALLAB-087261

Date Received : 18 APR 96 10:00

[redacted]

(414) 272-0209

Project ID,
EPA Case, RMA Lot : [redacted]
P.O. Number : 122623
Delivered By :
Storage Location : W1A
Logged in by : RBONALY

Four incinerator ash samples received under Chain-of-Custody in good condition. Delivered by Express Western Delivery.

Sample ID	Ensece ID	Client's label info	Date/Time Samp.	Containers
				Method Blank
087261-0001-MB	414045	Method Blank		
087261-0001-SA	414046	01NE	14 APR 96 16:30	3-500CGJ
087261-0002-SA	414047	02SE	14 APR 96 17:00	3-500CGJ
087261-0003-SA	414048	03SW	14 APR 96 17:30	3-500CGJ
087261-0004-SA	414049	04NW	14 APR 96 18:00	3-500CGJ

Samples not destroyed in testing are retained a maximum of thirty (30) days unless otherwise requested.

Client Manager: Karen Verrue

EXAMPLE 9

Quanterra Incorporated - West Sacramento
DATA CONTROL INSTRUCTION SHEET

EXAMPLE 9

Project/_____

Resubmittal ☐ Old Project/_____

CC:_____(Name)

_____(LIMS ID)

Send by:_____(Date)

MAIL PROCESS:

☐ Quanterra Courier
☐ DHL
☐ California Courier
☐ Federal Express
☐ Regular Mail
☐ Other:_____

☐ Raw Data
☐ Disk Deliverables
☐ Client Accounts
☐ E-Mail
☐ Tier I
☐ Tier II

INVOICE

☐ LIMS ONLY (Print invoice from lims only)
☐ PLIN (Print invoice w/PLIN in front of expense)
☐ PLIN & PJ DIST (Print invoice w/PLIN and Project discount, surcharge is by unit)
☐ PROJ DIST (Print invoice w/project discount, surcharge is by unit)
☐ PROJ DIST/SUR (Print invoice w/project discount and project surcharge)

☐ Mail with report
☐ Mail to accounts payable w/copy sent with report
☐ Other _____

ADDED INSTRUCTIONS

_____
_____
_____
_____
_____
_____
_____
_____
_____
_____
_____
_____
_____
_____

(OVER)

12/96 MCD

EXAMPLE 9 (cont.)

EXMPLE 9

ADVANCED TECHNOLOGY
REPORTING INSTRUCTION SHEET

Quanterra

Project #_____

[ ] Fax Date _____

Fax #_____

Send Original Report By:
[ ] Fed-X
[ ] Regular Mail
[ ] International Mail
[ ] Quanterra Courier
[ ] Electronic Delivery
[ ] _____

Data Due to Client_____

Report To: _____

Include: _____ Copy/Copies
[ ] Summary Data Sheets & COC
[ ] Summary Results Only
[ ] Chromatograms
[ ] Initial Calibration
[ ] Continuing Calibration
[ ] LCS/OPR
[ ] Disk (Format) _____
[ ] Paginated
[ ] Wet Weight [ ] Dry Weight
[ ] TEQ's
[ ] Ion Ratio's
[ ] QAPjP, SOW in place
[ ] Separate Dioxin and Furan Results
[ ] _____

CC Report Copy To:

_____

_____

_____

CC Deliverables:
[ ] Same As Above
[ ] Cover Letter Only
[ ] Summary Data Sheets & COC
[ ] _____

Mail Invoice:
[ ] To Accounts Payable with Copy in Report
[ ] With Report
[ ] _____

Cover Letter Comments:

_____

_____

_____

Additional Instructions:

_____

_____

_____

EXAMPLE 10

EXAMPLE 10

Quanterra Incorporated - West Sacramento

IN-HOUSE CHAIN-OF-CUSTODY

PROJECT ID:________ LOC:________ Page____

SAMPLE#	CONTAINER	CHECK-OUT INIT	TEST	DATE	TIME	W	SAMPLE#	CONTAINER	CHECK-IN INIT	DATE	TIME	STRM LOC	COMMENT

[illegible]

EXAMPLE 11

Quanterra Incorporated - West Sacramento
Project Review Checklist #1

EXAMPLE 11

Project Number_____

Shipment Receipt:

__ Bill(s) of lading received (signature if necessary)
__ COC/Letter of Transmittal signed and dated with time of receipt
__ Condition of samples noted on COC
__ Project Receipt Checklist completed
__ COC generated for undocumented samples (from label info)

Project Folder:

__ Data Control Instruction Sheet included
__ Sample ID(s) and collection dates/time(s) match COC or discrepancies explained
__ Sample list screen report stapled to groupcode screen report
__ Groupcode(s) verified
__ Number, matrix, and condition of samples present in comments
__ Means of delivery present in comments
__ Correct receipt date and time reported
__ Ticket report stapled to copy of COC
__ Ticket report, original COC, and bill(s) of lading stapled to left side of folder
__ Folder color reflects TAT assigned (red for Rush, Manila for Normal)
__ Project label is correct
__ E-mail notification sent to Project Manager
__ Billing calculated
__ Move projects to log book

__ Watchers notified of SHT(s) and metals notified of filtering/preservation
__ HTV filed for samples with expired hold times upon receipt (if applicable)
__ Anomaly sent
__ Subbed out analyses changed to SENT-OUT (or @CEL, @WADS, etc.)
__ RUSH e-mail notification sent to appropriate department(s)

In-House COC Folder:

__ In-house COC stapled to right side of folder
__ Ticket report and copy of bottle inventory (if needed) stapled to left side of folder
__ Labels are correct
__ Folder filed

Initial/Date _____

QA255 03/96 MCD

EXAMPLE 12

QUANTERRA INCORPORATED
[illegible]
Temperature Log

EXAMPLE 12

Thermometer ID: N/A

Location: Sample Receiving

Unit #: WALK-IN

Month/Year: 5/96

Temperature must be within 2 - 6°C.

If temperature readings fall outside this range, inform the QA Department. If the date indicated falls on a weekend, write "WKND" in the space.

Date	Temperature	Initials	Date	Temperature	Initials
1	___	___	17	___	___
2	___	___	18	___	___
3	___	___	19	___	___
4	___	___	20	___	___
5	___	___	21	___	___
6	___	___	22	___	___
7	___	___	23	___	___
8	___	___	24	___	___
9	___	___	25	___	___
10	___	___	26	___	___
11	___	___	27	___	___
12	___	___	28	___	___
13	___	___	29	___	___
14	___	___	30	___	___
15	___	___	31	___	___
16	___	___			

Comments: _____

Please be sure to anomalize any excursions or omissions on this log sheet.

EXAMPLE 13

EPA / CLP SAMPLE LOG-IN SHEET EXAMPLE 13

QUANTERRA Environmental Services, Inc. (West Sacramento)	

	Sample ID	EPA Tag #	Assigned Lab #	Condition of Sample Shipment, etc.
1. Custody Seals — Present / Absent; Intact / Broken				Sample Condition:
2. Custody Seal Nos.				
3. Chained-Custody Form/ Traffic Report — Present / Absent				
4. Delivery Vehicle/Airbill — Air / Water; Present / Absent				
5. Vehicle/Airbill Number				
6. Sample Tags — Present / Absent / NA				Discrepancies:
7. Sample Tag Numbers — Listed / Not Listed / NA				
8. Data consistent on all forms signed? — Yes / No				
9. Shipping Container — Intact / Damaged				
10. Temperature(s) — Blank; Samples				
11. Date Received at lab				
12. Time Received				

Sample Storage and Transfer			
Relinquished	Location	By	On
Accepted	Location	By	On

Initiate this form with full EPA or full CLP projects.
1100 SCD

SA-001

SOP AMENDMENT

SOP Number:	SAC-QA-0003
SOP Title:	SAMPLE RECEIPT AND PROCEDURES
SOP Sections Affected by Change:	All.
Reason for Addition or Change:	An appendix has been added to deal specifically with chemical warfare agents and their degradates. The appendix must be reviewed prior to first-time handling of samples suspected of containing degradate contaminants.
Change Effective From [Date]:	3/13/97
Change or Addition (Specific Section; use additional sheets if necessary.):	See attached Appendix 1 to SAC-QA-0003.

Submitted by: **Date:**

Approved by:*

Technical Reviewer: Michael S. Fitzgerald *Date:* 3/14/97

EH&S Signature: Cheryl Stitt *Date:* 3/14/97

QA Signature: Gail Chr Clark *Date:* 3/14/97

Management Signature: Bruce E Walker *Date* 3/14/97

Must be same signature authorities of SOP being revised. d:\\qsacca01\sacqa\sop\amend.dot

Appendix 1

Chemical Warfare Degradates - Potential Hazards in Sample Receipt

Background

Quanterra has begun receiving samples to be analyzed for degradates of chemical warfare agents. These degradates are not particularly toxic, as compared with most of the compounds we deal with every day. The fact that these compounds are degradates of chemical warfare agents does, however, present a **new type of potential hazard** for us. We have developed policies regarding the handling of such samples but as we begin to get real-world samples, we are finding that some of our initial assumptions regarding them are not holding true. The purpose of this document is to discuss these compounds and the potential hazards involved in handling them.

Review of Agent Compounds

Chemical warfare agents fall into a wide variety of categories, ranging from defoliants such as Agent Orange (tear gas) to nerve agents such as Sarin. The two types of agents we are most concerned with are the **nerve agents** and the **blistering agents**, such as sulfur mustard. Other lethal agents have been developed and tested for use in chemical warfare; however, these other compounds are either extremely volatile and reactive (and therefore highly unlikely to be present in an environmental sample) or were never produced in significant quantities in the U.S.

Nerve agents

These compounds are members of the organophosphate class of compounds. These compounds are similar to many common household pesticides such as diazinon. The difference is that the nerve agents are far more toxic to humans. The first nerve agent, Tabun, was discovered by a German pesticide company during the process of screening new compounds for pesticide activity. Once the toxicity of Tabun was determined, various military organizations began screening many related compounds in hopes of finding even more efficient ways of annihilating large numbers of people. Out of this effort have come five established (non-classified, i.e. public domain) agents. --- **Tabun (or GA), Sarin (or GB), GD, GF, and VX.**

All of these compounds are toxic via inhalation, ingestion, skin contact, or just about any other route of entry into the body.

A **characteristic of these compounds is their volatility.** A more volatile nerve agent will disperse in air more effectively than a less volatile one, producing increased numbers of casualties through inhalation.

Less volatile agents, on the other hand, will stick around on soil, vegetation, clothing, etc. and will therefore last longer. Out of these various agents, the only two produced in significant quantities in the U.S. are Sarin and VX. **Sarin is the most volatile** of the above agents while VX is the least volatile. Both are exquisitely toxic - a drop of pure VX barely visible to the naked eye is enough to kill a person through skin contact.

Blistering agents

These were the first of the modern agents developed specifically for the purpose of killing people. Unlike the nerve agents, the blistering agents are basically different mixtures of one compound, sulfur mustard, with other non-toxic chemicals which affect its dispersion characteristics.

The primary hazard with sulfur mustard involves skin contact. The term "mustard gas" is somewhat of a misnomer - the stuff will kill you if you inhale enough of it, but this not likely to occur as it is not particularly volatile under normal conditions. Significant quantities of sulfur mustard (a.k.a. HD or HT) have been produced in the U.S.

Chemical Agents in the Environment

It is unlikely (but never impossible) that we at Quanterra will receive any sample which contains a dangerous concentration of active agent. We **should not** receive samples from any area known to be contaminated with bulk amounts of active chemical agent (areas with buried drums, old munitions, etc.) because the Army policy for such sites is to destroy the agent onsite.

Additionally, most of the areas in which degradate analysis is required are areas in which agent was used decades ago - all active agent is likely to have degraded. Nonetheless, it is imperative that we know as much as possible regarding the compounds and the samples in order to protect ourselves against any potential hazard.

The behavior of these compounds in the environment has been extensively studied. Most of the information we have is from a study titled **Environmental Chemistry and Fate of Chemical Warfare Agents**. This study was prepared for the Army Corps of Engineers by Southwest Research Institute in 1994.

Nerve Agents

All of the compounds we are concerned with (Sarin and VX) undergo **hydrolysis in the presence of water**. This hydrolysis proceeds at different rates, depending upon the compound. At worst (cool temperature, normal pH, no dissolved ions, no microbes), either of these compounds getting into water would be degraded to extremely low levels ($< 1 \times 10^{-6}$ of the original concentration) within a couple of years. It is more likely that this level of degradation would occur much more quickly.

Degradation in soil is a far more complex issue. The rate of hydrolysis will depend upon a variety of factors, including soil moisture, pH, mineral content, microbes, temperature, etc. Most available studies show that these compounds last no more than a few days in the tested soil types.

Sulfur Mustard

In some ways, sulfur mustard behaves in a fashion similar to the nerve agents - i.e. it hydrolyzes rapidly in the presence of water. There is, however, an important difference. Under the right conditions, **the hydrolysis products of mustard can polymerize and form bubbles containing active mustard**. The mustard inside of these bubbles is shielded from further hydrolysis by the hydrolysis products. This has turned out to be a problem in areas where large amounts of mustard were dumped at sea. Fishermen in such areas have been injured due to pulling up nets contaminated with blobs of active mustard. An indication that this may have occurred would be a biphasic sample. This situation can also occur in soil.

Potential Hazards to Quanterra Personnel

Following steps listed in this Appendix and other safety policies will help reduce hazards to the greatest degree possible. However, such policies are no substitute for educated, observant personnel.

As always, you must think about what you are doing when you handle these materials. No policy can account for every potential situation. Staff are expected to follow all sample handling policies identified in the Chemical Hygiene Plan and steps following sample receipt listed in this Appendix.

Soil samples should be screened for agent prior to their shipment to Quanterra. Data should be reviewed at the project management and EH&S staff level to ensure samples are "safe" for staff to handle. If the screening status is unknown (i.e. no data is available), project management personnel (Karla Buechler or Mike Filigenzi) should be consulted. Samples **should not** be handled. If staff are unavailable, the cooler should be left in cold storage until the situation is resolved.

Water samples for degradate analysis are not likely to be screened for active agent prior to our receipt. This was not our original understanding, but the Army's policy appears to be that active agents degrade quickly enough in aqueous systems that no screening is needed. The data provided in the Environmental Chemical Fate and Chemical Warfare agents seems to support this notion. Half-life for many of the agents have been documented and is readily available for review by staff. None-the-less, it is still important to exercise care with such samples.

When in doubt, seek assistance from project management, a TC or EH&S staff.

Sample Receipt Procedures

⇒ Follow SAC-QA-003, Section 4 (Procedure)

⇒ Follow steps 4.2.1 - 4.2.5

⇒ Opening Shipment -- Prioritizing Workload

⇒ Review screen data BEFORE opening the cooler if soil samples.

Note: Double gloves are required when handling chemical warfare degradates samples.

⇒ Following established safety policies, open the cooler, remove any paperwork, and check the interior condition.

⇒ Suspect coolers should be opened in the Hood to reduce any potential for exposure.

⇒ If a sample contains a broken or leaking sample, isolate the cooler in the hood and IMMEDIATELY contact the project manager, TC and/or EH&S staff.

⇒ Any sample which appears to be biphasic in appearance must be isolated. Immediate notification to project management, TC and/or EH&S staff is required.

⇒ When in doubt, get help regarding sample receipt. Worker, health and safety is paramount to sample analysis.

<u>Scope of Work</u>

It is possible that we will receive a large number of samples from the **Aberdeen Proving Grounds (APG)** over the next few years. APG has been the center for chemical warfare training and research in the U.S. since 1918. We have been told by our client (EA Engineering) that the procedure for investigating sites at APG is as follows:

- APG personnel perform a **hazard analysis** to determine whether there is an potential for agent contamination in soil based on historical information.
- If there is reason to believe chemical agents may have been used at the site, soil samples are taken and **screened** for agent.
- If the screening shows no agent contamination, sampling for other parameters may proceed.

We do not have enough experience with these projects to know whether this procedure is unique to APG or a standard Army procedure. We are conducting additional investigation to further verify this.

In any case, results of screening will be made available to Quanterra.

Regardless of screening data, any cooler containing samples for degradate analysis (or any other samples from an area suspected of potential agent contamination) **should be inspected carefully** upon receipt. Anyone inspecting the samples, logging them in, or handling them for any other reason should observe all of Quanterra's regular safety procedure.

In addition, **two pairs of gloves** should be worn in order to minimize any potential for skin contact with toxic compounds. Please note that **skin contact appears to be the most likely potential route of exposure.** This is based on the fact that nerve agents are likely to have degraded leaving sulfur mustard as the most likely potential contaminant and due to the likelihood that the samples will be cold. The temperature in the cooler is important from the standpoint of safety as well as sample integrity - cold samples mean a significantly lower potential for any kind of toxic vapor formation. Coolers containing broken jars or bottles should be placed in a hood immediately and left there until the client has been contacted. EH&S staff and/or the Project managers will give instructions regarding return to client or disposal based on the screen data.

In conclusion, it must be emphasized that these samples must be handled with the appropriate level of care. Observant, educated personnel are our best defense against exposure to any kind of toxic materials found in our samples.

APPENDIX F
EXAMPLE DATA DELIVERABLE PACKAGE (EXPLOSIVES)

TABLE OF CONTENTS

	PAGE
Data Deliverable Package from Southwest Laboratories	F-1
Data Deliverable Package from Quanterra Environmental Services	F1-1

Quanterra
Environmental Services

Quanterra Incorporated
880 Riverside Parkway
West Sacramento, California 95605

916 373-5600 Telephone
916 372-1059 Fax

August 7, 1998

QUANTERRA INCORPORATED PROJECT NUMBER: 300612
PO/CONTRACT: [redacted]

[redacted]

Dear Mr. [redacted]

This report contains the analytical results for the one sample received under chain of custody by Quanterra Incorporated on July 25, 1998. This sample is associated with your [redacted] project.

The case narrative is an integral part of this report.

If you have any questions, please feel free to call me at (916)374-4362.

Sincerely,

Diana Brooks
Project Manager

Quanterra

TABLE OF CONTENTS

QUANTERRA INCORPORATED PROJECT NUMBER 300612

Case Narrative

Quanterra's Quality Assurance Program

Sample Description Information

Chain of Custody Documentation

Summary Report

Nitroaromatics and Nitramines (Aqueous) - Method 8330
Sample(s): 1
- Sample Data
- Instrument Retention Time Windows
- Initial Calibration
- Continuing Calibration
- Method Blank Data
- Laboratory Control Sample Data
- Matrix Spike/Matrix Spike Duplicate
- Instrument Analysis Log Copies
- Sample Extraction/Preparation Log Copies

Quanterra

CASE NARRATIVE

QUANTERRA INCORPORATED PROJECT NUMBER 300612

There were no anomalies associated with this project.

Quanterra

Quanterra - Western Region

Quality Control Definitions

QC Parameter	Definition
QC Batch	A set of up to 20 field samples plus associated laboratory QC samples that are similar in composition (matrix) and that are processed within the same time period with the same reagent and standard lots.
Duplicate Control Sample (DCS)	Consist of a pair of LCSs analyzed within the same QC batch to monitor precision and accuracy independent of sample matrix effects. This QC is performed only if required by client or when insufficient sample is available to perform MS/MSD.
Duplicate Sample (DU)	A second aliquot of an environmental sample, taken from the same sample container when possible, that is processed independently with the first sample aliquot. The results are used to assess the effect of the sample matrix on the precision of the analytical process. The precision estimated using this sample is not necessarily representative of the precision for other samples in the batch.
Laboratory Control Sample (LCS)	A volume of reagent water for aqueous samples or a contaminant-free solid matrix (Ottawa sand) for soil and sediment samples which is spiked with known amounts of representative target analytes and required surrogates. An LCS is carried through the entire analytical process and is used to monitor the accuracy of the analytical process independent of potential matrix effects.
Matrix Spike and Matrix Spike Duplicate (MS/MSD)	A field sample fortified with known quantities of target analytes that are also added to the LCS. Matrix spike duplicate is a second matrix spike sample. MSs/MSDs are carried through the entire analytical process and are used to determine sample matrix effect on accuracy of the measurement system. The accuracy and precision estimated using MS/MSD is only representative of the precision of the sample that was spiked.
Method Blank (MB)	A sample composed of all the reagents (in the same quantities) in reagent water carried through the entire analytical process. The method blank is used to monitor the level of contamination introduced during sample preparation steps.
Surrogate Spike	Organic constituents not expected to be detected in environmental media and are added to every sample and QC at a known concentration. Surrogates are used to determine the efficiency of the sample preparation and the analytical process.

Source: Quanterra® Quality Control Program, Policy QA-003, Rev. 0, 8/19/96.

Quanterra

SAMPLE DESCRIPTION INFORMATION
for
[redacted] Corporation

Lab ID	Client ID	Matrix	Sampled Date	Sampled Time	Received Date
300612-0001-SA	[redacted]	AQUEOUS	24 JUL 98	12:30	25 JUL 98

Chain of Custody Record

Quanterra

QUA-4124

Client	Project Manager	Date	Chain Of Custody Number 31517
Address	Telephone Number (Area Code)/Fax Number	Lab Number	Page _____ of _____
City / State / Zip Code	Site Contact		
Project Name	Carrier/Waybill Number		
Contract/Purchase Order/Quote No.			

Sample I.D. No. and Description	Date	Time	Sample Type	Total Volume	Containers Type	Containers No.	Preservative	Condition on Receipt	Analysis

Special Instructions

Possible Hazard Identification
☐ Non-Hazard ☐ Flammable ☐ Skin Irritant ☐ Poison B ☐ Unknown

Sample Disposal
☐ Return To Client ☐ Disposal By Lab ☐ Archive For _____ Months

Turn Around Time Required
☐ Normal ☐ Rush

QC Level
☐ I. ☐ II. ☐ III.

Project Specific (Specify)

	Date	Time		Date	Time
1. Relinquished By			1. Received By		
2. Relinquished By			2. Received By		
3. Relinquished By			3. Received By		

Comments

DISTRIBUTION: WHITE - Stays with Sample; CANARY - Returned to Client with Report; PINK - Field Copy

FL6

Quanterra

Nitroaromatics and Nitramines (Aqueous) - Method 8330

Quanterra

Nitroaromatics and Nitramines by HPLC
Method 8330

Client Name: [redacted]
Client ID: [redacted]
LAB ID: 300612-0001-SA
Matrix: AQUEOUS
Authorized: 25 JUL 98

Sampled: 24 JUL 98
Prepared: 28 JUL 98

Received: 25 JUL 98
Analyzed: 04 AUG 98

Dilution Factor: 1.0

Parameter	Result	Units	Reporting Limit	Qualifier
RDX	1.3	ug/L	5.0	J
1,3-Dinitrobenzene	ND	ug/L	5.0	
Nitrobenzene	ND	ug/L	5.0	
2,4,6-Trinitrotoluene	ND	ug/L	5.0	
Tetryl	ND	ug/L	5.0	
2,4-Dinitrotoluene	ND	ug/L	5.0	
2,6-Dinitrotoluene	ND	ug/L	5.0	
2-Am-DNT	ND	ug/L	5.0	
4-Am-DNT	ND	ug/L	5.0	
2-Nitrotoluene	ND	ug/L	10	
4-Nitrotoluene	ND	ug/L	8.5	
3-Nitrotoluene	ND	ug/L	7.9	

Surrogate	Recovery	Acceptable Range
2,4-Dinitrofluorobenzene	87 %	65 - 135

Note J = Result is detected below the reporting limit or is an estimated concentration.
ND = Not Detected

Reported By: Jon Edmondson

Approved By: Emily Uebelhoer

The cover letter is an integral part of this report.
Rev 230787

F-8

Quanterra

QC LOT ASSIGNMENT REPORT
HPLC Analysis Area

Laboratory Sample Number	QC Matrix	QC Category	QC Lot Number (DCS)	QC Run Number (LCS/BLANK)
300612-0001-SA	AQUEOUS	8330-COE-A	28 JUL 98-13D	28 JUL 98-13D

Quanterra

METHOD BLANK REPORT
HPLC Analysis Area
Project: 300612

Test: 8330-IRPMS-1C-A
Method: 8330
Matrix: AQUEOUS
QC Lot: 28 JUL 98-13D
Analyzed: 04 AUG 98

Nitroaromatics and Nitramines by HPLC

QC Run: 28 JUL 98-13D
Time: 18:16

Analyte	Result	Units	Reporting Limit	Qualifier
RDX	ND	ug/L	5.0	
1,3-Dinitrobenzene	ND	ug/L	5.0	
Nitrobenzene	ND	ug/L	5.0	
2,4,6-Trinitrotoluene	ND	ug/L	5.0	
Tetryl	ND	ug/L	5.0	
2,4-Dinitrotoluene	ND	ug/L	5.0	
2,6-Dinitrotoluene	ND	ug/L	5.0	
2-Am-DNT	ND	ug/L	5.0	
4-Am-DNT	ND	ug/L	5.0	
2-Nitrotoluene	ND	ug/L	10	
4-Nitrotoluene	ND	ug/L	8.5	
3-Nitrotoluene	ND	ug/L	7.9	

Surrogate	% Recovery	Acceptable Range
2,4-Dinitrofluorobenzene	89	65 -135

ND = Not Detected

Quanterra

DUPLICATE CONTROL SAMPLE REPORT
HPLC Analysis Area
Project: 300612

Category: 8330-COE-A Explosives by HPLC
Testcode: 8330-IRPMS-1C-A Method: 8330
Matrix: AQUEOUS Concentration Units: ug/L
QC Lot: 28 JUL 98-13D Analyzed Date: 04 AUG 98 Time: 19:28

Analyte	Concentration Spiked	Measured DCS1	Measured DCS2	Accuracy (%) DCS1	Accuracy (%) DCS2	Limits	Precision (RPD) DCS	Limit
HMX	0.500	0.468	0.473	94	95	65-135	1.2	35
1,3,5-Trinitrobenzene	0.500	0.468	0.470	94	94	65-135	0.51	35
RDX	0.500	0.432	0.453	86	91	65-135	4.6	35
1,3-Dinitrobenzene	0.500	0.425	0.461	85	92	65-135	8.1	35
Nitrobenzene	0.500	0.369	0.458	74	92	65-135	22	35
2,4,6-Trinitrotoluene	0.500	0.511	0.495	102	99	65-135	3.2	35
Tetryl	0.500	0.510	0.512	102	102	65-135	0.47	35
2,4-Dinitrotoluene	0.500	0.486	0.485	97	97	65-135	0.12	35
2,6-Dinitrotoluene	0.500	0.432	0.487	86	97	65-135	12	35
2-Am-DNT	0.500	0.453	0.478	91	96	65-135	5.2	35
4-Am-DNT	0.500	0.482	0.456	96	91	65-135	5.5	35
2-Nitrotoluene	0.500	0.414	0.504	83	101	65-135	19	35
4-Nitrotoluene	0.500	0.413	0.552	83	110	65-135	29	35
3-Nitrotoluene	0.500	0.430	0.523	86	105	65-135	20	35

Surrogate	Concentration Spiked	Measured DCS1	Measured DCS2	Accuracy(%) DCS1	Accuracy(%) DCS2	Limits
2,4-Dinitrofluorobenzene	2.6	2.4	2.4	90	92	65-135

Calculations are performed before rounding to avoid round-off errors in calculated results.

RUSH TAT

QUANTERRA - SACRAMENTO
PROJECT DETAIL: GC DEPARTMENT

PROJECT ID: 300612
CLIENT MANAGER: DBROOKS TEAM: Environmental Servic
PROJECT DUE DATE: 06 AUG 98
CLIENT/PROJECT NAME: [redacted] 072598

8330-IRPMS-1C-A (90.3) 1.(T: 1).

DATE DELIVERED FOR LIMS ENTRY : 05 AUG 98 TIME DELIVERED: 16:46

LIMS ENTER: _____ - 98 Formaster ENTER: _____ - 98

DATE TO FINAL REVIEW: _____ - 98 PEER: _____ - 98

FINAL: a 8/5 - 98 FINAL: _____ - 98

DATE DELIVERED TO DATA CONTROL: 8/5 - 98

_____ No problems (d+i) X See Anomaly Sheet (d+i)

COMMENTS:

GC-0037 (1.0) 7/9/91

F1-12

Organics Raw Data Package

Sample Data

Nitroaromatics and Nitramines by HPLC
Method 8330

Client Name: [redacted]
Client ID: [redacted]
LAB ID: 300612-0001-SA
Matrix: AQUEOUS
Authorized: 25 JUL 98

Sampled: 24 JUL 98
Prepared: 28 JUL 98

Received: 25 JUL 98
Analyzed: 04 AUG 98

Dilution Factor: 1.0

Parameter	Result	Units	Reporting Limit	Qualifier
RDX	1.3	ug/L	5.0	J
1,3-Dinitrobenzene	ND	ug/L	5.0	
Nitrobenzene	ND	ug/L	5.0	
2,4,6-Trinitrotoluene	ND	ug/L	5.0	
Tetryl	ND	ug/L	5.0	
2,4-Dinitrotoluene	ND	ug/L	5.0	
2,6-Dinitrotoluene	ND	ug/L	5.0	
2-Am-DNT	ND	ug/L	5.0	
4-Am-DNT	ND	ug/L	5.0	
2-Nitrotoluene	ND	ug/L	10	
4-Nitrotoluene	ND	ug/L	8.5	
3-Nitrotoluene	ND	ug/L	7.9	

Surrogate	Recovery	Acceptable Range
2,4-Dinitrofluorobenzene	87 %	65 - 135

Note J = Result is detected below the reporting limit or is an estimated concentration.
ND = Not Detected

Reported By: Jon Edmondson

Approved By: Emily Uebelhoer

The cover letter is an integral part of this report.
Rev 230787

Injection Report

Acquired on 4-AUG-1998 at 23:06

Quanterra, Inc (West Sacramento)

Analyst Name : LC4UV
Lims Id : 109LC4UV9050
Comment : 75:25:0/2-22-50:00:50/2"- H2O:2%THF/MeOH:Methanol (Analysis)
Method Title : Var-LC5500 UV Hypersil-C8 150x4.6mm 5um SN251486 8330M
Sample Name : 300612-1 770ML/20ML
Sample Id :
Sample Type : Sample Amount=1.00000
Bottle No : 8

PEAK INFORMATION

Peak	RT mins	RT Exp	Hght uV	ppb	Peak name	Width
1	3.422	0.000	18	0.00000		8.0
2	3.804	3.769	3377	7.31382	HMX	5.3
3	7.031	6.907	445	1.29424	RDX	11.7
4	8.000	0.000	130	0.00000		13.9
5	8.569	8.476	1564	2.25634	2,4-Dinitrofluorobenzene(Surr)	12.3
6	10.622	0.000	81	0.00000		12.8
7	13.138	0.000	127	0.00000		20.3
8	13.956	14.078	17	0.03354	TETRYL	11.2
9	14.453	0.000	29	0.00000		13.9
10	18.187	0.000	24	0.00000		18.1
11	21.467	0.000	74	0.00000		32.0
12	23.600	0.000	37	0.00000		14.9
13	24.373	0.000	64	0.00000		3.2
14	24.871	0.000	199	0.00000		5.3

Totals		
Unknowns	783	0.00000
Named	5403	10.89795
Grand Total	6186	10.89795

ANALYSIS SUMMARY

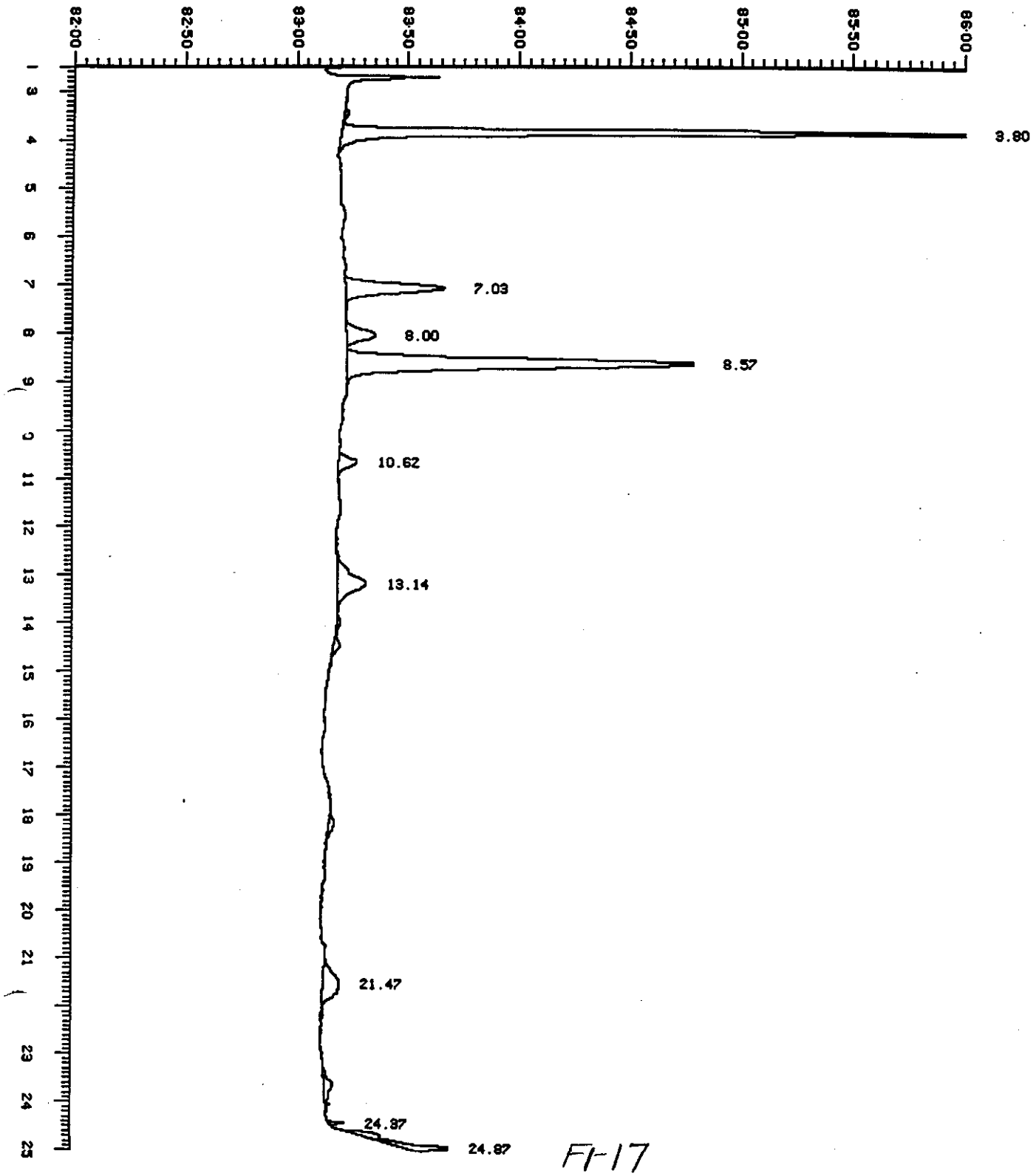
Method..... 8330 HY
Run sequence..... EXP4HY1
Calibration..... 03AUG98
External standard calibration using height
Calibration last modified on 4-AUG-1998 at 22:55
Calibration initialised on 12-DEC-1997 at 21:47
Method last modified on 4-AUG-1998 at 16:40

Uncalibrated peaks use user factor (0.0000)

Quanterra, Inc (West Sacramento)

[AUG_SV] 109 4H_04AUG981450,10,1

300612-1 770ML/20ML. Amount : 1.000.
Varian 5560/UV9050 @250nm flow=1.5 injection volume=200uL
Acquired on 4-AUG-1998 at 23:06
Reported on 4-AUG-1998 at 23:31
Box 1 (of 1)



F1-17

Quanterra / W. Sacramento
Chromatography Summary

Analysis ID: [AUG_SV] 109 4H_04AUG981450,10
Acquired: HPLC #4 4-AUG-1998 at 23:06

Sample: 300612-1 770ML/20ML **Mult:** 20.00 **Dilf:** 1.00 **Div:** 770.0
Method File: 8330_HY 75:25:0/2-22-50:00:50/2"- H2O:2%THF/MeOH:Methanol (Analysis)
Calibration: 03AUG98 8330H 2.5-1000ng/mL Phenomenex Hypersil C8 SN251486

Target Analyte Results

Channel 109 Var-LC5500 UV
Hypersil-C8
150x4.6mm 5um SN251486 8330M

	Peak Name	RT	Diff	Height	ppb	R	P	RL
1	HMX	3.804	+0.04	3377	7.3138			NA
2	1,3,5-Trinitrobenz							
3	RDX	7.031	+0.12	445	1.2942			5.00
4	2,4-Dinitrofluorob	8.569	+0.09	1564	2.2563		F	
5	1,3-Dinitrobenzene							5.00
6	Nitrobenzene							5.00
7	2,4,6-Trinitrotolu							5.00
8	TETRYL	13.956	-0.12	17	0.0335	U	F	5.00
9	2,4-Dinitrotoluene							5.00
10	2,6-Dinitrotoluene							5.00
11	2-AM-4,6-Dinitroto							5.00
12	4-AM-2,6-Dinitroto							5.00
13	2-Nitrotoluene							10.0
14	4-Nitrotoluene							8.50
15	3-Nitrotoluene							7.90

Surrogate Results

	Peak Name	Amount Spiked	Amount Found	%Rec	-Limits-
4	2,4-Dinitrofluorob	2.60	2.2563	87	(65 - 135)

Notes:
8330-IRPMS-1C-A

Range Flags (R)
U - Under calibration Range
O - Over calibration Range
X - Not calibrated

Peak Type Flags (P)
* - Flat top Peak
F - Forced baseline
M - Modified baseline

Q/WS SUM 3.29A

Reviewer: a Date: 8/5/98
Printed: 08/04/98 23:31:27

FT 18

Retention Time Windows

Lab Name: QUANTERRA SACRAMENTO (8330 RT WINDOWS)
Instrument ID: LC#4
Column: Hypersil C8
Serial #: 237022

	DATE	TIME
STANDARD 1:	07/28/98	13:55
STANDARD 2:	07/28/98	19:38
STANDARD 3:	07/29/98	09:17

Component	RT 1	RT 2	RT 3	3X STD. DEVIATION
HMX	3.689	3.724	3.751	0.093
1,3,5-Trinitrobenzene	6.533	6.507	6.498	0.055
RDX	6.942	6.924	6.916	0.040
2,4-Dinitrofluorobenzen	8.578	8.560	8.551	0.041
1,3-Dinitrobenzene	9.564	9.547	9.538	0.040
Nitrobenzene	11.396	11.378	11.440	0.096
2,4,6-Trinitrotoluene	12.924	12.889	12.907	0.053
TETRYL	14.302	14.231	14.213	0.141
2,4-Dinitrotoluene	15.671	15.609	15.644	0.093
2,6-Dinitrotoluene	16.871	16.800	16.853	0.111
2-AM-4,6-Dinitrotoluene	17.476	17.404	17.422	0.112
4-AM-2,6-Dinitrotoluene	18.142	18.089	18.080	0.101
2-Nitrotoluene (o)	18.987	18.951	19.031	0.120
4-Nitrotoluene (p)	19.378	19.333	19.431	0.147
3-Nitrotoluene (m)	20.213	20.187	20.267	0.122

Initial Calibration

Quanterra / W. Sacramento
Calibration Statistics Summary

Calibration ID: [AUG_SV] 109 03AUG98
Instrument ID: 1C4 Page 1 of 1

File: 03AUG98 Title: 8330H 2.5-1000ng/mL Phenomenex Hypersil C8 SN251486
Curve Type: Line forced thro' zero (AVG RF) Weighting: 1/X sqd

Peaks: 15 Calibration based on: Height

RT	Component Name	Calibration Factor	RSD %	Dynamic Range Low	Dynamic Range High
3.77	HMX	11.993	3.7	5.00	1000
6.49	1,3,5-Trinitrobenzene	21.426	9.5	5.00	1000
6.91	RDX	8.9406	6.4	5.00	1000
8.48	2,4-Dinitrofluorobenzene(Surr)	17.998	4.6	5.00	150
9.44	1,3-Dinitrobenzene	21.547	8.7	2.50	1000
11.27	Nitrobenzene	10.802	6.0	5.00	1000
12.73	2,4,6-Trinitrotoluene (TNT)	13.462	6.9	5.00	1000
14.08	TETRYL	13.361	3.5	5.00	1000
15.41	2,4-Dinitrotoluene	14.020	8.6	2.50	1000
16.58	2,6-Dinitrotoluene	8.3239	3.1	5.00	1000
17.18	2-AM-4,6-Dinitrotoluene	10.488	8.4	5.00	1000
17.84	4-AM-2,6-Dinitrotoluene	8.7060	8.6	5.00	1000
18.69	2-Nitrotoluene	5.3495	12	10.0	1000
19.08	4-Nitrotoluene	4.3077	10	10.0	1000
19.94	3-Nitrotoluene	5.6389	6.4	10.0	1000

Analyst: JEDMONDSON Date: 04-AUG-1998 Reviewed: MHK Date: 8/5/98
CalibStats Ver 2.0 Printed at: 04-AUG-1998 16:44:34

F1-22

Quanterra / W. Sacramento
Calibration Statistics Report

Calibration ID: [AUG SV] 109 03AUG98
Instrument ID: LC4 PAGE 1 of 4

File: 03AUG98 Title: 8330H 2.5-1000ng/mL Phenomenex Hypersil C8 SN251486
Curve Type: Line forced thro' zero (AVG RF) Weighting: 1/X sqd
Quant Base: Height

HMX RT: 3.77 min Window: 3.57 - 3.97 min

Level	ID	Amount/Conc'n	Response Factor
1	1	5.0000	11.772
2	2	10.000	12.276
3	3	25.000	11.789
4	5	100.00	11.308
5	6	500.00	12.501
6	7	1000.0	12.312

Calib Factor: 11.993 RSD: 3.7%

1,3,5-Trinitrobenzene RT: 6.49 min Window: 6.29 - 6.69 min

Level	ID	Amount/Conc'n	Response Factor
1	1	5.0000	18.919
2	2	10.000	20.209
3	3	25.000	20.936
4	5	100.00	20.697
5	6	500.00	23.882
6	7	1000.0	23.916

Calib Factor: 21.426 RSD: 9.5%

RDX RT: 6.91 min Window: 6.71 - 7.11 min

Level	ID	Amount/Conc'n	Response Factor
1	1	5.0000	8.5905
2	2	10.000	8.6367
3	3	25.000	9.2256
4	5	100.00	8.1404
5	6	500.00	9.5120
6	7	1000.0	9.5382

Calib Factor: 8.9406 RSD: 6.4%

2,4-Dinitrofluorobenzene(Surr) RT: 8.48 min Window: 8.23 - 8.73 min

Level	ID	Amount/Conc'n	Response Factor
1	T1	5.0000	19.021
2	1	25.000	16.885
3	2	50.000	18.272
5	3	100.00	18.313
6	5	150.00	17.500

Calib Factor: 17.998 RSD: 4.6%

Analyst: JEDMONDSON Date: 04-AUG-1998 Reviewed: MH Date: 8/5/98
CalibStats Ver 2.0 Printed at: 04-AUG-1998 16:44:34

Quanterra / W. Sacramento
Calibration Statistics Report

Calibration ID: [AUG_SV] 109 03AUG98
Instrument ID: 1C4 PAGE 2 of 4

File: 03AUG98 Title: 8330H 2.5-1000ng/mL Phenomenex Hypersil C8 SN251486
Curve Type: Line forced thro' zero (AVG RF) Weighting: 1/X sqd
Quant Base: Height

1,3-Dinitrobenzene RT: 9.44 min Window: 9.24 - 9.64 min

Level	ID	Amount/Conc'n	Response Factor
1	T1	2.5000	22.319
2	1	5.0000	18.866
3	2	10.000	19.956
4	3	25.000	21.606
5	5	100.00	20.575
6	6	500.00	23.796
7	7	1000.0	23.711

Calib Factor: 21.547 RSD: 8.7%

Nitrobenzene RT: 11.27 min Window: 11.07 - 11.47 min

Level	ID	Amount/Conc'n	Response Factor
1	1	5.0000	9.5636
2	2	10.000	10.884
3	3	25.000	11.206
4	5	100.00	10.715
5	6	500.00	11.317
6	7	1000.0	11.128

Calib Factor: 10.802 RSD: 6.0%

2,4,6-Trinitrotoluene (TNT) RT: 12.73 min Window: 12.43 - 13.03 min

Level	ID	Amount/Conc'n	Response Factor
1	1	5.0000	12.085
2	2	10.000	12.604
3	3	25.000	13.714
4	5	100.00	13.700
5	6	500.00	14.412
6	7	1000.0	14.256

Calib Factor: 13.462 RSD: 6.9%

TETRYL RT: 14.08 min Window: 13.72 - 14.44 min

Level	ID	Amount/Conc'n	Response Factor
1	1	5.0000	14.086
2	2	10.000	13.145
3	3	25.000	13.717
4	5	100.00	13.365
5	6	500.00	13.058
6	7	1000.0	12.798

Calib Factor: 13.361 RSD: 3.5%

2,4-Dinitrotoluene RT: 15.41 min Window: 15.16 - 15.66 min

Level	ID	Amount/Conc'n	Response Factor
1	T1	2.5000	13.816
2	1	5.0000	11.651
3	2	10.000	14.095
4	3	25.000	13.806
5	5	100.00	14.289
6	6	500.00	15.297
7	7	1000.0	15.187

Calib Factor: 14.020 RSD: 8.6%

Quanterra / W. Sacramento
Calibration Statistics Report

Calibration ID: [AUG_SV] 109 03AUG98
Instrument ID: Lc4 PAGE 3 of 4

File: 03AUG98 Title: 8330H 2.5-1000ng/mL Phenomenex Hypersil C8 SN251486
Curve Type: Line forced thro' zero (AVG RF) Weighting: 1/X sqd
Quant Base: Height

2,6-Dinitrotoluene RT: 16.58 min Window: 16.33 - 16.83 min

Level	ID	Amount/Conc'n	Response Factor
1	1	5.0000	8.5235
2	2	10.000	7.9513
3	3	25.000	8.4391
4	5	100.00	8.0365
5	6	500.00	8.5480
6	7	1000.0	8.4452

Calib Factor: 8.3239 RSD: 3.1%

2-AM-4,6-Dinitrotoluene RT: 17.18 min Window: 16.93 - 17.43 min

Level	ID	Amount/Conc'n	Response Factor
1	1	5.0000	12.092
2	2	10.000	9.8497
3	3	25.000	10.011
4	5	100.00	9.6919
5	6	500.00	10.729
6	7	1000.0	10.555

Calib Factor: 10.488 RSD: 8.4%

4-AM-2,6-Dinitrotoluene RT: 17.84 min Window: 17.59 - 18.09 min

Level	ID	Amount/Conc'n	Response Factor
1	1	5.0000	9.8184
2	2	10.000	9.4242
3	3	25.000	8.5127
4	5	100.00	7.8534
5	6	500.00	8.3735
6	7	1000.0	8.2538

Calib Factor: 8.7060 RSD: 8.6%

2-Nitrotoluene RT: 18.69 min Window: 18.44 - 18.94 min

Level	ID	Amount/Conc'n	Response Factor
1	2	10.000	5.7161
2	3	25.000	6.1083
3	5	100.00	5.5956
4	6	500.00	4.7541
5	7	1000.0	4.5735

Calib Factor: 5.3495 RSD: 12 %

4-Nitrotoluene RT: 19.08 min Window: 18.83 - 19.33 min

Level	ID	Amount/Conc'n	Response Factor
1	2	10.000	4.1112
2	3	25.000	5.0372
3	5	100.00	4.4265
4	6	500.00	4.0640
5	7	1000.0	3.8996

Calib Factor: 4.3077 RSD: 10 %

Quanterra / W. Sacramento
Calibration Statistics Report

Calibration ID: [AUG_SV] 109 03AUG98
Instrument ID: LC4 PAGE 4 of 4

File: 03AUG98 Title: 8330H 2.5-1000ng/mL Phenomenex Hypersil C8 SN251486
Curve Type: Line forced thro' zero (AVG RF) Weighting: 1/X sqd
Quant Base: Height

3-Nitrotoluene RT: 19.94 min Window: 19.69 - 20.19 min

Level	ID	Amount/Conc'n	Response Factor
1	2	10.000	5.4292
2	3	25.000	5.9947
3	5	100.00	6.0630
4	6	500.00	5.4304
5	7	1000.0	5.2772

Calib Factor: 5.6389 RSD: 6.4%

Injection Report

Acquired on 4-AUG-1998 at 11:30

Quanterra, Inc (West Sacramento)

Analyst Name : LC4UV
Lims Id : 109LC4UV9050
Comment : 75:25:0/2-22-50:00:50/2" H2O:2%THF/MeOH:Methanol (Curve)
Method Title : Var-LC5500 UV Hypersil-C8 150x4.6mm 5um SN251486 8330M
Sample Name : E070898F 25/100ng/mL STD=I
Sample Id :
Sample Type : Standard Amount=1.00000
Bottle No : 8

PEAK INFORMATION

Peak	RT mins	RT Exp	Hght uV	ppb	Peak name	Width
1	3.893	3.893	295	25.00000	HMX	7.5
2	6.551	6.551	523	25.00000	1,3,5-Trinitrobenzene	11.7
3	7.031	7.031	231	25.00000	RDX	13.3
4	8.569	8.569	1831	100.00000	2,4-Dinitrofluorobenzene(Surr)	12.8
5	9.547	9.547	540	25.00000	1,3-Dinitrobenzene	12.8
6	11.360	11.360	280	25.00000	Nitrobenzene	12.8
7	12.836	12.836	343	25.00000	2,4,6-Trinitrotoluene (TNT)	12.8
8	14.222	14.222	343	25.00000	TETRYL	12.8
9	15.573	15.573	345	25.00000	2,4-Dinitrotoluene	16.5
10	16.782	16.782	215	25.00000	2,6-Dinitrotoluene	16.5
11	17.449	17.449	263	25.00000	2-AM-4,6-Dinitrotoluene	17.6
12	18.116	18.116	233	25.00000	4-AM-2,6-Dinitrotoluene	17.1
13	18.844	18.844	176	25.00000	2-Nitrotoluene	19.7
14	19.262	19.262	142	25.00000	4-Nitrotoluene	17.1A
15	20.107	20.107	156	25.00000	3-Nitrotoluene	15.5

Totals		
Unknowns	0	0.00000
Named	5916	450.00000
Grand Total	5916	450.00000

See Reintegration 8-4-98

ANALYSIS SUMMARY

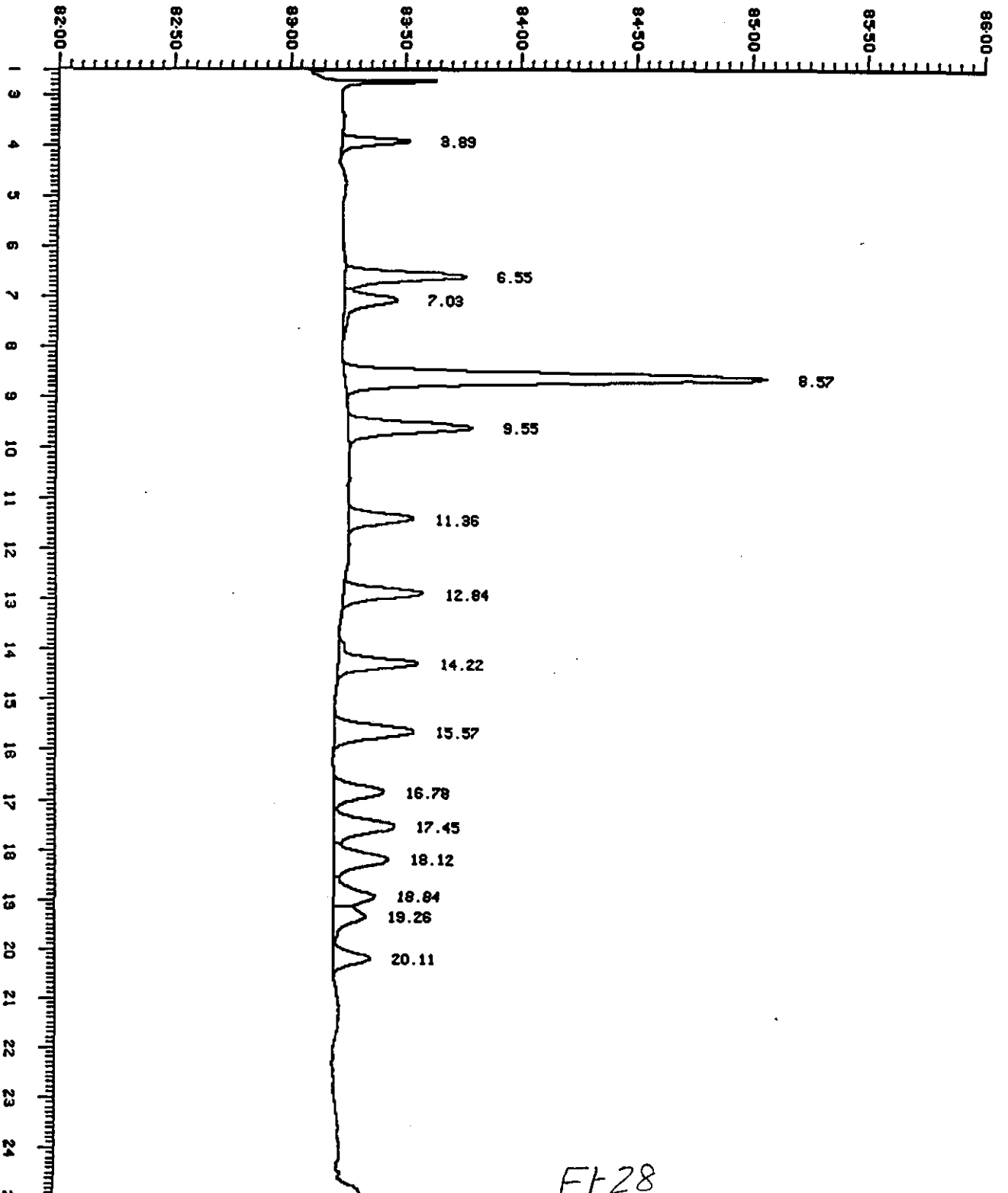
Method..... 8330 HY
Run sequence..... EXP4HY
Calibration..... 03AUG98
External standard calibration using height
Calibration last modified on 4-AUG-1998 at 16:12
Calibration initialised on 12-DEC-1997 at 21:47
Method last modified on 4-AUG-1998 at 15:54

Uncalibrated peaks use user factor (0.0000)

Quanterra, Inc (West Sacramento)

[AUG_SV] 109 4PC04AUG980849,5,1

E070898F 25/100ng/mL STD=I. Amount : 1.000.
Varian 5560/UV9050 @250nm flow=1.5 injection volume=200uL
Acquired on 4-AUG-1998 at 11:30
Reported on 4-AUG-1998 at 16:12
Box 1 (of 1)



Ft28

Injection Report

Acquired on 4-AUG-1998 at 11:30

Quanterra, Inc (West Sacramento)

Analyst Name : LC4UV
Lims Id : 109LC4UV9050
Comment : 75:25:0/2-22-50:00:50/2" H2O:2%THF/MeOH:Methanol (Curve)
Method Title : Var-LC5500 UV Hypersil-C8 150x4.6mm 5um SN251486 8330M
Sample Name : E070898F 25/100ng/mL STD=I
Sample Id :
Sample Type : Standard Amount=1.00000
Bottle No : 8

PEAK INFORMATION

Peak	RT mins	RT Exp	Hght uV	ppb	Peak name	Width
1	3.893	3.893	295	25.00000	HMX	7.5
2	6.551	6.551	523	25.00000	1,3,5-Trinitrobenzene	11.7
3	7.031	7.031	231	25.00000	RDX	13.3
4	8.569	8.569	1831	100.00000	2,4-Dinitrofluorobenzene(Surr)	12.8
5	9.547	9.547	540	25.00000	1,3-Dinitrobenzene	12.8
6	11.360	11.360	280	25.00000	Nitrobenzene	12.8
7	12.836	12.836	343	25.00000	2,4,6-Trinitrotoluene (TNT)	12.8
8	14.222	14.222	343	25.00000	TETRYL	12.8
9	15.573	15.573	345	25.00000	2,4-Dinitrotoluene	16.5
10	16.782	16.782	211	25.00000	2,6-Dinitrotoluene	16.5
11	17.449	17.449	250	25.00000	2-AM-4,6-Dinitrotoluene	17.1
12	18.116	18.116	213	25.00000	4-AM-2,6-Dinitrotoluene	16.5
13	18.844	18.844	153	25.00000	2-Nitrotoluene	17.1
14	19.262	19.262	126	25.00000	4-Nitrotoluene	16.0A
15	20.107	20.107	150	25.00000	3-Nitrotoluene	15.5

Totals		
Unknowns	0	0.00000
Named	5834	450.00000
Grand Total	5834	450.00000

RE-INTEGRATION CODES
1 [illegible] Shape
2 [illegible] Resolution
3 Peak [illegible] Integrated
4 [illegible] Matrix Interference
5 Column Bleed
6 Instrument Noise
7 Other: (reason must be stated)
ALL RE-INTEGRATIONS MUST BE INITIALED, DATED, AND CODED

ANALYSIS SUMMARY

Method..... 8330_HY
Run sequence..... EXP4HY
Calibration..... 03AUG98
External standard calibration using height
Calibration last modified on 4-AUG-1998 at 16:39
Calibration initialised on 12-DEC-1997 at 21:47
Method last modified on 4-AUG-1998 at 16:38

Uncalibrated peaks use user factor (0.0000)

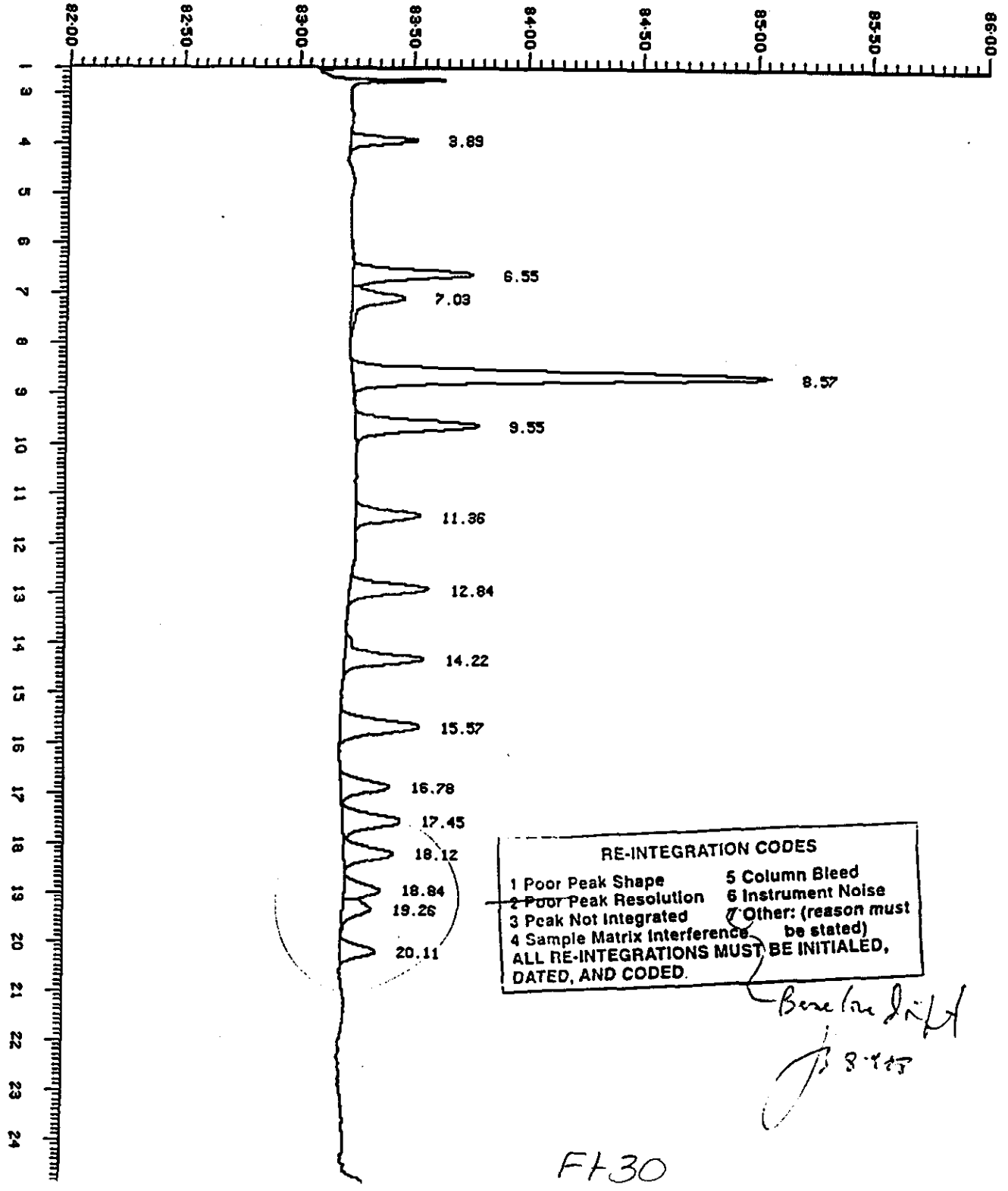
Baseline drift

8-4-98

Quanterra, Inc (West Sacramento)

[AUG_SV] 109 4PC04AUG980849,5,1

E070898F 25/100ng/mL STD=I. Amount : 1.000.
Varian 5560/UV9050 @250nm flow=1.5 injection volume=200uL
Acquired on 4-AUG-1998 at 11:30
Reported on 4-AUG-1998 at 16:39
Box 1 (of 1)



RE-INTEGRATION CODES

1 Poor Peak Shape
2 Poor Peak Resolution
3 Peak Not Integrated
4 Sample Matrix Interference
5 Column Bleed
6 Instrument Noise
7 Other: (reason must be stated)

ALL RE-INTEGRATIONS MUST BE INITIALED, DATED, AND CODED.

Baseline drift

8-4-98

FH30

Injection Report

Acquired on 4-AUG-1998 at 12:06

Quanterra, Inc (West Sacramento)

Analyst Name : LC4UV
Lims Id : 109LC4UV9050
Comment : 75:25:0/2-22-50:00:50/2" H2O:2%THF/MeOH:Methanol (Curve)
Method Title : Var-LC5500 UV Hypersil-C8 150x4.6mm 5um SN251486 8330M
Sample Name : E070898C 2.5/5.0ng/ml STD=I
Sample Id :
Sample Type : Standard Amount=1.00000
Bottle No : 8

PEAK INFORMATION

Peak	RT mins	RT Exp	Hght uV	ppb	Peak name	Width
1	3.867	3.769	47	3.93875	HMX	8.5
2	6.542	6.493	68	3.17024	1,3,5-Trinitrobenzene	11.2
3	6.969	6.907	35	3.96672	RDX	11.2
4	8.551	8.476	95	5.28418	2,4-Dinitrofluorobenzene(Surr)	13.9
5	9.538	9.444	56	2.58955	1,3-Dinitrobenzene	12.8
6	11.333	11.271	29	2.65410	Nitrobenzene	12.8
7	12.809	12.729	43	3.22501	2,4,6-Trinitrotoluene (TNT)	12.8
8	14.196	14.078	36	2.66488	TETRYL	12.3
9	15.564	15.413	35	2.46364	2,4-Dinitrotoluene	14.9
10	16.711	16.576	16	1.97965	2,6-Dinitrotoluene	12.3
11	17.431	0.000	26	0.00000		14.9
12	18.071	17.836	34	3.85652	4-AM-2,6-Dinitrotoluene	19.7
13	20.062	19.942	13	2.34294	3-Nitrotoluene	9.1
14	24.747	0.000	60	0.00000		16.0

Totals		
Unknowns	86	0.00000
Named	507	38.13620
Grand Total	593	38.13620

ANALYSIS SUMMARY

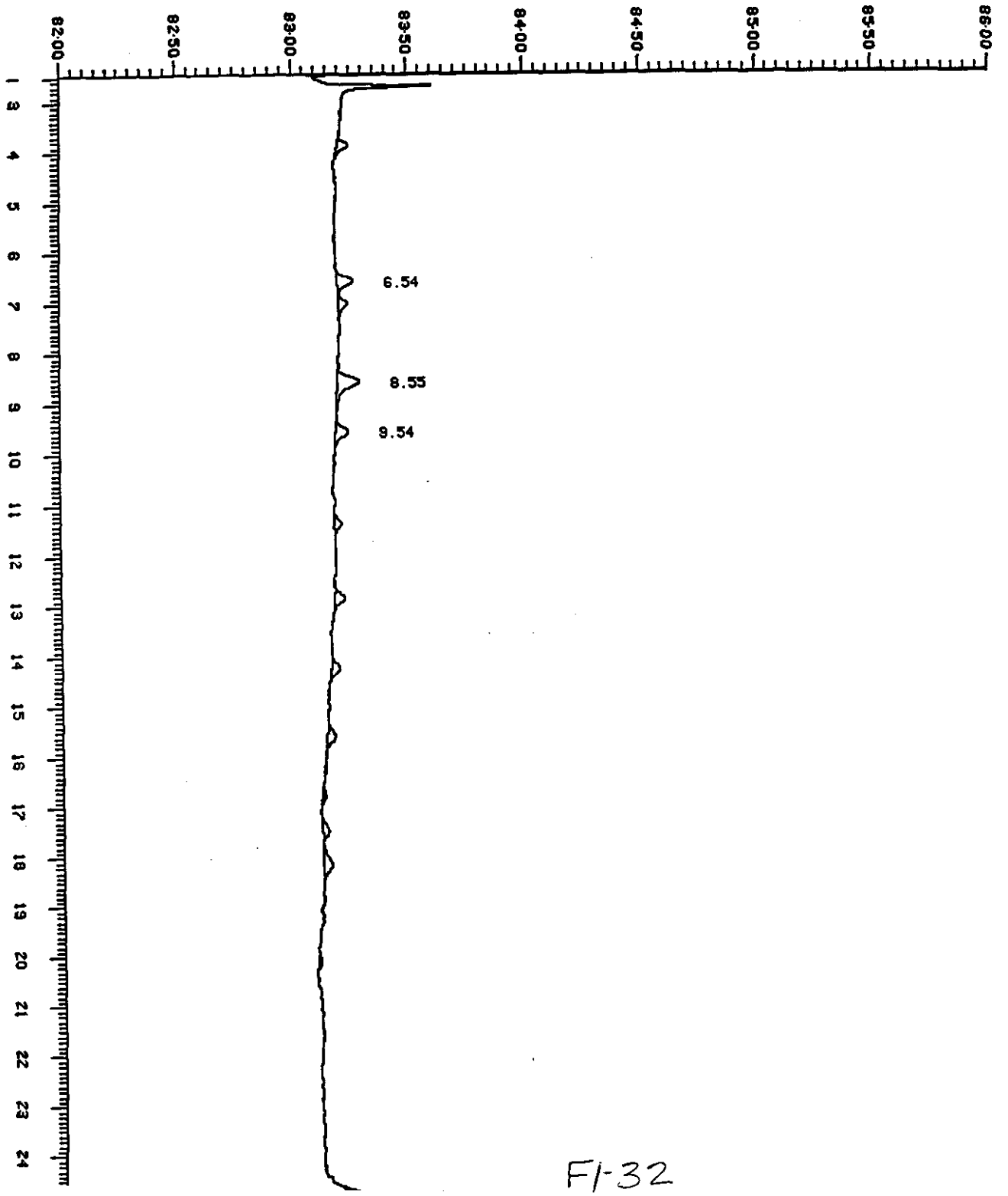
Method..... 8330 HY
Run sequence..... EXP4HY
Calibration..... 03AUG98
External standard calibration using height
Calibration last modified on 4-AUG-1998 at 15:50
Calibration initialised on 12-DEC-1997 at 21:47
Method last modified on 4-AUG-1998 at 15:54

Uncalibrated peaks use user factor (0.0000)

Quanterra, Inc (West Sacramento)

[AUG_SV] 109 4PC04AUG980849,6,1

E070898C 2.5/5.0ng/ml STD=I. Amount : 1.000.
Varian 5560/UV9050 @250nm flow=1.5 injection volume=200uL
Acquired on 4-AUG-1998 at 12:06
Reported on 4-AUG-1998 at 15:55
Box 1 (of 1)



Injection Report

Acquired on 4-AUG-1998 at 12:43

Quanterra, Inc (West Sacramento)

Analyst Name : LC4UV
Lims Id : 109LC4UV9050
Comment : 75:25:0/2-22-50:00:50/2" H2O:2%THF/MeOH:Methanol (Curve)
Method Title : Var-LC5500 UV Hypersil-C8 150x4.6mm 5um SN251486 8330M
Sample Name : E070898D 5.0/25ng/mL STD=I
Sample Id :
Sample Type : Standard Amount=1.00000
Bottle No : 8

PEAK INFORMATION

Peak	RT mins	RT Exp	Hght uV	ppb	Peak name	Width
1	3.849	3.769	59	4.90791	HMX	8.5
2	6.542	6.493	95	4.41476	1,3,5-Trinitrobenzene	11.7
3	6.978	6.907	43	4.80423	RDX	12.3
4	8.542	8.476	422	23.45408	2,4-Dinitrofluorobenzene(Surr)	12.8
5	9.538	9.444	94	4.37793	1,3-Dinitrobenzene	13.9
6	10.596	0.000	10	0.00000		9.6
7	11.316	11.271	48	4.42673	Nitrobenzene	12.3
8	12.800	12.729	60	4.48856	2,4,6-Trinitrotoluene (TNT)	12.8
9	14.196	14.078	70	5.27121	TETRYL	12.3
10	15.538	15.413	58	4.15513	2,4-Dinitrotoluene	17.1
11	16.711	16.576	43	5.19907	2,6-Dinitrotoluene	16.0
12	17.378	17.178	60	5.85455	2-AM-4,6-Dinitrotoluene	18.1
13	18.000	17.836	49	5.63678	4-AM-2,6-Dinitrotoluene	19.2
14	18.782	18.687	29	5.30190	2-Nitrotoluene	10.7A
15	19.218	19.080	30	6.84271	4-Nitrotoluene	21.9
16	20.062	19.942	31	5.40084	3-Nitrotoluene	13.9
17	20.747	0.000	11	0.00000		8.5
18	24.720	0.000	46	0.00000		15.5

Totals		
Unknowns	67	0.00000
Named	1192	94.53639
Grand Total	1259	94.53639

ANALYSIS SUMMARY

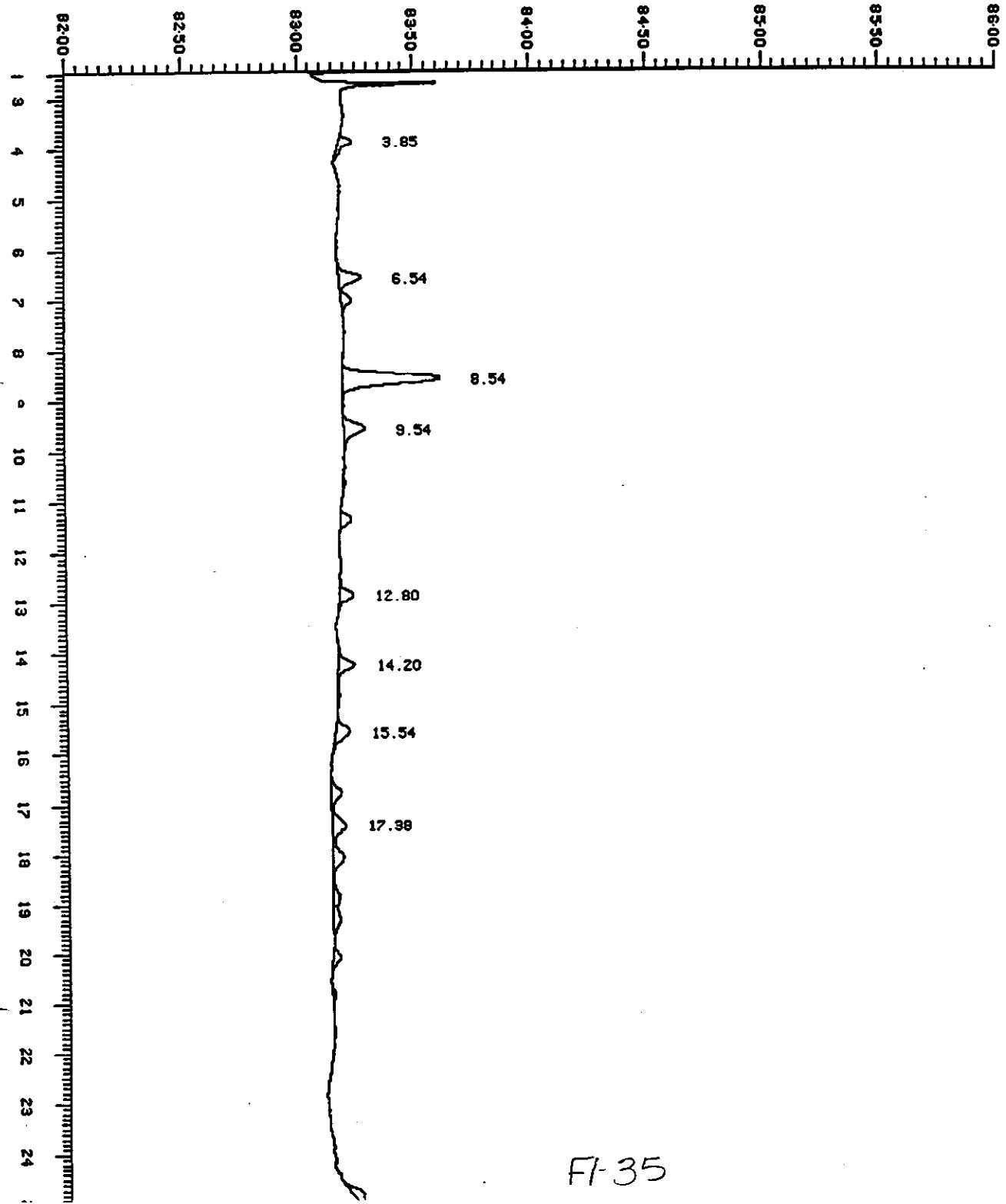
Method..... 8330_HY
Run sequence..... EXP4HY
Calibration..... 03AUG98
External standard calibration using height
Calibration last modified on 4-AUG-1998 at 15:50
Calibration initialised on 12-DEC-1997 at 21:47
Method last modified on 4-AUG-1998 at 15:54

Uncalibrated peaks use user factor (0.0000)

Quanterra, Inc (West Sacramento)

[AUG_SV] 109 4PC04AUG980849,7,1

E070898D 5.0/25ng/mL STD=I. Amount : 1.000.
Varian 5560/UV9050 @250nm flow=1.5 injection volume=200uL
Acquired on 4-AUG-1998 at 12:43
Reported on 4-AUG-1998 at 15:56
Box 1 (of 1)



F1-35

Injection Report

Acquired on 4-AUG-1998 at 13:19

Quanterra, Inc (West Sacramento)

Analyst Name : LC4UV
Lims Id : 109LC4UV9050
Comment : 75:25:0/2-22-50:00:50/2" H2O:2%THF/MeOH:Methanol (Curve)
Method Title : Var-LC5500 UV Hypersil-C8 150x4.6mm 5um SN251486 8330M
Sample Name : E070898E 10/50ng/ml STD=I
Sample Id :
Sample Type : Standard Amount=1.00000
Bottle No : 8

PEAK INFORMATION

Peak	RT mins	RT Exp	Hght uV	ppb	Peak name	Width
1	3.849	3.769	123	10.23578	HMX	8.0
2	6.524	6.493	202	9.43180	1,3,5-Trinitrobenzene	11.7
3	6.969	6.907	86	9.66013	RDX	11.7
4	8.524	8.476	914	50.75963	2,4-Dinitrofluorobenzene(Surr)	12.8
5	9.484	9.444	200	9.26140	1,3-Dinitrobenzene	13.3
6	11.307	11.271	109	10.07566	Nitrobenzene	12.3
7	12.782	12.729	126	9.36288	2,4,6-Trinitrotoluene (TNT)	12.8
8	14.151	14.078	131	9.83814	TETRYL	12.3
9	15.493	15.413	141	10.05330	2,4-Dinitrotoluene	16.0
10	16.667	16.576	80	9.70008	2,6-Dinitrotoluene	16.5
11	17.333	17.178	98	9.53755	2-AM-4,6-Dinitrotoluene	16.5
12	17.956	17.836	94	10.82101	4-AM-2,6-Dinitrotoluene	19.7
13	18.773	18.687	57	10.37658	2-Nitrotoluene	18.1
14	19.164	19.080	41	9.30460	4-Nitrotoluene	20.3
15	20.009	19.942	54	9.55937	3-Nitrotoluene	14.9
16	20.684	0.000	13	0.00000		6.9
17	24.773	0.000	61	0.00000		16.5

Totals		
Unknowns	74	0.00000
Named	2456	187.97791
Grand Total	2530	187.97791

ANALYSIS SUMMARY

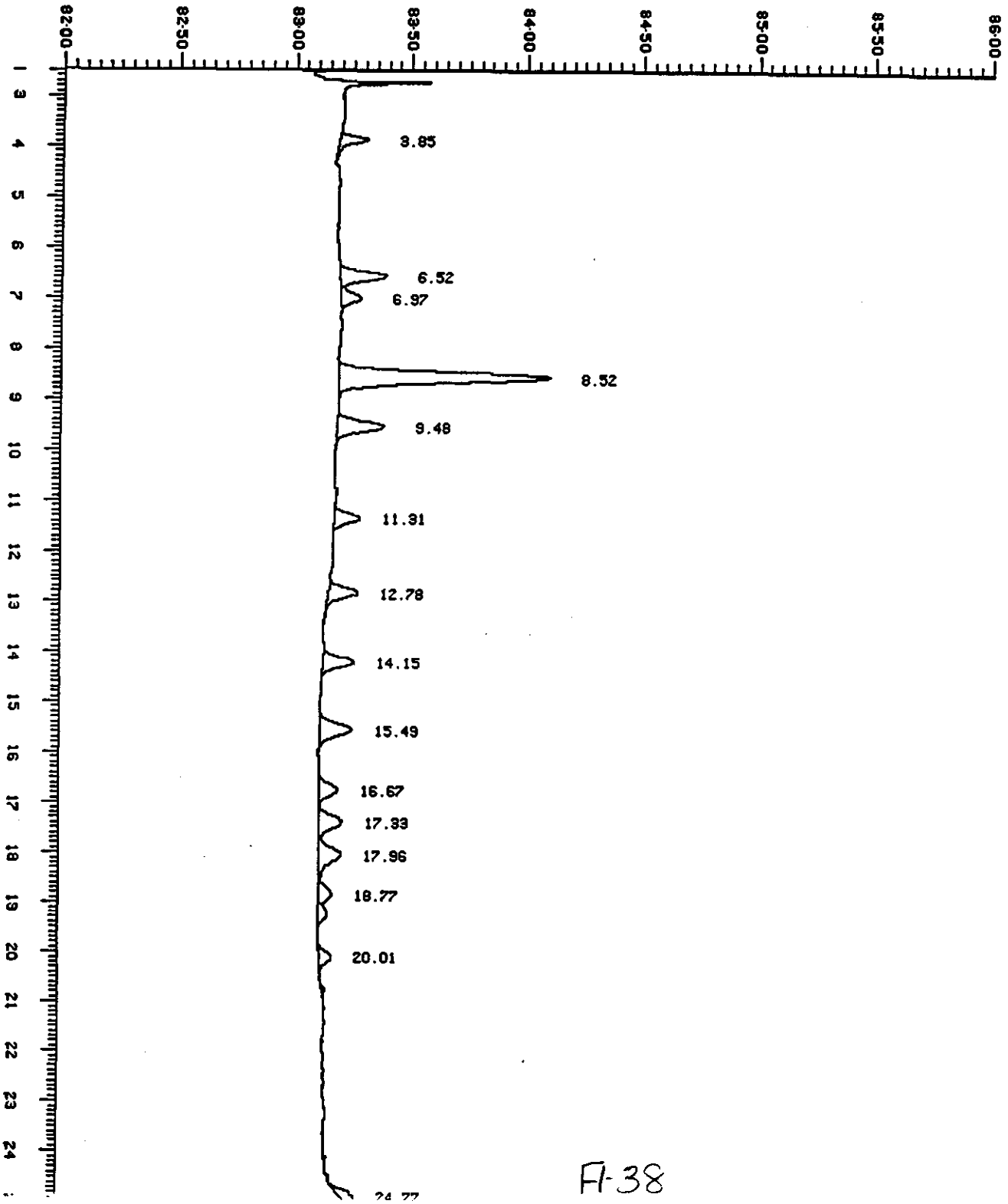
Method..... 8330 HY
Run sequence..... EXP4HY
Calibration..... 03AUG98
External standard calibration using height
Calibration last modified on 4-AUG-1998 at 15:50
Calibration initialised on 12-DEC-1997 at 21:47
Method last modified on 4-AUG-1998 at 15:54

Uncalibrated peaks use user factor (0.0000)

Quanterra, Inc (West Sacramento)

[AUG_SV] 109 4PC04AUG980849,8,1

E070898E 10/50ng/ml STD=I. Amount : 1.000.
Varian 5560/UV9050 @250nm flow=1.5 injection volume=200uL
Acquired on 4-AUG-1998 at 13:19
Reported on 4-AUG-1998 at 15:56
Box 1 (of 1)



A1-38

Injection Report

Acquired on 4-AUG-1998 at 13:55

Quanterra, Inc (West Sacramento)

Analyst Name : LC4UV
Lims Id : 109LC4UV9050
Comment : 75:25:0/2-22-50:00:50/2" H2O:2%THF/MeOH:Methanol (Curve)
Method Title : Var-LC5500 UV Hypersil-C8 150x4.6mm 5um SN251486 8330M
Sample Name : E070898G 100/150 STD=I
Sample Id :
Sample Type : Standard Amount=1.00000
Bottle No : 8

PEAK INFORMATION

Peak	RT mins	RT Exp	Hght uV	ppb	Peak name	Width
1	3.804	3.769	1131	94.28961	HMX	8.0
2	6.489	6.493	2070	96.59766	1,3,5-Trinitrobenzene	11.7
3	6.907	6.907	814	91.05035	RDX	13.3
4	8.471	8.476	2625	145.84637	2,4-Dinitrofluorobenzene(Surr)	12.8
5	9.431	9.444	2058	95.48882	1,3-Dinitrobenzene	13.3
6	11.244	11.271	1071	99.19094	Nitrobenzene	13.3
7	12.729	12.729	1370	101.77075	2,4,6-Trinitrotoluene (TNT)	12.8
8	14.098	14.078	1336	100.02416	TETRYL	13.3
9	15.413	15.413	1429	101.91945	2,4-Dinitrotoluene	16.0
10	16.569	16.576	804	98.04065	2,6-Dinitrotoluene	16.0
11	17.182	17.178	969	93.84743	2-AM-4,6-Dinitrotoluene	17.1
12	17.840	17.836	785	90.17307	4-AM-2,6-Dinitrotoluene	17.1
13	18.640	18.687	560	101.57875	2-Nitrotoluene	19.2
14	19.031	19.080	443	100.18039	4-Nitrotoluene	19.2A
15	19.911	19.942	606	106.75216	3-Nitrotoluene	16.0
16	21.591	0.000	23	0.00000		48.5
17	24.747	0.000	57	0.00000		16.5

Totals		
Unknowns	80	0.00000
Named	18071	1516.75061
Grand Total	18151	1516.75061

ANALYSIS SUMMARY

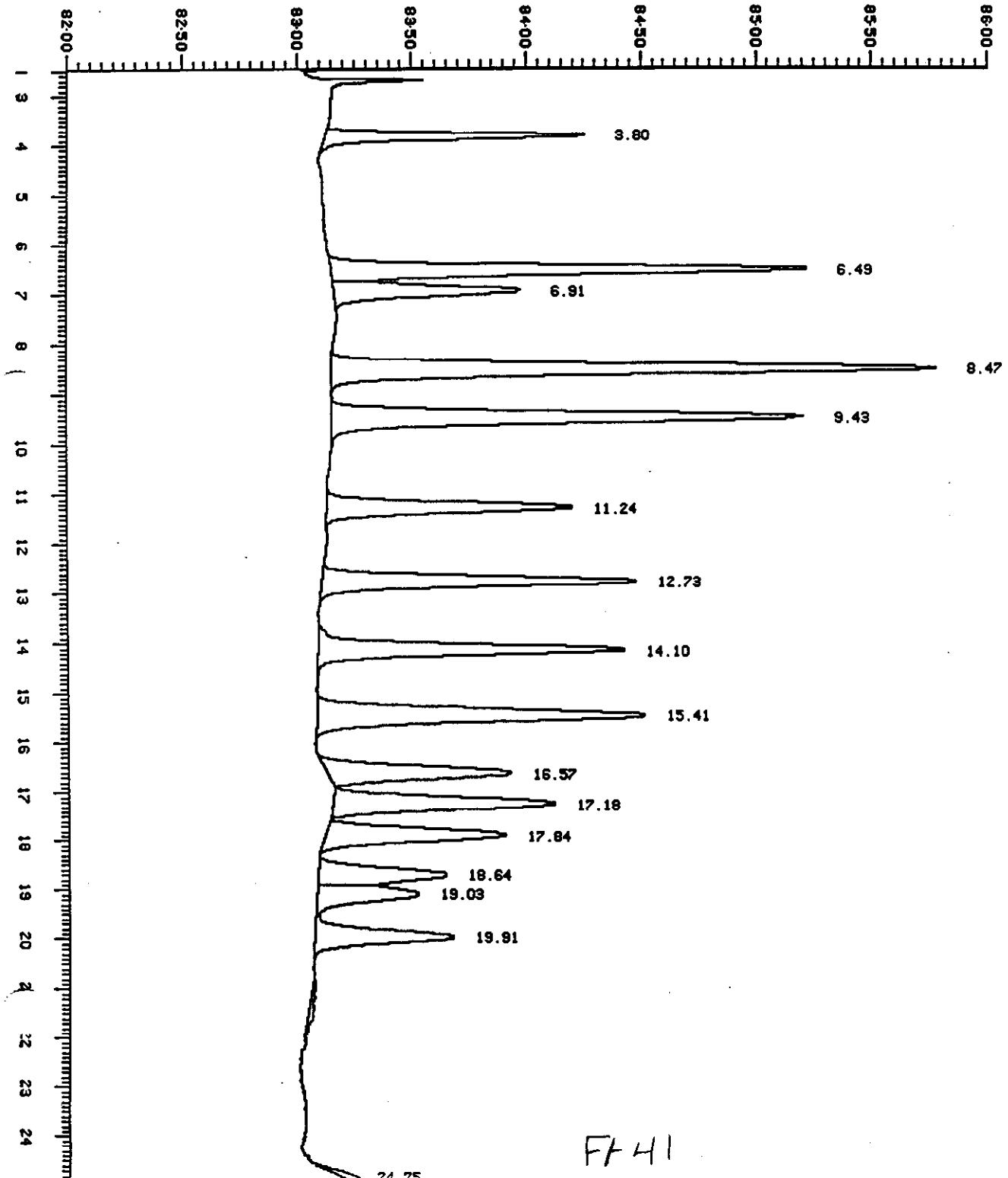
Method..... 8330 HY
Run sequence..... EXP4HY
Calibration..... 03AUG98
External standard calibration using height
Calibration last modified on 4-AUG-1998 at 15:50
Calibration initialised on 12-DEC-1997 at 21:47
Method last modified on 4-AUG-1998 at 15:54

Uncalibrated peaks use user factor (0.0000)

Quanterra, Inc (West Sacramento)

[AUG_SV] 109 4PC04AUG980849,9,1

E070898G 100/150 STD=I. Amount : 1.000.
Varian 5560/UV9050 @250nm flow=1.5 injection volume=200uL
Acquired on 4-AUG-1998 at 13:55
Reported on 4-AUG-1998 at 15:56
Box 1 (of 1)



F41

Injection Report

Acquired on 4-AUG-1998 at 14:31

Quanterra, Inc (West Sacramento)

Analyst Name : LC4UV
Lims Id : 109LC4UV9050
Comment : 75:25:0/2-22-50:00:50/2" H2O:2%THF/MeOH:Methanol (Curve)
Method Title : Var-LC5500 UV Hypersil-C8 150x4.6mm 5um SN251486 8330M
Sample Name : E070898H 500 ng/mL 8330 Mix STD=I
Sample Id :
Sample Type : Standard Amount=1.00000
Bottle No : 8

PEAK INFORMATION

Peak	RT mins	RT Exp	Hght uV	ppb	Peak name	Width
1	3.467	0.000	14	0.00000		6.9
2	3.769	3.769	6251	521.19122	HMX	7.5
3	6.507	6.493	11941	557.29205	1,3,5-Trinitrobenzene	10.1
4	6.924	6.907	4756	531.95862	RDX	11.2
5	8.480	8.476	18	1.00468	2,4-Dinitrofluorobenzene(Surr)	10.7
6	9.467	9.444	11898	552.18610	1,3-Dinitrobenzene	12.3
7	11.298	11.271	5658	523.81244	Nitrobenzene	12.3
8	12.747	12.729	7206	535.29248	2,4,6-Trinitrotoluene (TNT)	12.8
9	13.556	0.000	40	0.00000		12.3
10	14.089	14.078	6529	488.65332	TETRYL	13.3
11	15.431	15.413	7648	545.52191	2,4-Dinitrotoluene	16.0
12	16.596	16.576	4274	521.40314	2,6-Dinitrotoluene	16.5
13	17.200	17.178	5364	519.43842	2-AM-4,6-Dinitrotoluene	17.6
14	17.858	17.836	4187	480.72772	4-AM-2,6-Dinitrotoluene	17.6
15	18.720	18.687	2377	431.51663	2-Nitrotoluene	18.1
16	19.102	19.080	2032	459.88449	4-Nitrotoluene	18.1A
17	19.964	19.942	2715	478.07458	3-Nitrotoluene	16.0
18	21.520	0.000	10	0.00000		13.9
19	24.036	0.000	12	0.00000		9.1
20	24.373	0.000	18	0.00000		4.3A
21	24.889	0.000	65	0.00000		4.8

Totals		
Unknowns	160	0.00000
Named	82855	7147.95752
Grand Total	83014	7147.95752

ANALYSIS SUMMARY

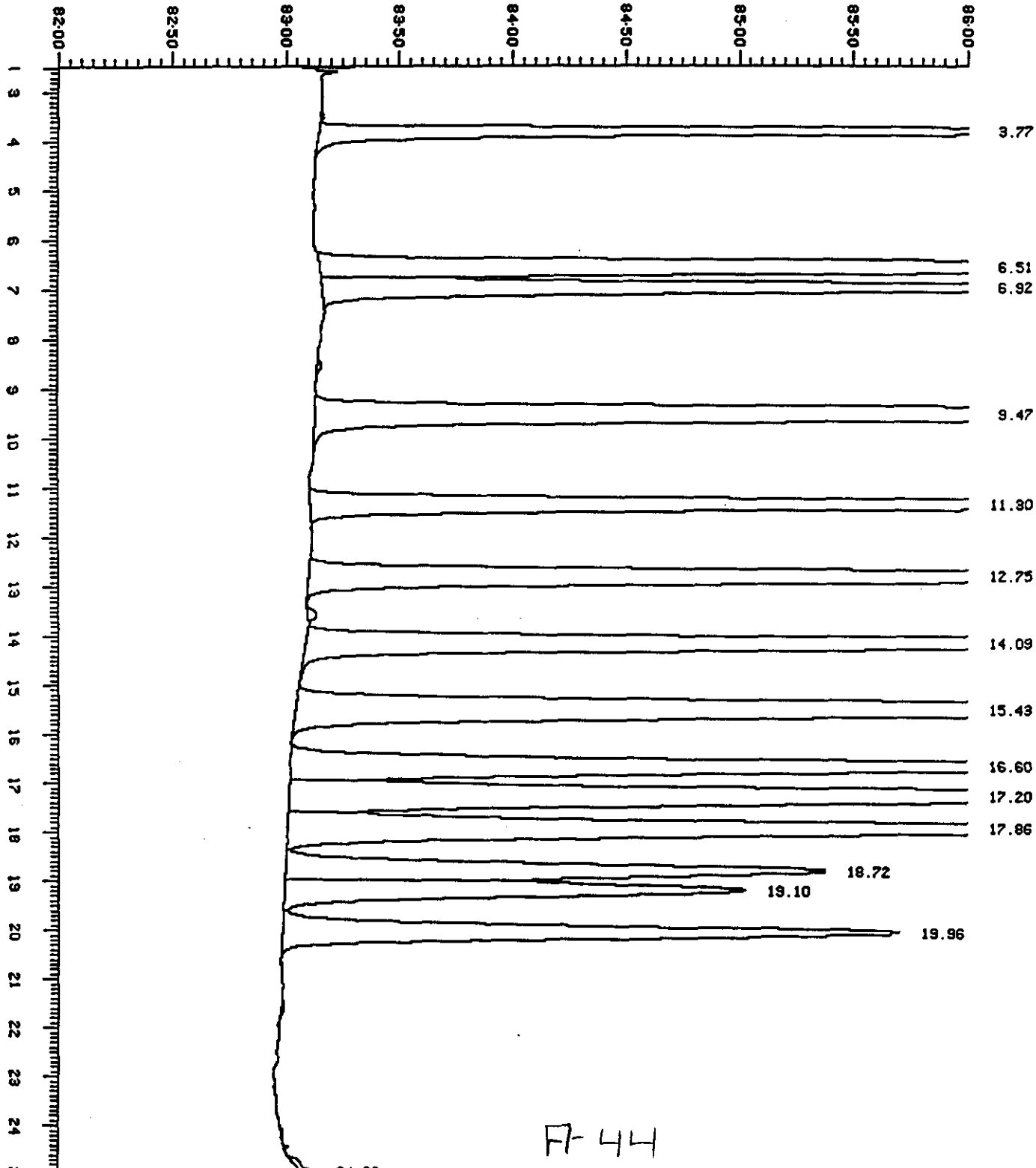
Method..... 8330_HY
Run sequence..... EXP4HY
Calibration..... 03AUG98
External standard calibration using height
Calibration last modified on 4-AUG-1998 at 15:50
Calibration initialised on 12-DEC-1997 at 21:47
Method last modified on 4-AUG-1998 at 15:54

Uncalibrated peaks use user factor (0.0000)

Quanterra, Inc (West Sacramento)

[AUG_SV] 109 4PC04AUG980849,10,1

E070898H 500 ng/mL 8330 Mix STD=I. Amount : 1.000.
Varian 5560/UV9050 @250nm flow=1.5 injection volume=200uL
Acquired on 4-AUG-1998 at 14:31
Reported on 4-AUG-1998 at 15:56
Box 1 (of 1)



A-44

Injection Report

Acquired on 4-AUG-1998 at 15:08

Quanterra, Inc (West Sacramento)

Analyst Name : LC4UV
Lims Id : 109LC4UV9050
Comment : 75:25:0/2-22-50:00:50/2" H2O:2%THF/MeOH:Methanol (Curve)
Method Title : Var-LC5500 UV Hypersil-C8 150x4.6mm 5um SN251486 8330M
Sample Name : E070898I 1000 ng/mL 8330 Mix STD=I
Sample Id :
Sample Type : Standard Amount=1.00000
Bottle No : 8

PEAK INFORMATION

Peak	RT mins	RT Exp	Hght uV	ppb	Peak name	Width
1	3.369	0.000	11	0.00000		5.9
2	3.751	3.769	12312	1026.58838	HMX	7.5
3	5.307	0.000	26	0.00000		8.5
4	6.489	6.493	23916	1116.17505	1,3,5-Trinitrobenzene	10.7
5	6.898	6.907	9538	1066.84058	RDX	11.2
6	9.440	9.444	23711	1100.43970	1,3-Dinitrobenzene	11.7
7	11.271	11.271	11128	1030.19116	Nitrobenzene	11.7
8	12.720	12.729	14256	1059.00940	2,4,6-Trinitrotoluene (TNT)	12.8
9	13.511	0.000	92	0.00000		12.8
10	14.062	14.078	12798	957.81213	TETRYL	13.3
11	15.404	15.413	15187	1083.23999	2,4-Dinitrotoluene	16.0
12	16.569	16.576	8445	1030.26306	2,6-Dinitrotoluene	17.1
13	17.164	17.178	10555	1022.03387	2-AM-4,6-Dinitrotoluene	17.6
14	17.822	17.836	8254	947.71387	4-AM-2,6-Dinitrotoluene	18.1
15	18.693	18.687	4573	830.23987	2-Nitrotoluene	18.7
16	19.093	19.080	3900	882.55566	4-Nitrotoluene	17.1A
17	19.947	19.942	5277	929.16205	3-Nitrotoluene	15.5
18	20.916	0.000	16	0.00000		14.4
19	21.538	0.000	20	0.00000		16.0
20	24.871	0.000	115	0.00000		4.3

Totals		
Unknowns	279	0.00000
Named	163850	14082.26563
Grand Total	164129	14082.26563

ANALYSIS SUMMARY

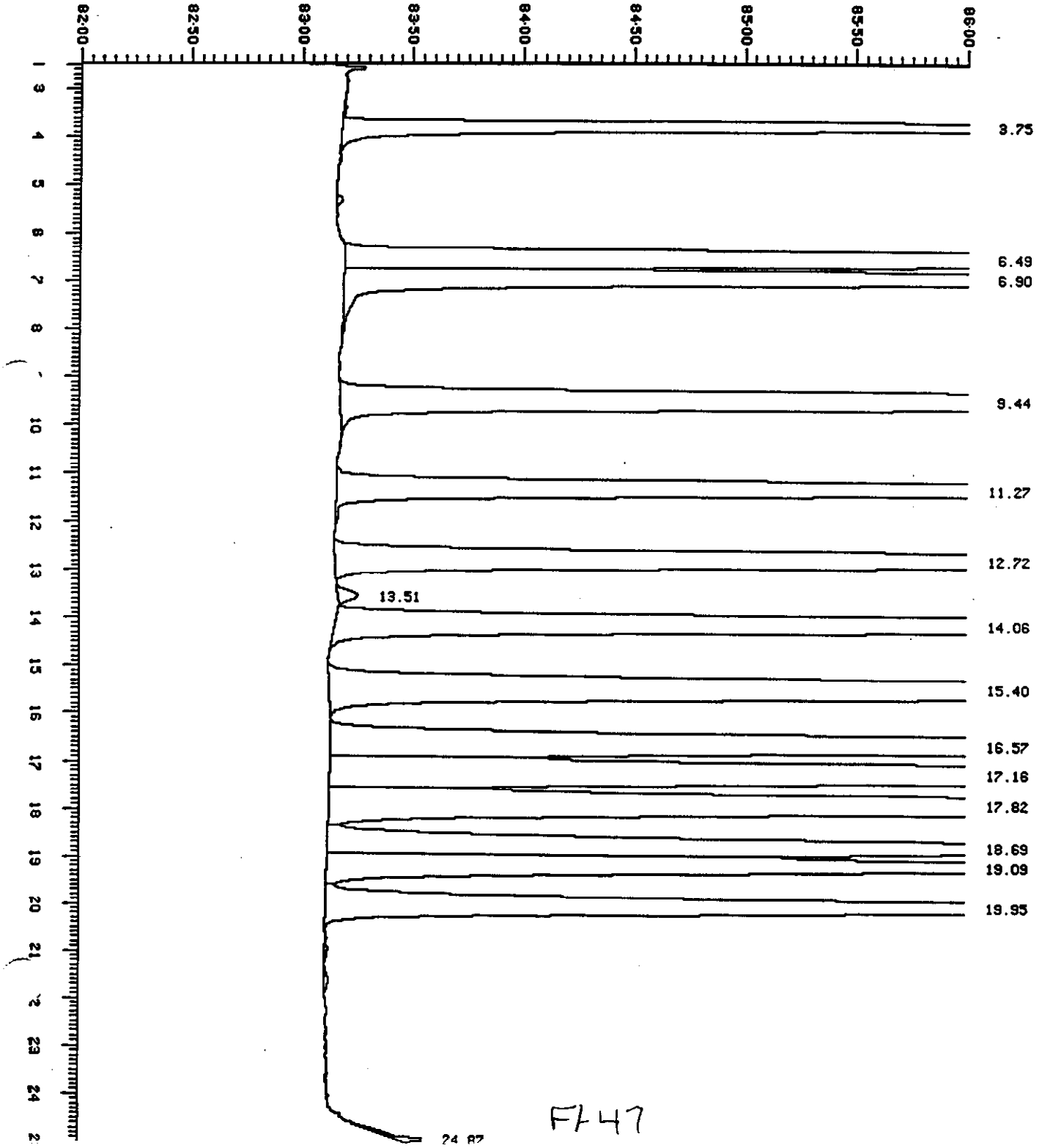
Method..... 8330 HY
Run sequence..... EXP4HY
Calibration..... 03AUG98
External standard calibration using height
Calibration last modified on 4-AUG-1998 at 15:50
Calibration initialised on 12-DEC-1997 at 21:47
Method last modified on 4-AUG-1998 at 15:54

Uncalibrated peaks use user factor (0.0000)

Quanterra, Inc (West Sacramento)

[AUG_SV] 109 4PC04AUG980849,11,1

E070898I 1000 ng/mL 8330 Mix STD=I. Amount : 1.000.
Varian 5560/UV9050 @250nm flow=1.5 injection volume=200uL
Acquired on 4-AUG-1998 at 15:08
Reported on 4-AUG-1998 at 15:56
Box 1 (of 1)



F147

Injection Report

Acquired on 4-AUG-1998 at 15:44

Quanterra, Inc (West Sacramento)

Analyst Name : LC4UV
Lims Id : 109LC4UV9050
Comment : 75:25:0/2-22-50:00:50/2" H2O:2%THF/MeOH:Methanol (Curve)
Method Title : Var-LC5500 UV Hypersil-C8 150x4.6mm 5um SN251486 8330M
Sample Name : Water blank
Sample Id :
Sample Type : Sample Amount=1.00000
Bottle No : 8

PEAK INFORMATION

Peak	RT mins	RT Exp	Hght uV	ppb	Peak name	Width
1	3.467	0.000	10	0.00000		4.3
2	20.942	0.000	13	0.00000		14.4
3	24.782	0.000	54	0.00000		16.0

Totals		
Unknowns	78	0.00000
Named	0	0.00000
Grand Total	78	0.00000

ANALYSIS SUMMARY

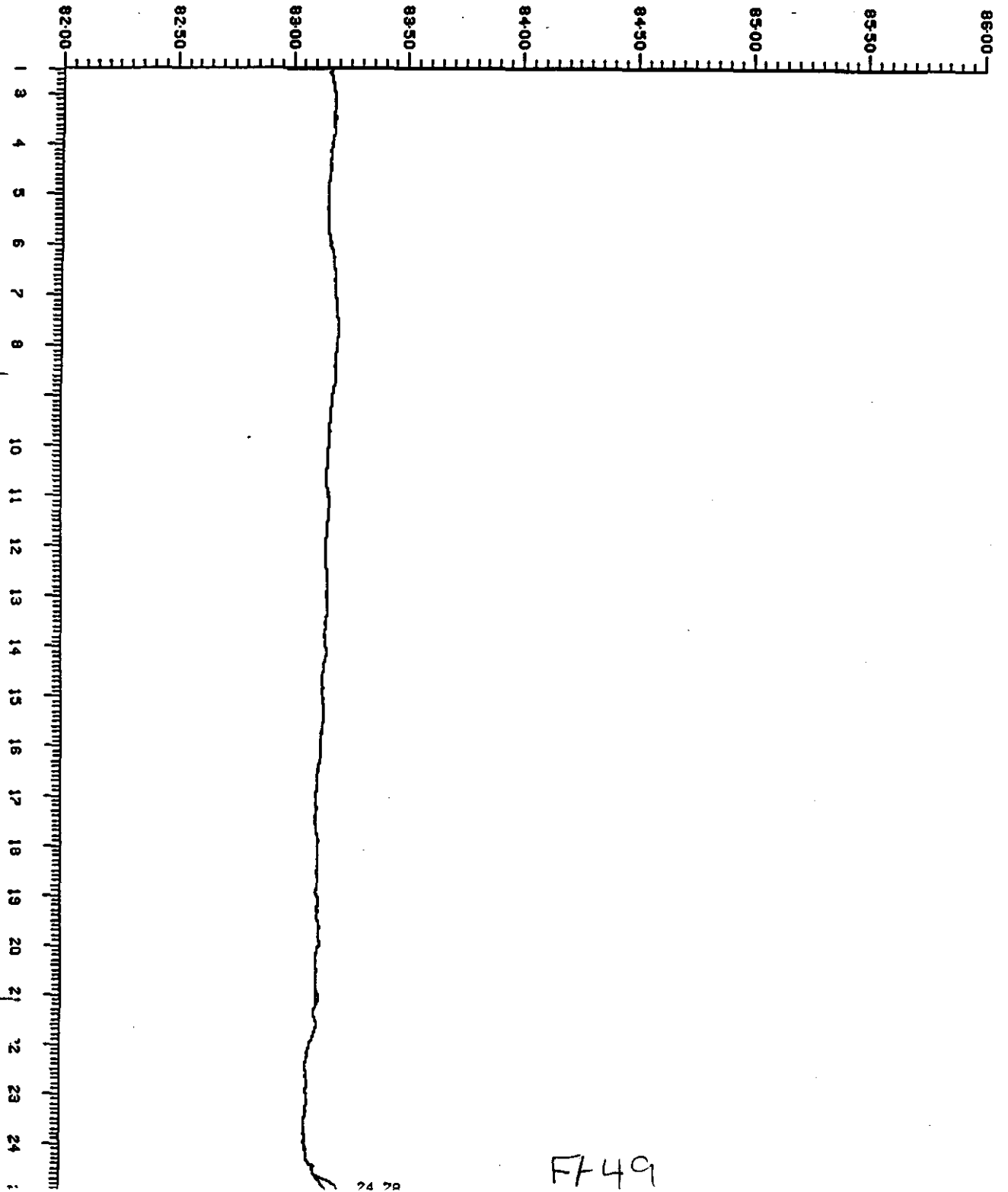
Method..... 8330 HY
Run sequence..... EXP4HY
Calibration..... 03AUG98
External standard calibration using height
Calibration last modified on 4-AUG-1998 at 16:07
Calibration initialised on 12-DEC-1997 at 21:47
Method last modified on 4-AUG-1998 at 15:54

Uncalibrated peaks use user factor (0.0000)

Quanterra, Inc (West Sacramento)

[AUG_SV] 109 4PC04AUG980849,12,1

Water blank. Amount : 1.000.
Varian 5560/UV9050 @250nm flow=1.5 injection volume=200uL
Acquired on 4-AUG-1998 at 15:44
Reported on 4-AUG-1998 at 16:09
Box 1 (of 1)



F-49

Continuing Calibration

Injection Report

Acquired on 4-AUG-1998 at 16:47

Quanterra, Inc (West Sacramento)

Analyst Name : LC4UV
Lims Id : 109LC4UV9050
Comment : 75:25:0/2-22-50:00:50/2"- H2O:2%THF/MeOH:Methanol (Analysis)
Method Title : Var-LC5500 UV Hypersil-C8 150x4.6mm 5um SN251486 8330M
Sample Name : E070898F 25/100 8330MIX CV
Sample Id :
Sample Type : Standard Amount=1.00000
Bottle No : 8

PEAK INFORMATION

Peak	RT mins	RT Exp	Hght uV	ppb	Peak name	Width
1	3.804	3.769	290	24.19226	HMX	8.0
2	6.498	6.493	510	23.80516	1,3,5-Trinitrobenzene	11.2
3	6.889	6.907	205	22.94697	RDX	13.3
4	8.480	8.476	1796	99.80174	2,4-Dinitrofluorobenzene(Surr)	13.3
5	9.422	9.444	514	23.86339	1,3-Dinitrobenzene	13.3
6	11.244	11.271	277	25.63266	Nitrobenzene	12.8
7	12.738	12.729	328	24.36662	2,4,6-Trinitrotoluene (TNT)	12.8
8	14.062	14.078	331	24.80697	TETRYL	13.3
9	15.387	15.413	349	24.86063	2,4-Dinitrotoluene	16.5
10	16.551	16.576	190	22.81024	2,6-Dinitrotoluene	17.1
11	17.147	17.178	234	22.29460	2-AM-4,6-Dinitrotoluene	17.6
12	17.822	17.836	190	21.85193	4-AM-2,6-Dinitrotoluene	18.1
13	18.667	18.687	141	26.30777	2-Nitrotoluene	18.1
14	19.022	19.080	110	25.64306	4-Nitrotoluene	20.3A
15	19.902	19.942	155	27.52489	3-Nitrotoluene	16.0
16	24.382	0.000	47	0.00000		10.1

Totals		
Unknowns	47	0.00000
Named	5621	440.70889
Grand Total	5668	440.70889

ANALYSIS SUMMARY

Method..... 8330 HY
Run sequence..... EXP4HY1
Calibration..... 03AUG98
External standard calibration using height
Calibration last modified on 4-AUG-1998 at 17:12
Calibration initialised on 12-DEC-1997 at 21:47
Method last modified on 4-AUG-1998 at 16:40

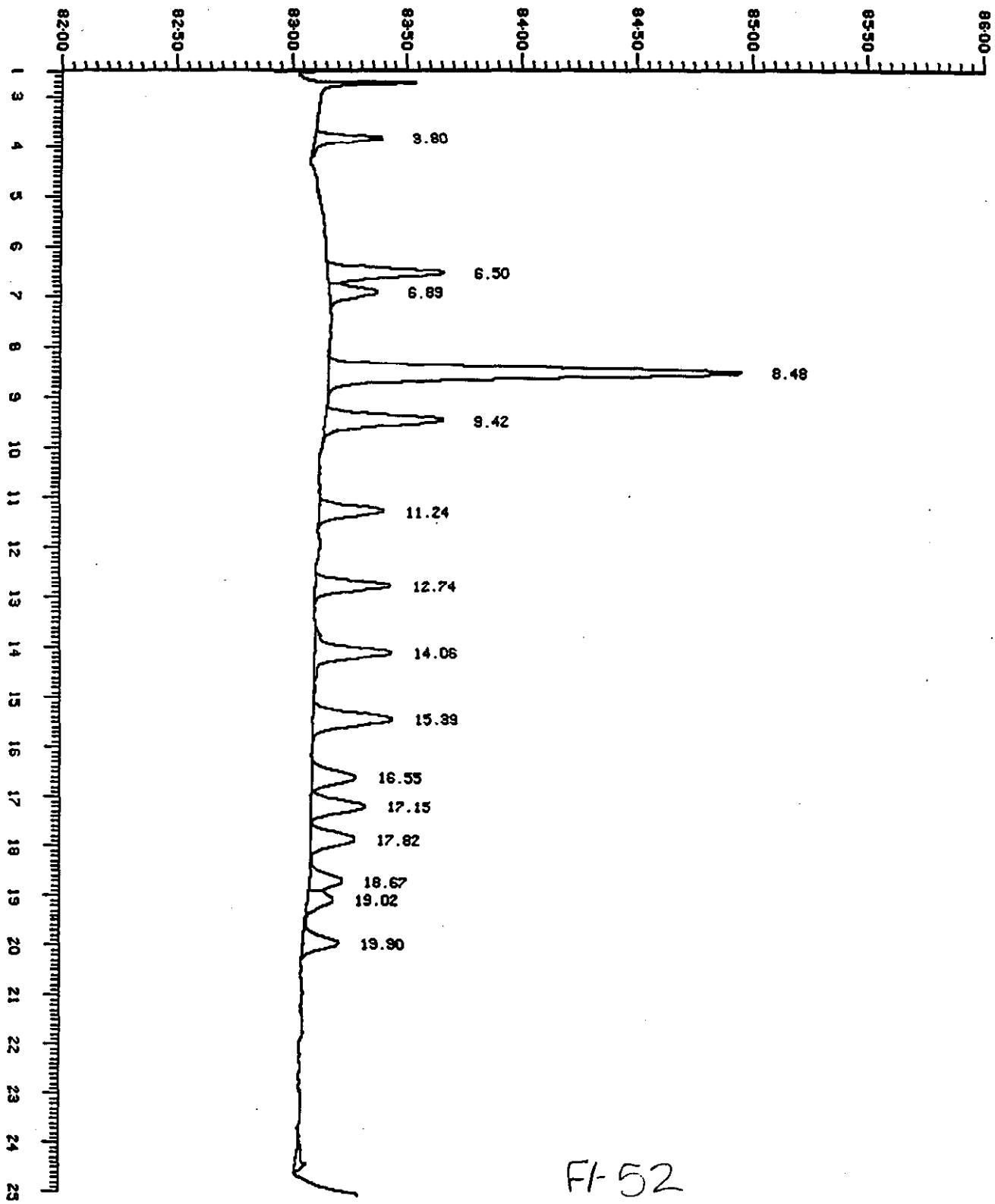
Uncalibrated peaks use user factor (0.0000)

F-51

Quanterra, Inc (West Sacramento)

[AUG_SV] 109 4H_04AUG981450,1,1

E070898F 25/100 8330MIX CV. Amount : 1.000.
Varian 5560/UV9050 @250nm flow=1.5 injection volume=200uL
Acquired on 4-AUG-1998 at 16:47
Reported on 4-AUG-1998 at 17:12
Box 1 (of 1)



F1-52

Quanterra / W. Sacramento
Chromatography Summary

Analysis ID: [AUG_SV] 109 4H_04AUG981450, 1
Acquired: HPLC #4 4-AUG-1998 at 16:47

Sample: E070898F 25/100 8330MIX CV **Mult:** 1.00 **Dilf:** 1.00 **Div:** 1.000
Method File: 8330_HY 75:25:0/2-22-50:00:50/2*- H2O:2%THF/MeOH:Methanol (Analysis)
Calibration: 03AUG98 8330H 2.5-1000ng/mL Phenomenex Hypersil C8 SN251486

Calibration Check

Channel 109 Var-LC5500 UV
Hypersil-C8
150x4.6mm 5um SN251486 8330M

	Peak Name	RT	True	Calc.	%D	-Limits-
1	HMX	3.804	25.00	24.1923	-3.2	(15)
2	1,3,5-Trinitrobenz	6.498	25.00	23.8052	-4.8	(15)
3	RDX	6.889	25.00	22.9470	-8.2	(15)
4	2,4-Dinitrofluorob	8.480	100.0	99.802	-0.20	(15)
5	1,3-Dinitrobenzene	9.422	25.00	23.8634	-4.5	(15)
6	Nitrobenzene	11.244	25.00	25.6327	2.5	(15)
7	2,4,6-Trinitrotolu	12.738	25.00	24.3666	-2.5	(15)
8	TETRYL	14.062	25.00	24.8070	-0.77	(15)
9	2,4-Dinitrotoluene	15.387	25.00	24.8606	-0.56	(15)
10	2,6-Dinitrotoluene	16.551	25.00	22.8102	-8.8	(15)
11	2-AM-4,6-Dinitroto	17.147	25.00	22.2946	-11	(15)
12	4-AM-2,6-Dinitroto	17.822	25.00	21.8519	-13	(15)
13	2-Nitrotoluene	18.667	25.00	26.3078	5.2	(15)
14	4-Nitrotoluene	19.022	25.00	25.6431	2.6	(15)
15	3-Nitrotoluene	19.902	25.00	27.5249	10	(15)

Notes:
No limits found 15% used

Average %D = 5.2

Q/WS SUM 3.29A

8-5-98

Reviewer: G Date: 8/5/98

Printed: 08/04/98 17:13:03

F1-53

Injection Report

Acquired on 5-AUG-1998 at 00:18

Quanterra, Inc (West Sacramento)

Analyst Name : LC4UV
Lims Id : 109LC4UV9050
Comment : 75:25:0/2-22-50:00:50/2"- H2O:2%THF/MeOH:Methanol (Analysis)
Method Title : Var-LC5500 UV Hypersil-C8 150x4.6mm 5um SN251486 8330M
Sample Name : E070898F 25/100 8330MIX CV
Sample Id :
Sample Type : Standard Amount=1.00000
Bottle No : 8

PEAK INFORMATION

Peak	RT mins	RT Exp	Hght uV	ppb	Peak name	Width
1	3.893	3.890	293	24.43827	HMX	7.5
2	6.551	6.550	503	23.45500	1,3,5-Trinitrobenzene	11.7
3	7.076	7.080	215	24.01365	RDX	13.3
4	8.596	8.600	1752	97.34324	2,4-Dinitrofluorobenzene(Surr)	13.3
5	9.591	9.590	521	24.15938	1,3-Dinitrobenzene	13.3
6	11.396	11.400	269	24.93740	Nitrobenzene	13.3
7	12.898	12.900	323	23.97477	2,4,6-Trinitrotoluene (TNT)	12.8
8	14.338	14.340	340	25.44824	TETRYL	13.3
9	15.680	15.680	342	24.39936	2,4-Dinitrotoluene	16.5
10	16.889	16.890	217	26.12652	2,6-Dinitrotoluene	18.1
11	17.618	17.620	277	26.40487	2-AM-4,6-Dinitrotoluene	17.6
12	18.276	18.280	212	24.36284	4-AM-2,6-Dinitrotoluene	17.6
13	18.987	18.990	151	28.17546	2-Nitrotoluene	17.1A
14	19.360	19.360	129	29.93246	4-Nitrotoluene	20.3A
15	20.187	20.190	166	29.48434	3-Nitrotoluene	16.0
16	22.098	0.000	13	0.00000		14.9
17	22.311	0.000	19	0.00000		12.8
18	24.373	0.000	69	0.00000		3.2
19	24.702	0.000	59	0.00000		18.1

see reintegration

JRS 5-5-97

Totals		
Unknowns	160	0.00000
Named	5710	456.65582
Grand Total	5870	456.65582

ANALYSIS SUMMARY

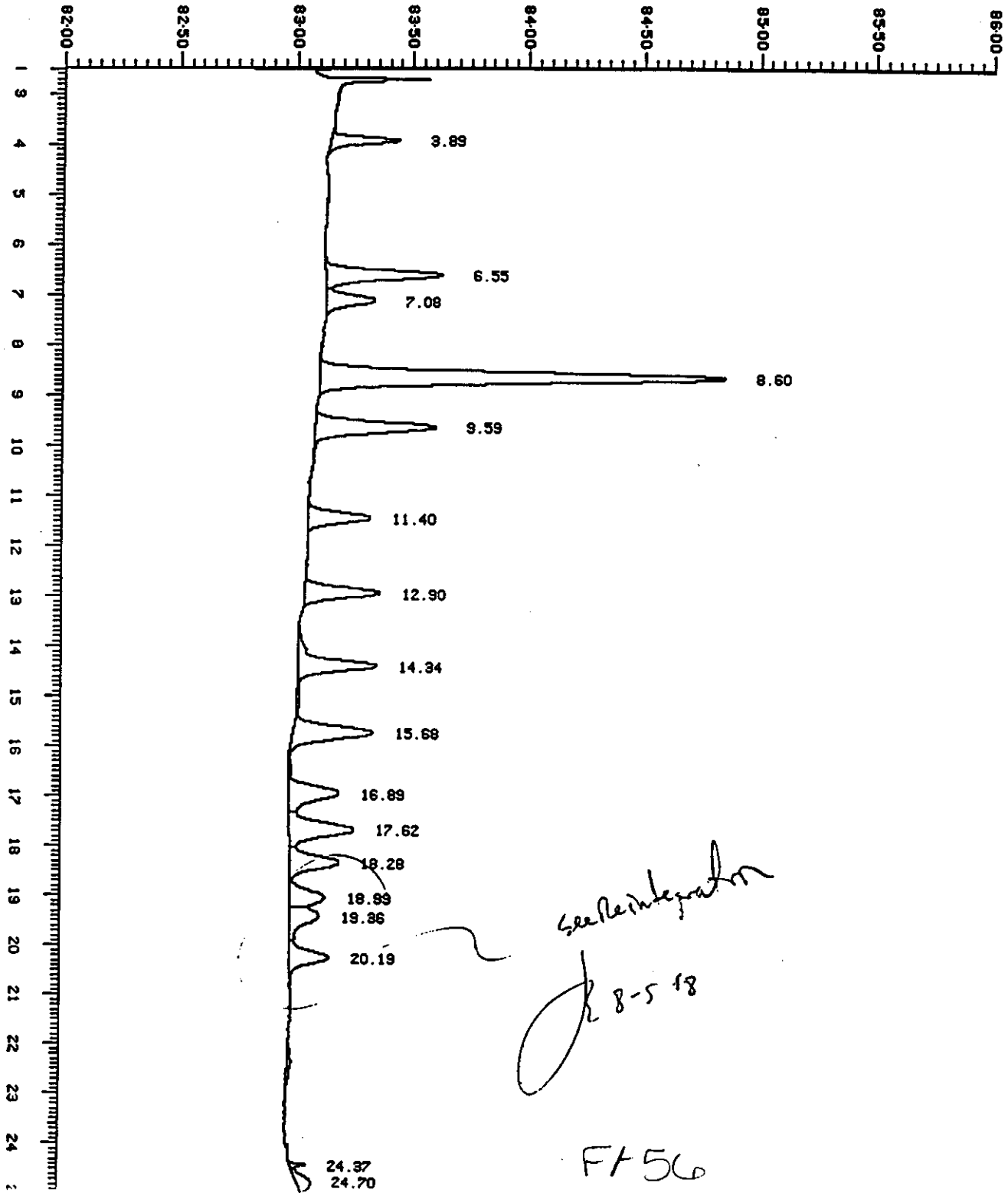
Method..... 8330_HY
Run sequence..... EXP4HY1
Calibration..... 03AUG98
External standard calibration using height
Calibration last modified on 5-AUG-1998 at 14:13
Calibration initialised on 12-DEC-1997 at 21:47
Method last modified on 4-AUG-1998 at 16:40

Uncalibrated peaks use user factor (0.0000)

Quanterra, Inc (West Sacramento)

[AUG_SV] 109 4H_04AUG981450,12,1

E070898F 25/100 8330MIX CV. Amount : 1.000.
Varian 5560/UV9050 @250nm flow=1.5 injection volume=200uL
Acquired on 5-AUG-1998 at 00:18
Reported on 5-AUG-1998 at 14:13
Box 1 (of 1)



see reintegration

8-5 98

F/56

Quanterra / W. Sacramento **Analysis ID: [AUG_SV]** 109 4H_04AUG981450,12
Chromatography Summary **Acquired: HPLC #4** 5-AUG-1998 at 00:18

Sample: E070898F 25/100 8330MIX CV **Mult:** 1.00 **Dilf:** 1.00 **Div:** 1.000
Method File: 8330_HY 75:25:0/2-22-50:00:50/2"- H2O:2%THF/MeOH:Methanol (Analysis)
Calibration: 03AUG98 8330H 2.5-1000ng/mL Phenomenex Hypersil C8 SN251486

Calibration Check

Channel 109 Var-LC5500 UV
Hypersil-C8
150x4.6mm 5um SN251486 8330M

	Peak Name	RT	True	Calc.	%D	-Limits-
1	HMX	3.893	25.00	24.4383	-2.2	(15)
2	1,3,5-Trinitrobenz	6.551	25.00	23.4550	-6.2	(15)
3	RDX	7.076	25.00	24.0136	-3.9	(15)
4	2,4-Dinitrofluorob	8.596	100.0	97.3432	-2.7	(15)
5	1,3-Dinitrobenzene	9.591	25.00	24.1594	-3.4	(15)
6	Nitrobenzene	11.396	25.00	24.9374	-0.25	(15)
7	2,4,6-Trinitrotolu	12.898	25.00	23.9748	-4.1	(15)
8	TETRYL	14.338	25.00	25.4482	1.8	(15)
9	2,4-Dinitrotoluene	15.680	25.00	24.3994	-2.4	(15)
10	2,6-Dinitrotoluene	16.889	25.00	26.1265	4.5	(15)
11	2-AM-4,6-Dinitroto	17.618	25.00	26.4049	5.6	(15)
12	4-AM-2,6-Dinitroto	18.276	25.00	24.3628	-2.5	(15)
13	2-Nitrotoluene	18.987	25.00	28.1755	13	(15)
14	4-Nitrotoluene	19.360	25.00	29.9325	20 *	(15)
15	3-Nitrotoluene	20.187	25.00	29.4843	18 *	(15)

See the integration
5-5-98

Notes:
No limits found 15% used

Average %D = 6.0

Q/WS SUM 3.29A 8-5-98

Reviewer: _____ Date: _____
Printed: 08/05/98 14:13:16

Ft 57

Injection Report

Acquired on 5-AUG-1998 at 00:18

Quanterra, Inc (West Sacramento)

Analyst Name : LC4UV
Lims Id : 109LC4UV9050
Comment : 75:25:0/2-22-50:00:50/2"- H2O:2%THF/MeOH:Methanol (Analysis)
Method Title : Var-LC5500 UV Hypersil-C8 150x4.6mm 5um SN251486 8330M
Sample Name : E070898F 25/100 8330MIX CV
Sample Id :
Sample Type : Standard Amount=1.00000
Bottle No : 8

PEAK INFORMATION

Peak	RT mins	RT Exp	Hght uV	ppb	Peak name	Width
1	3.893	3.890	293	24.43827	HMX	7.5
2	6.551	6.550	503	23.45500	1,3,5-Trinitrobenzene	11.7
3	7.076	7.080	215	24.01365	RDX	13.3
4	8.596	8.600	1752	97.34324	2,4-Dinitrofluorobenzene(Surr)	13.3
5	9.591	9.590	521	24.15938	1,3-Dinitrobenzene	13.3
6	11.396	11.400	269	24.93740	Nitrobenzene	13.3
7	12.898	12.900	323	23.97477	2,4,6-Trinitrotoluene (TNT)	12.8
8	14.338	14.340	340	25.44824	TETRYL	13.3
9	15.680	15.680	342	24.39936	2,4-Dinitrotoluene	16.5
10	16.889	16.890	214	25.76401	2,6-Dinitrotoluene	18.1
11	17.618	17.620	271	25.80247	2-AM-4,6-Dinitrotoluene	17.6
12	18.276	18.280	203	23.29481	4-AM-2,6-Dinitrotoluene	17.1
13	18.987	18.990	138	25.83562	2-Nitrotoluene	19.2
14	19.360	19.360	115	26.63412	4-Nitrotoluene	18.1A
15	20.196	20.190	155	27.40993	3-Nitrotoluene	15.5
16	22.098	0.000	13	0.00000		14.9
17	22.311	0.000	19	0.00000		12.8
18	24.373	0.000	69	0.00000		3.2
19	24.702	0.000	59	0.00000		18.1

Totals		
Unknowns	160	0.00000
Named	5653	446.91028
Grand Total	5813	446.91028

RE-INTEGRATION CODES

1 Poor Peak Shape
2 Poor Peak Resolution
3 Peak Not Integrated
4 Sample Matrix Interference
5 Column Bleed
6 Instrument Noise
7 Other: (reason must be stated)

ALL RE-INTEGRATIONS MUST BE INITIALED, DATED, AND CODED.

Baseline drift
8-5-98

F1-58

ANALYSIS SUMMARY

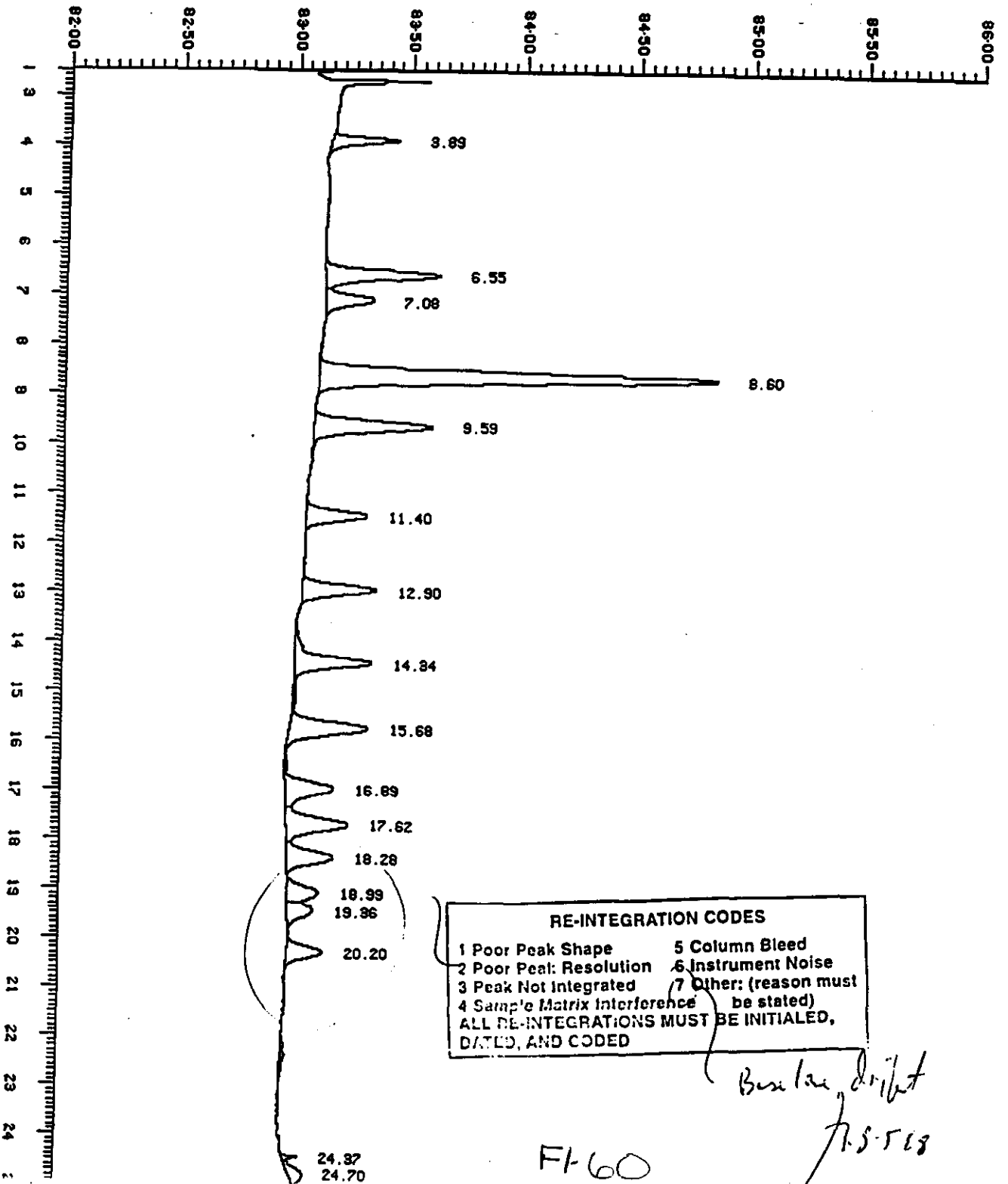
Method..... 8330_HY
Run sequence..... EXP4HY1
Calibration..... 03AUG98
External standard calibration using height
Calibration last modified on 5-AUG-1998 at 14:16
Calibration initialised on 12-DEC-1997 at 21:47
Method last modified on 5-AUG-1998 at 14:15

Uncalibrated peaks use user factor (0.0000)

Quanterra, Inc (West Sacramento)

[AUG_SV] 109 4H_04AUG981450,12,1

E070898F 25/100 8330MIX CV. Amount : 1.000.
Varian 5560/UV9050 @250nm flow=1.5 injection volume=200uL
Acquired on 5-AUG-1998 at 00:18
Reported on 5-AUG-1998 at 14:16
Box 1 (of 1)



RE-INTEGRATION CODES

1 Poor Peak Shape
2 Poor Peak Resolution
3 Peak Not Integrated
4 Sample Matrix Interference
5 Column Bleed
6 Instrument Noise
7 Other: (reason must be stated)

ALL RE-INTEGRATIONS MUST BE INITIALED, DATED, AND CODED

Baseline drift
7.5.98

F1-60

Quanterra / W. Sacramento **Analysis ID: [AUG_SV]** 109 4H_04AUG981450,12
Chromatography Summary **Acquired: HPLC #4** 5-AUG-1998 at 00:18

Sample: E070898F 25/100 8330MIX CV **Mult:** 1.00 **Dilf:** 1.00 **Div:** 1.000
Method File: 8330_HY 75:25:0/2-22-50:00:50/2"- H2O:2%THF/MeOH:Methanol (Analysis)
Calibration: 03AUG98 8330H 2.5-1000ng/mL Phenomenex Hypersil C8 SN251486

Calibration Check

Channel 109 Var-LC5500 UV
Hypersil-C8
150x4.6mm 5um SN251486 8330M

	Peak Name	RT	True	Calc.	%D	-Limits-
1	HMX	3.893	25.00	24.4383	-2.2	(15)
2	1,3,5-Trinitrobenz	6.551	25.00	23.4550	-6.2	(15)
3	RDX	7.076	25.00	24.0136	-3.9	(15)
4	2,4-Dinitrofluorob	8.596	100.0	97.3432	-2.7	(15)
5	1,3-Dinitrobenzene	9.591	25.00	24.1594	-3.4	(15)
6	Nitrobenzene	11.396	25.00	24.9374	-0.25	(15)
7	2,4,6-Trinitrotolu	12.898	25.00	23.9748	-4.1	(15)
8	TETRYL	14.338	25.00	25.4482	1.8	(15)
9	2,4-Dinitrotoluene	15.680	25.00	24.3994	-2.4	(15)
10	2,6-Dinitrotoluene	16.889	25.00	25.7640	3.1	(15)
11	2-AM-4,6-Dinitroto	17.618	25.00	25.8025	3.2	(15)
12	4-AM-2,6-Dinitroto	18.276	25.00	23.2948	-6.8	(15)
13	2-Nitrotoluene	18.987	25.00	25.8356	3.3	(15)
14	4-Nitrotoluene	19.360	25.00	26.6341	6.5	(15)
15	3-Nitrotoluene	20.196	25.00	27.4099	9.6	(15)

RE-INTEGRATION CODES
Peak Shape 5 Column B
Resolution 6 Ins
Integrated
Interfe.
RATIONS MUST BE INITI
CODED

Baseline drift
9-5-98

Notes:
No limits found 15% used

Average %D = 4.0

Q/WS SUM 3.29A

9-5-98

Reviewer: a **Date:** 8/5/98
Printed: 08/05/98 14:16:11

F-61

Method Blank (w/SCS)

F-62

METHOD BLANK REPORT
HPLC Analysis Area
Project: 300612

Test: 8330-IRPMS-1C-A Nitroaromatics and Nitramines by HPLC
Method: 8330
Matrix: AQUEOUS
QC Lot: 28 JUL 98-13D QC Run: 28 JUL 98-13D
Analyzed: 04 AUG 98 Time: 18:16

Analyte	Result	Units	Reporting Limit	Qualifier
RDX	ND	ug/L	5.0	
1,3-Dinitrobenzene	ND	ug/L	5.0	
Nitrobenzene	ND	ug/L	5.0	
2,4,6-Trinitrotoluene	ND	ug/L	5.0	
Tetryl	ND	ug/L	5.0	
2,4-Dinitrotoluene	ND	ug/L	5.0	
2,6-Dinitrotoluene	ND	ug/L	5.0	
2-Am-DNT	ND	ug/L	5.0	
4-Am-DNT	ND	ug/L	5.0	
2-Nitrotoluene	ND	ug/L	10	
4-Nitrotoluene	ND	ug/L	8.5	
3-Nitrotoluene	ND	ug/L	7.9	

Surrogate	% Recovery	Acceptable Range
2,4-Dinitrofluorobenzene	89	65 -135

ND = Not Detected

Injection Report

Acquired on 4-AUG-1998 at 18:16

Quanterra, Inc (West Sacramento)

Analyst Name : LC4UV
Lims Id : 109LC4UV9050
Comment : 75:25:0/2-22-50:00:50/2"- H2O:2%THF/MeOH:Methanol (Analysis)
Method Title : Var-LC5500 UV Hypersil-C8 150x4.6mm 5um SN251486 8330M
Sample Name : MB 28JUL98-13D 770ML/20ML
Sample Id :
Sample Type : Sample Amount=1.00000
Bottle No : 8

PEAK INFORMATION

Peak	RT mins	RT Exp	Hght uV	ppb	Peak name	Width
1	8.507	8.476	1595	2.30113	2,4-Dinitrofluorobenzene(Surr)	12.8
2	13.876	14.078	34	0.06702	TETRYL	12.3
3	21.618	0.000	27	0.00000		38.4
4	22.649	0.000	14	0.00000		12.3
5	24.311	0.000	59	0.00000		23.5A
6	24.400	0.000	109	0.00000		8.5A
7	24.658	0.000	32	0.00000		4.8

Totals		
Unknowns	241	0.00000
Named	1629	2.36814
Grand Total	1870	2.36814

ANALYSIS SUMMARY

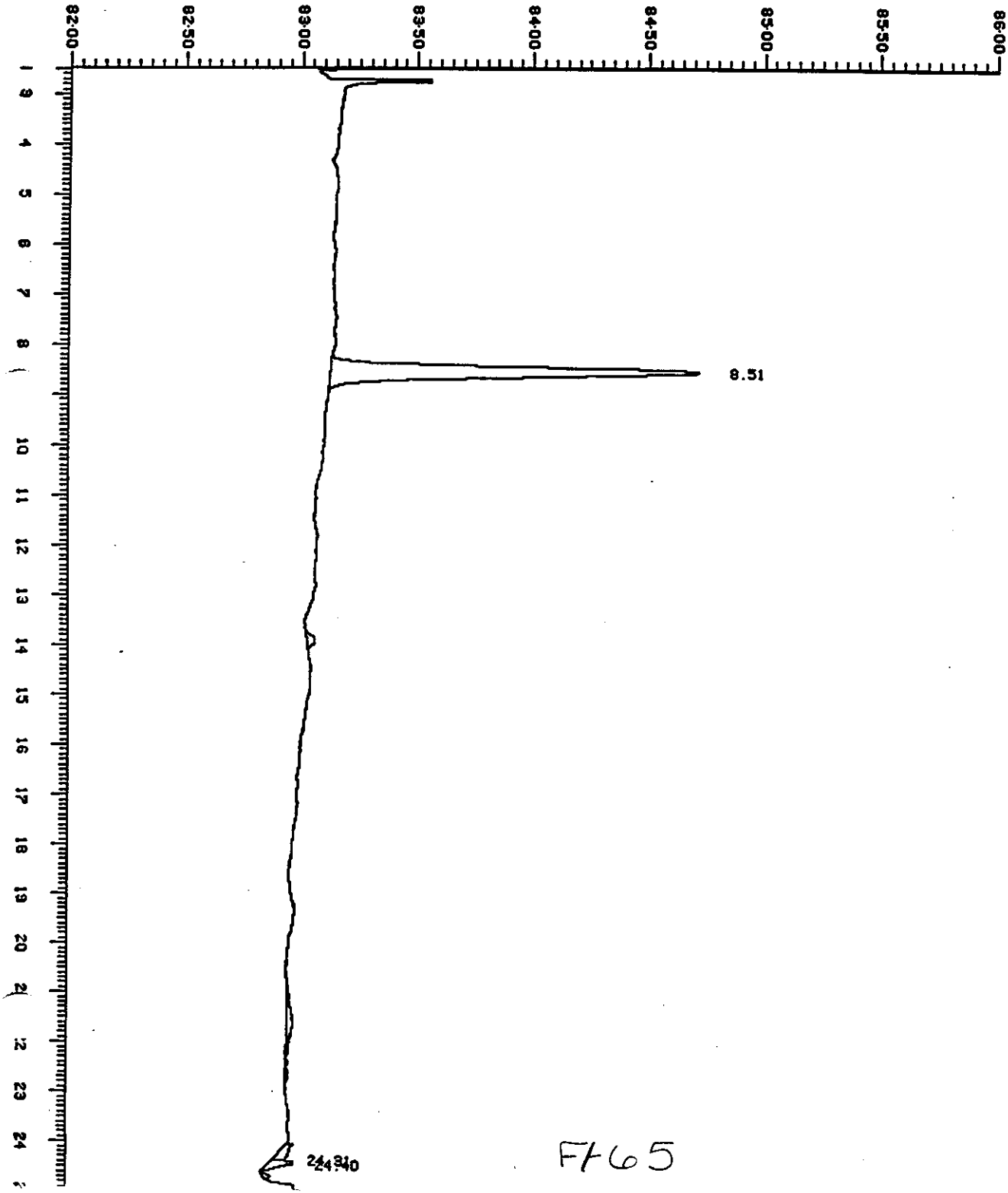
Method..... 8330 HY
Run sequence..... EXP4HY1
Calibration..... 03AUG98
External standard calibration using height
Calibration last modified on 4-AUG-1998 at 17:13
Calibration initialised on 12-DEC-1997 at 21:47
Method last modified on 4-AUG-1998 at 16:40

Uncalibrated peaks use user factor (0.0000)

Quanterra, Inc (West Sacramento)

[AUG_SV] 109 4H_04AUG981450,2,1

MB 28JUL98-13D 770ML/20ML. Amount : 1.000.
Varian 5560/UV9050 @250nm flow=1.5 injection volume=200uL
Acquired on 4-AUG-1998 at 18:16
Reported on 4-AUG-1998 at 18:41
Box 1 (of 1)



F465

Quanterra / W. Sacramento
Chromatography Summary

Analysis ID: [AUG_SV] 109 4H_04AUG981450, 2
Acquired: HPLC #4 4-AUG-1998 at 18:16

Sample: MB 28JUL98-13D 770ML/20ML **Mult:** 20.00 **Dilf:** 1.00 **Div:** 770.0
Method File: 8330_HY 75:25:0/2-22-50:00:50/2"- H2O:2%THF/MeOH:Methanol (Analysis)
Calibration: 03AUG98 8330H 2.5-1000ng/mL Phenomenex Hypersil C8 SN251486

Target Analyte Results

Channel 109 Var-LC5500 UV
Hypersil-C8
150x4.6mm 5um SN251486 8330M

	Peak Name	RT	Diff	Height	ppb	R	P	RL
1	HMX							1.00
2	1,3,5-Trinitrobenz							0.300
3	RDX							0.800
4	2,4-Dinitrofluorob	8.507	+0.03	1595	2.3011		F	
5	1,3-Dinitrobenzene							0.100
6	Nitrobenzene							1.00
7	2,4,6-Trinitrotolu							0.130
8	TETRYL	13.876	-0.20	34	0.0670	U	F	1.00
9	2,4-Dinitrotoluene							0.100
10	2,6-Dinitrotoluene							0.300
11	2-AM-4,6-Dinitroto							0.130
12	4-AM-2,6-Dinitroto							0.130
13	2-Nitrotoluene							1.00
14	4-Nitrotoluene							1.00
15	3-Nitrotoluene							1.00

Surrogate Results

	Peak Name	Amount Spiked	Amount Found	%Rec	-Limits-
4	2,4-Dinitrofluorob	2.60	2.3011	89	(65 - 135)

Notes:
8330
Quantitation limits exceed RL

Range Flags (R)
U - Under calibration Range
O - Over calibration Range
X - Not calibrated

Peak Type Flags (P)
* - Flat top Peak
F - Forced baseline
M - Modified baseline

Q/WS SUM 3.29A

5.5.98

Reviewer: a Date: 8/5/98
Printed: 08/04/98 18:41:36

FF66

Laboratory/Duplicate Control Sample

DUPLICATE CONTROL SAMPLE REPORT
HPLC Analysis Area
Project: 300612

Category: 8330-COE-A Explosives by HPLC
Testcode: 8330-IRPMS-1C-A Method: 8330
Matrix: AQUEOUS Concentration Units: ug/L
QC Lot: 28 JUL 98-13D Analyzed Date: 04 AUG 98 Time: 19:28

Analyte	Concentration Spiked	Measured DCS1	Measured DCS2	Accuracy (%) DCS1	Accuracy (%) DCS2	Limits	Precision (RPD) DCS	Limit
HMX	0.500	0.468	0.473	94	95	65-135	1.2	35
1,3,5-Trinitrobenzene	0.500	0.468	0.470	94	94	65-135	0.51	35
RDX	0.500	0.432	0.453	86	91	65-135	4.6	35
1,3-Dinitrobenzene	0.500	0.425	0.461	85	92	65-135	8.1	35
Nitrobenzene	0.500	0.369	0.458	74	92	65-135	22	35
2,4,6-Trinitrotoluene	0.500	0.511	0.495	102	99	65-135	3.2	35
Tetryl	0.500	0.510	0.512	102	102	65-135	0.47	35
2,4-Dinitrotoluene	0.500	0.486	0.485	97	97	65-135	0.12	35
2,6-Dinitrotoluene	0.500	0.432	0.487	86	97	65-135	12	35
2-Am-DNT	0.500	0.453	0.478	91	96	65-135	5.2	35
4-Am-DNT	0.500	0.482	0.456	96	91	65-135	5.5	35
2-Nitrotoluene	0.500	0.414	0.504	83	101	65-135	19	35
4-Nitrotoluene	0.500	0.413	0.552	83	110	65-135	29	35
3-Nitrotoluene	0.500	0.430	0.523	86	105	65-135	20	35

Surrogate	Concentration Spiked	Measured DCS1	Measured DCS2	Accuracy(%) DCS1	Accuracy(%) DCS2	Limits
2,4-Dinitrofluorobenzene	2.6	2.4	2.4	90	92	65-135

Calculations are performed before rounding to avoid round-off errors in calculated resul

Injection Report

Acquired on 4-AUG-1998 at 18:52

Quanterra, Inc (West Sacramento)

Analyst Name : LC4UV
Lims Id : 109LC4UV9050
Comment : 75:25:0/2-22-50:00:50/2"- H2O:2%THF/MeOH:Methanol (Analysis)
Method Title : Var-LC5500 UV Hypersil-C8 150x4.6mm 5um SN251486 8330M
Sample Name : DCS1 28JUL98-13D 770ML/20ML
Sample Id :
Sample Type : Sample Amount=1.00000
Bottle No : 8

PEAK INFORMATION

Peak	RT mins	RT Exp	Hght uV	ppb	Peak name	Width
1	3.804	3.769	216	0.46767	HMX	8.0
2	6.480	6.493	386	0.46791	1,3,5-Trinitrobenzene	11.2
3	6.880	6.907	149	0.43213	RDX	12.8
4	8.462	8.476	1629	2.35050	2,4-Dinitrofluorobenzene(Surr)	12.3
5	9.422	9.444	352	0.42461	1,3-Dinitrobenzene	13.3
6	10.276	0.000	12	0.00000		14.9
7	11.253	11.271	153	0.36882	Nitrobenzene	12.3
8	12.711	12.729	265	0.51105	2,4,6-Trinitrotoluene (TNT)	12.8
9	14.036	14.078	262	0.50981	TETRYL	12.3
10	15.369	15.413	262	0.48551	2,4-Dinitrotoluene	16.5
11	16.516	16.576	139	0.43250	2,6-Dinitrotoluene	17.1
12	17.129	17.178	183	0.45330	2-AM-4,6-Dinitrotoluene	16.5
13	17.778	17.836	161	0.48171	4-AM-2,6-Dinitrotoluene	17.1
14	18.622	18.687	85	0.41414	2-Nitrotoluene	18.7
15	19.022	19.080	69	0.41340	4-Nitrotoluene	19.7
16	19.902	19.942	93	0.43015	3-Nitrotoluene	15.5
17	24.382	0.000	59	0.00000		3.2
18	24.773	0.000	98	0.00000		12.3

Totals		
Unknowns	169	0.00000
Named	4405	8.64320
Grand Total	4574	8.64320

F469

ANALYSIS SUMMARY

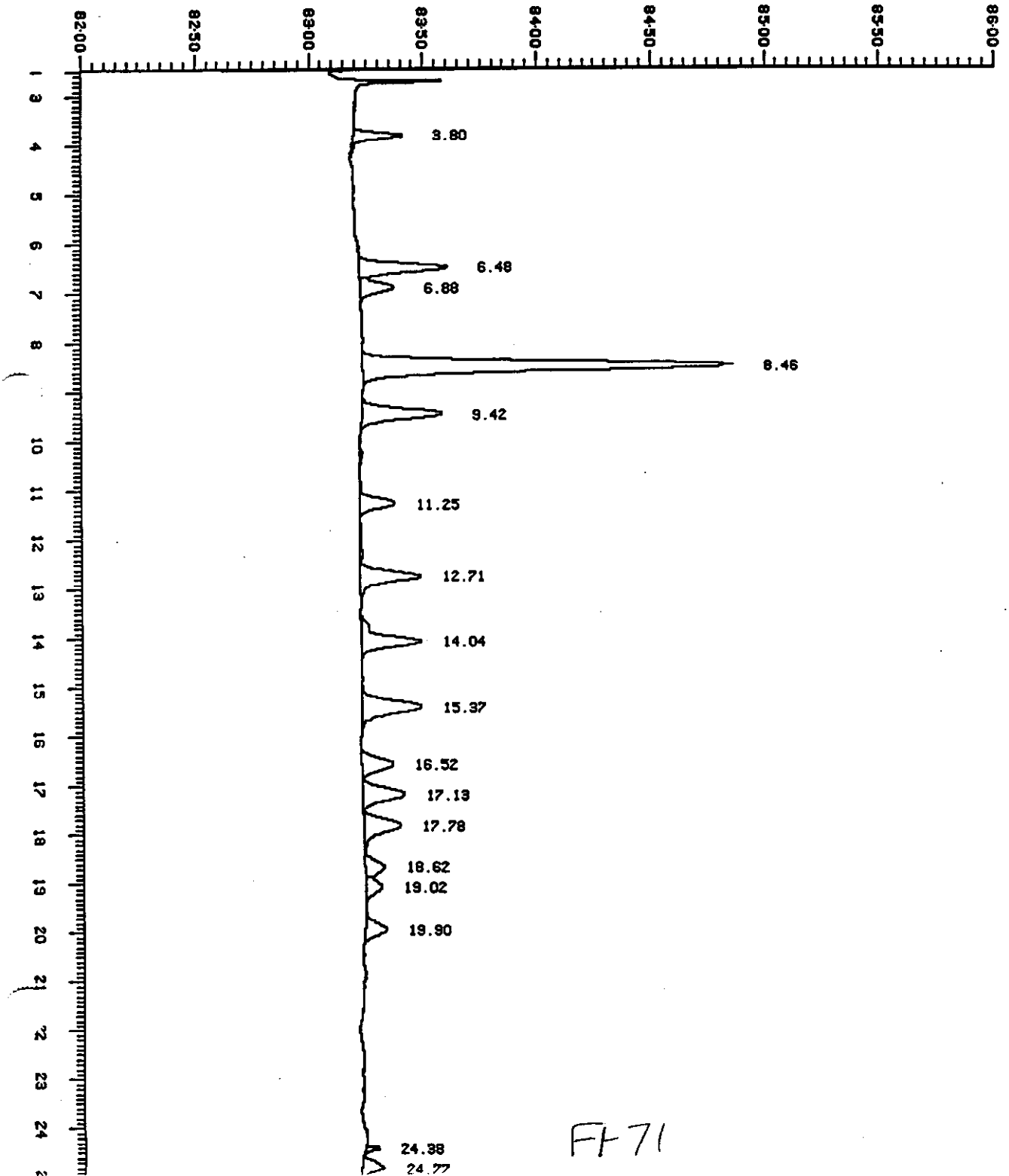
Method..... 8330 HY
Run sequence..... EXP4HY1
Calibration..... 03AUG98
External standard calibration using height
Calibration last modified on 4-AUG-1998 at 18:41
Calibration initialised on 12-DEC-1997 at 21:47
Method last modified on 4-AUG-1998 at 16:40

Uncalibrated peaks use user factor (0.0000)

Quanterra, Inc (West Sacramento)

[AUG_SV] 109 4H_04AUG981450,3,1

DCS1 28JUL98-13D 770ML/20ML. Amount : 1.000.
Varian 5560/UV9050 @250nm flow=1.5 injection volume=200uL
Acquired on 4-AUG-1998 at 18:52
Reported on 4-AUG-1998 at 19:17
Box 1 (of 1)



FF71

Quanterra / W. Sacramento **Analysis ID:** [AUG_SV] 109 4H_04AUG981450, 3
Chromatography Summary **Acquired:** HPLC #4 4-AUG-1998 at 18:52

Sample: DCS1 28JUL98-13D 770ML/20ML **Mult:** 20.00 **Dilf:** 1.00 **Div:** 770.0
Method File: 8330_HY 75:25:0/2-22-50:00:50/2"- H2O:2%THF/MeOH:Methanol (Analysis)
Calibration: 03AUG98 8330H 2.5-1000ng/mL Phenomenex Hypersil C8 SN251486

Channel 109 Var-LC5500 UV
Laboratory Control Sample Hypersil-C8
150x4.6mm 5um SN251486 8330M

	Peak Name	RT	Spiked	Found	Rec	-Limits-
1	HMX	3.804	0.500	0.4677	94	(65 - 135)
2	1,3,5-Trinitrobenz	6.480	0.500	0.4679	94	(65 - 135)
3	RDX	6.880	0.500	0.4321	86	(65 - 135)
4	2,4-Dinitrofluorob	8.462	2.60	2.3505	90	(65 - 135)
5	1,3-Dinitrobenzene	9.422	0.500	0.4246	85	(65 - 135)
6	Nitrobenzene	11.253	0.500	0.3688	74	(65 - 135)
7	2,4,6-Trinitrotolu	12.711	0.500	0.5111	102	(65 - 135)
8	TETRYL	14.036	0.500	0.5098	102	(65 - 135)
9	2,4-Dinitrotoluene	15.369	0.500	0.4855	97	(65 - 135)
10	2,6-Dinitrotoluene	16.516	0.500	0.4325	86	(65 - 135)
11	2-AM-4,6-Dinitroto	17.129	0.500	0.4533	91	(65 - 135)
12	4-AM-2,6-Dinitroto	17.778	0.500	0.4817	96	(65 - 135)
13	2-Nitrotoluene	18.622	0.500	0.4141	83	(65 - 135)
14	4-Nitrotoluene	19.022	0.500	0.4134	83	(65 - 135)
15	3-Nitrotoluene	19.902	0.500	0.4302	86	(65 - 135)

Surrogate Results

	Peak Name	Amount Spiked	Amount Found	%Rec	-Limits-
4	2,4-Dinitrofluorob	2.60	2.3505	90	(65 - 135)

Notes:
8330

Reviewer: a Date: 8/8/98

Q/WS SUM 3.29A **Printed:** 08/05/98 17:04:06

F172

Injection Report

Acquired on 4-AUG-1998 at 19:28

Quanterra, Inc (West Sacramento)

Analyst Name : LC4UV
Lims Id : 109LC4UV9050
Comment : 75:25:0/2-22-50:00:50/2"- H2O:2%THF/MeOH:Methanol (Analysis)
Method Title : Var-LC5500 UV Hypersil-C8 150x4.6mm 5um SN251486 8330M
Sample Name : DCS2 28JUL98-13D 770ML/20ML
Sample Id :
Sample Type : Sample Amount=1.00000
Bottle No : 8

PEAK INFORMATION

Peak	RT mins	RT Exp	Hght uV	ppb	Peak name	Width
1	3.813	3.769	219	0.47340	HMX	8.0
2	6.489	6.493	388	0.47031	1,3,5-Trinitrobenzene	11.2
3	6.889	6.907	156	0.45265	RDX	12.8
4	8.471	8.476	1663	2.39968	2,4-Dinitrofluorobenzene(Surr)	12.3
5	9.431	9.444	382	0.46064	1,3-Dinitrobenzene	12.8
6	10.276	0.000	13	0.00000		9.6
7	11.262	11.271	190	0.45786	Nitrobenzene	12.3
8	12.720	12.729	256	0.49485	2,4,6-Trinitrotoluene (TNT)	12.8
9	14.062	14.078	263	0.51216	TETRYL	13.9
10	15.396	15.413	262	0.48493	2,4-Dinitrotoluene	16.0
11	16.569	16.576	156	0.48725	2,6-Dinitrotoluene	17.1
12	17.173	17.178	193	0.47763	2-AM-4,6-Dinitrotoluene	17.1
13	17.849	17.836	153	0.45595	4-AM-2,6-Dinitrotoluene	17.1
14	18.676	18.687	104	0.50345	2-Nitrotoluene	13.9A
15	19.084	19.080	92	0.55199	4-Nitrotoluene	21.3A
16	19.956	19.942	114	0.52330	3-Nitrotoluene	15.5
17	24.382	0.000	64	0.00000		3.2
18	24.773	0.000	84	0.00000		11.2

Totals		
Unknowns	161	0.00000
Named	4590	9.20605
Grand Total	4751	9.20605

ANALYSIS SUMMARY

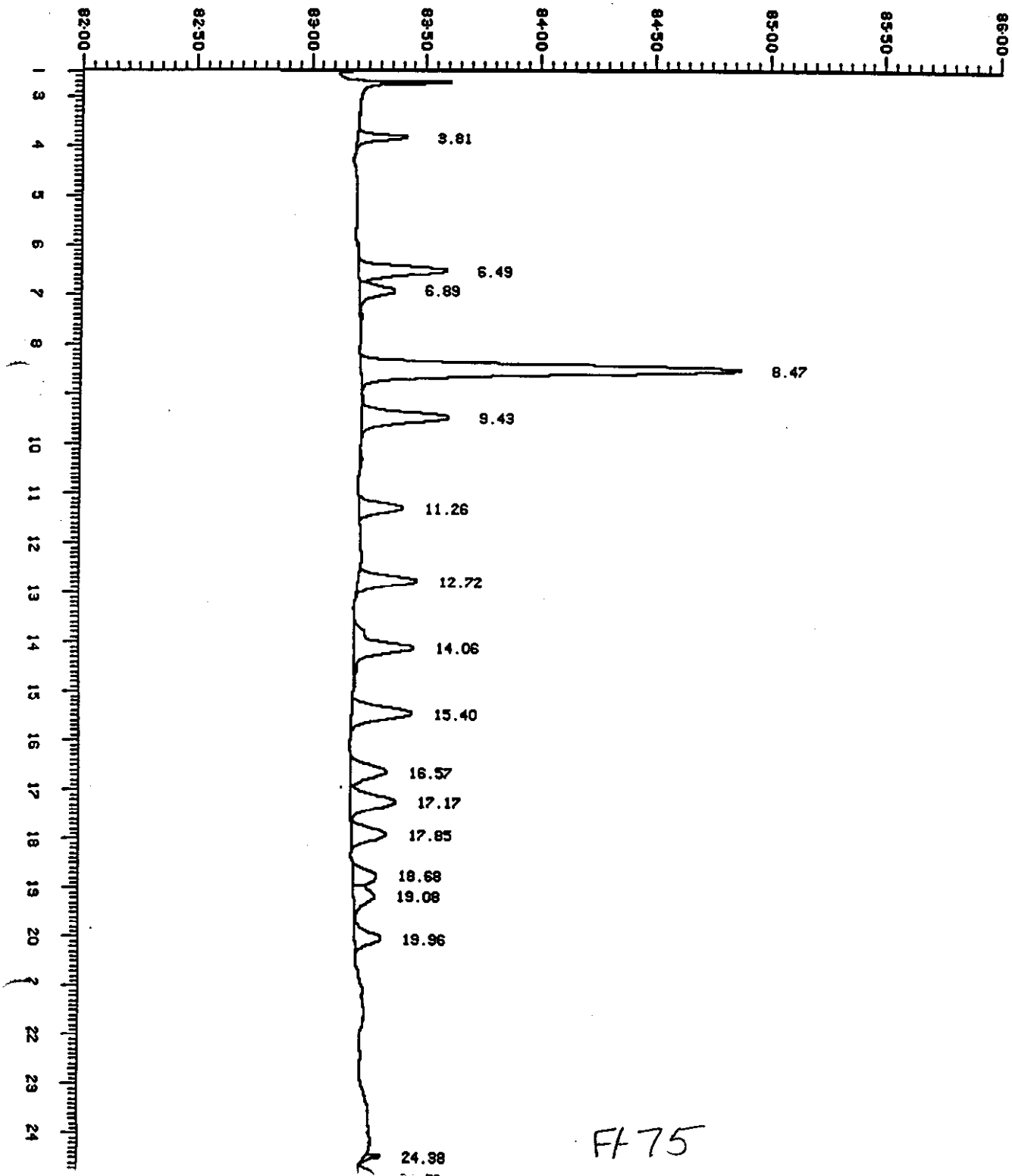
Method..... 8330_HY
Run sequence..... EXP4HY1
Calibration..... 03AUG98
External standard calibration using height
Calibration last modified on 4-AUG-1998 at 19:17
Calibration initialised on 12-DEC-1997 at 21:47
Method last modified on 4-AUG-1998 at 16:40

Uncalibrated peaks use user factor (0.0000)

Quanterra, Inc (West Sacramento)

[AUG_SV] 109 4H_04AUG981450,4,1

DCS2 28JUL98-13D 770ML/20ML. Amount : 1.000.
Varian 5560/UV9050 @250nm flow=1.5 injection volume=200uL
Acquired on 4-AUG-1998 at 19:28
Reported on 4-AUG-1998 at 19:54
Box 1 (of 1)



F475

Quanterra / W. Sacramento **Analysis ID:** [AUG_SV] 109 4H_04AUG981450, 4
Chromatography Summary **Acquired:** HPLC #4 4-AUG-1998 at 19:28

Sample: DCS2 28JUL98-13D 770ML/20ML **Mult:** 20.00 **Dilf:** 1.00 **Div:** 770.0
Method File: 8330_HY 75:25:0/2-22-50:00:50/2"- H2O:2%THF/MeOH:Methanol (Analysis)
Calibration: 03AUG98 8330H 2.5-1000ng/mL Phenomenex Hypersil C8 SN251486

Channel 109 Var-LC5500 UV
Laboratory Control Sample Hypersil-C8
150x4.6mm 5um SN251486 8330M

	Peak Name	RT	Spiked	Found	Rec	-Limits-
1	HMX	3.813	0.500	0.4734	95	(65 - 135)
2	1,3,5-Trinitrobenz	6.489	0.500	0.4703	94	(65 - 135)
3	RDX	6.889	0.500	0.4526	91	(65 - 135)
4	2,4-Dinitrofluorob	8.471	2.60	2.3997	92	(65 - 135)
5	1,3-Dinitrobenzene	9.431	0.500	0.4606	92	(65 - 135)
6	Nitrobenzene	11.262	0.500	0.4579	92	(65 - 135)
7	2,4,6-Trinitrotolu	12.720	0.500	0.4949	99	(65 - 135)
8	TETRYL	14.062	0.500	0.5122	102	(65 - 135)
9	2,4-Dinitrotoluene	15.396	0.500	0.4849	97	(65 - 135)
10	2,6-Dinitrotoluene	16.569	0.500	0.4872	97	(65 - 135)
11	2-AM-4,6-Dinitroto	17.173	0.500	0.4776	96	(65 - 135)
12	4-AM-2,6-Dinitroto	17.849	0.500	0.4560	91	(65 - 135)
13	2-Nitrotoluene	18.676	0.500	0.5035	101	(65 - 135)
14	4-Nitrotoluene	19.084	0.500	0.5520	110	(65 - 135)
15	3-Nitrotoluene	19.956	0.500	0.5233	105	(65 - 135)

Surrogate Results

	Peak Name	Amount Spiked	Amount Found	%Rec	-Limits-
4	2,4-Dinitrofluorob	2.60	2.3997	92	(65 - 135)

Notes:
8330

Reviewer: a Date: 8/8/98

Q/WS SUM 3.29A **Printed:** 08/05/98 17:05:40

FF 76

Analysis Log Copies

Quanterra / W. Sacramento
CHROMATOGRAPHY RUN LOG

Analysis: HPLC #4 [AUG_SV] 4PC04AUG980849

Channel Column Detector
109 / Hypersil-C8 / UV

Method File: 8330_HY
Calib File: 03AUG98
8330H 2.5-1000ng/mL Phenomenex Hypersil C8 SN251486

Analysis Info: 75:25:0/2-22-50:00:50/2" H2O:2%THF/MeOH:Methanol (Curve)

#	SAMPLE NAME	MULT DIVS	DATE	TIME	LVL
1	PRIMER	1.00 / 1.000	4-AUG-1998	09:05	
2	PRIMER	1.00 / 1.000	4-AUG-1998	09:42	
3	Water blank	1.00 / 1.000	4-AUG-1998	10:18	
4	Water blank	1.00 / 1.000	4-AUG-1998	10:54	
5	E070898F 25/100ng/mL STD=I	1.00 / 1.000	4-AUG-1998	11:30	3
6	E070898C 2.5/5.0ng/ml STD=I	1.00 / 1.000	4-AUG-1998	12:06	T1
7	E070898D 5.0/25ng/mL STD=I	1.00 / 1.000	4-AUG-1998	12:43	1
8	E070898E 10/50ng/ml STD=I	1.00 / 1.000	4-AUG-1998	13:19	2
9	E070898G 100/150 STD=I	1.00 / 1.000	4-AUG-1998	13:55	5
10	E070898H 500 ng/mL 8330 Mix STD=I	1.00 / 1.000	4-AUG-1998	14:31	6
11	E070898I 1000 ng/mL 8330 Mix STD=I	1.00 / 1.000	4-AUG-1998	15:08	7
12	Water blank	1.00 / 1.000	4-AUG-1998	15:44	

Analyst: _____ Date: 5-4-98 Page: 148

F1-78

Quanterra / W. Sacramento
CHROMATOGRAPHY RUN LOG

Analysis: HPLC #4 [AUG_SV] 4H_04AUG981450

Channel Column Detector
109 / Hypersil-C8 / UV

Method File: 8330_HY
Calib File: 03AUG98
8330H 2.5-1000ng/mL Phenomenex Hypersil C8 SN251486

Analysis Info: 75:25:0/2-22-50:00:50/2"- H2O:2%THF/MeOH:Methanol (Analysis)

#	SAMPLE NAME	MULT / DIVS	DATE	TIME	LVL
1	E070898F 25/100 8330MIX CV	1.00 / 1.000	4-AUG-1998	16:47	3
2	MB 28JUL98-13D 770ML/20ML	20.0 / 770.0	4-AUG-1998	18:16	LA
3	DCS1 28JUL98-13D 770ML/20ML	20.0 / 770.0	4-AUG-1998	18:52	LA
4	DCS2 28JUL98-13D 770ML/20ML	20.0 / 770.0	4-AUG-1998	19:28	LA
5	300592-2 770ML/20ML	20.0 / 770.0	4-AUG-1998	20:04	LA
6	300602-1 770ML/20ML	20.0 / 770.0	4-AUG-1998	20:41	LA
7	300602-2 770ML/20ML	20.0 / 770.0	4-AUG-1998	21:17	LA
8	300602-2-MS 770ML/20ML	20.0 / 770.0	4-AUG-1998	21:53	LA
9	300602-2-SD 770ML/20ML	20.0 / 770.0	4-AUG-1998	22:29	LA
10	300612-1 770ML/20ML	20.0 / 770.0	4-AUG-1998	23:06	LA
11	300551-11 770ML/20ML	20.0 / 770.0	4-AUG-1998	23:42	LA
12	E070898F 25/100 8330MIX CV	1.00 / 1.000	5-AUG-1998	00:18	3
13	MB 29JUL98-11D 770ML/20ML	20.0 / 770.0	5-AUG-1998	00:54	LA
14	DCS1 29JUL98-11D 770ML/20ML	20.0 / 770.0	5-AUG-1998	01:31	LA
15	DCS2 29JUL98-11D 770ML/20ML	20.0 / 770.0	5-AUG-1998	02:07	LA
16	300575-1 770ML/20ML	20.0 / 770.0	5-AUG-1998	02:43	LA
17	300575-2 770ML/20ML	20.0 / 770.0	5-AUG-1998	03:19	LA
18	MB 28JUL98-11D 0.4G/8ML	8.00 / 0.400	5-AUG-1998	03:55	LS
19	DCS1 28JUL98-11D 0.4G/8ML	8.00 / 0.400	5-AUG-1998	04:32	LS
20	DCS2 28JUL98-11D 0.4G/8ML	8.00 / 0.400	5-AUG-1998	05:08	LS
21	132219-1 0.4G/8ML	8.00 / 0.400	5-AUG-1998	05:44	LS
22	132219-2 0.4G/8ML	8.00 / 0.400	5-AUG-1998	06:20	LS
23	E070898F 25/100 8330MIX CV	1.00 / 1.000	5-AUG-1998	06:57	3
24	132219-3 0.4G/8ML	8.00 / 0.400	5-AUG-1998	07:33	LS
25	132219-4 0.4G/8ML	8.00 / 0.400	5-AUG-1998	08:09	LS
26	132219-5 0.4G/8ML	8.00 / 0.400	5-AUG-1998	08:45	LS
27	132219-6 0.4G/8ML	8.00 / 0.400	5-AUG-1998	09:21	LS
28	132219-7 0.4G/8ML	8.00 / 0.400	5-AUG-1998	09:58	LS
29	132219-7-MS 0.4G/8ML	8.00 / 0.400	5-AUG-1998	10:34	LS
30	132219-7-SD 0.4G/8ML	8.00 / 0.400	5-AUG-1998	11:10	LS
31	MB 28JUL98-12D 0.4G/8ML	8.00 / 0.400	5-AUG-1998	11:46	LS
32	LCS 28JUL98-12D 0.4G/8ML	8.00 / 0.400	5-AUG-1998	12:23	LS
33	300551-1 0.4G/8ML	8.00 / 0.400	5-AUG-1998	12:59	LS
34	E070898F 25/100 8330MIX CV	1.00 / 1.000	5-AUG-1998	13:35	3
35	300551-1-MS 0.4G/8ML	8.00 / 0.400	5-AUG-1998	14:11	LS

***** Printed before sequence complete *****

Analyst: [signature] Date: 5-5-98 Page: 149
F1-79 Printed: 05-AUG-1998 15:27:33

Sample Extraction /Preparation Log Copies

QUANTERRA INCORPORATED

West Sacramento

DUE DATE: 7-28

PROJECT ANOMALY RECORD AND DATA CHECKLIST

PROJECT # 300612 *TEST DESC.* 8330 - IRPMS - KC-A

DATA CHECKLIST

CALIBRATION
__ICAL or ICAL Summary & ICV/CCV included.
__ICAL, ICV/CCV criteria met.
__Standards labeled or correctly identified by data system
__Copy of logbook for ICAL included.
__Tune criteria met and copy included (GCMS only).
__Method number is identified on data.

QAQC VERIFICATION
__Method blank and DCS/LCS frequencies were met.
__DCS/LCS and MBcopies are included if applicable.
__DCS/LCS and MBdata are within control limits.
__If the batch QC data did not meet criteria, appropriate comments were made.
__MS/MSD data complete if applicable.
__Holding times were met.
__All samples within tune time (GCMS only).

SAMPLE ANALYSIS
__Logbook copies for all injections made, including ICV/CCV standards and ICAL.
__Logbooks/Prep bench sheets are properly filled out.
__Manual integrations are reviewed.
__All raw data is included.
__All analytes are reported correctly.
__Correct reporting limits were used.
__Spectra are present for all positive analytes (GCMS only).

LIMS
__Results were entered into LIMS correctly.
__The prepared and analytical dates are correct.
__The correct MB/DCS/LCS data were entered.
__The correct footnotes were used.
__The data sheets are complete and included
__Method blanks are included with correct prep and analyzed dates
__Anomalies are written and entered.

SIGNATURES BELOW INDICATE THE ABOVE CRITERIA HAVE BEEN MET.

CHEMIST [signature] DATE 8 5 98
REVIEWER Emily Lebelhoer DATE 8/5/98

SEE ELECTRONIC ANOMALY: X

NO ANOMALIES: _____

COMMENTS:

F1-81

QUANTERRA-CALLAB LABORATORY
Chromatography Assignment Sheet

Project #: 300612
Analysis: P-8330-COE-A Ver: 1
Client: [redacted]
PA: DBROOKS
Delivery Accepted: MGARCIA 25 JUL 98
Number of Samples: 1
Program: Explosives

Due Date: 06 AUG 98
Receipt Date: 25 JUL 98
Log Released: 27 JUL 98
Turnaround: RUSH
Storage Location: WR1G
Earliest Holding Time: 31 JUL 98

Project Description:
Aqueous for 8330-Total.

5-day TAT for 8330 level 2 and 10-day TAT for level 3.

Low PQLs for the aqueous sample - J values
IRPMS entry needed

Full raw data package - blue sheet order

Test Modified: N
Sample Instructions: N
Test Instructions: N
Group Instructions: N
QAS #: _____

Date Delivered to PA: _____

Comments:

__ No Problems Occurred with Analysis

__ See Anomaly Sheet

QUANTERRA-CALLAB LABORATORY
Bottle Descriptions

Project #: 300612 Test: P-8330-COE-A

0001SA - 2-AGB

QUANTERRA-CALLAB LABORATORY
Analytical Bench Sheet

Project Number: 300612 Date: 27 JUL 98

Test Code: P-8330-COE-A Test Description: Prep - Explosives by HPLC

Turnaround Status: RUSH Sample Matrix: AQUEOUS Date Assigned:_____ Prep Completion Due:_____

SOP-Section/Page (Revision):_____

Extractions: [ ] Separatory Funnel [ ] Continuous Liquid/Liquid [ ] Sonication [ ] Soxhlet [ ] KD

[ ] Roto Vap [ ] Shaker [ ] Other_____

Samp ID	Holding Time	Sample Size	Extractn Date/ Initials	Init. pH	Adj pH Date/ Initials	Hydrolysis Date/ Initials	Acid Ext Date/ Initials	Conc Date/ Initials	CH2N2 Date/ Initials	N2 Date/ Initials	Final Sample Concentn
0001SA	31 JUL 98										

Project Due: 06 AUG 98 DCS With Project:_____ DCS Code:_____

LCS With Project:_____ LCS Code:_____

Method Blank With Project:_____ Extraction Includes Project(s):_____

Notes/Comments:
** PLEASE PUT COMMENTS ON ANOMALY SHEET ** MSQC With Project: _____ MSQC Code: _____

F1-84

Date To Instruments: By: Peer Approval By:

QUANTERRA-CALLAB LABORATORY
Quality Assurance Sheet

Project #: 300612 Test: P-8330-COE-A

Quality Assurance/Quality Control:

Sample ID:_____ Spiking Standard Name:_____

Spike Code ID:_____ Volume: _____ Conc: _____

Spiked By:_____ Witness:_____

Sample ID:_____ Spiking Standard Name:_____

Spike Code ID:_____ Volume: _____ Conc: _____

Spiked By:_____ Witness:_____

Sample ID:_____ Spiking Standard Name:_____

Spike Code ID:_____ Volume: _____ Conc: _____

Spiked By:_____ Witness:_____

Sample ID:_____ Spiking Standard Name:_____

Spike Code ID:_____ Volume: _____ Conc: _____

Spiked By:_____ Witness:_____

Solvent: Manufacturer And Lot Number:

Extracting Solvent:_____

Solvent Keeper:_____

Transfer Solvent:_____

Derivatizing Reagent:_____

Final Solvent:_____

Other:_____

_____

Was an aliquot taken? Y / N

If yes, how much was aliquoted?_____mL _____mL

What was the sample concentration before aliquot?_____

_____

Why was the aliquot taken?_____

_____

_____

_____

QUANTERRA-CALLAB LABORATORY
Instruction Sheet

Project #: 300612 Test: P-8330-COE-A

Sample Instructions:

None.

Test Instructions:

None.

Group Instructions:

None.

QUANTERRA-CALLAB LABORATORY
Anomaly Report Form

Project #: 300612 Test: P-8330-COE-A

Anomalies:	Sample(s) Affected:
__ Sample broken during shipping.	_____
__ Limited sample volume.	_____
__ Matrix required a subsample to facilitate extraction.	_____
__ pH of sample required excess acid/base (please circle).	_____
__ Solvent and sample were miscible.	_____
__ Emulsion problems.	_____
__ Emulsion problem required centrifuging.	_____
__ Sample lost during preparation. Re-extraction performed? Yes/No	_____
__ Concentration volume went lower than specified in the SOP.	_____
__ Concentration time longer than normal.	_____
__ Extraction solvent saturated with organic material, therefore sample volume was not concentrated but adjusted based on the screening.	_____
__ Final concentration greater than specified in the SOP due to the screening results.	_____
__ Precipitate observed in the extract.	_____
__ Other, please comment below.	_____

Comments:

Submitted By:_____ Date:_____

QUANTERRA-CALLAB LABORATORY
Prep (Extraction) Check List

Project #: 300612 Test: P-8330-COE-A

Chemist: _____ Date: _____

Reviewer: _____ Date: _____

Number of samples: 1 Number of extracts: _____

Benchsheet:	Initiated		Reviewed	
SOP section and page number filled out properly.	Y___	N___	Y___	N___
Each step of extraction procedure initial'ed and dated.	Y___	N___	Y___	N___
Anomalies or deviations from SOP documented.	Y___	N___	Y___	N___
pH adjustments noted.	Y___	N___	Y___	N___
Solvent manufacturer and lot number completed.	Y___	N___	Y___	N___
Volumes and weights of samples to two sig. figs.	Y___	N___	Y___	N___

QA/QC:	Initiated		Reviewed	
DCS/MS forms filled out properly.	Y___	N___	Y___	N___
Spike information correct on bench sheet.	Y___	N___	Y___	N___
Spike witness?	Y___	N___	Y___	N___
If not spiked per SOP, were spike amounts verified?	Y___	N___	Y___	N___

LIMS:	Initiated		Reviewed	
Extraction date (complete date) correct.	Y___	N___	Y___	N___
QC lot number present and correct.	Y___	N___	Y___	N___
QC Run number present and correct.	Y___	N___	Y___	N___
Prep released (date extract goes to Inst.).	Y___	N___	Y___	N___

Extracts:	Initiated		Reviewed	
Number of extracts agree with benchsheet.	Y___	N___	Y___	N___
Concentrations of extract agree with benchsheet.	Y___	N___	Y___	N___
Testtube rack labelled with project id and test.	Y___	N___	Y___	N___

Comments:

QUANTERRA-CALLAB LABORATORY
Client Descriptions

Project #: 300612 Test: P-8330-COE-A

0001SA - [redacted]

QUANTERRA / W. SACRAMENTO

PROJECT QC LIMITS

The S.M. Stoller Corporation

Project: CALLAB-300612
Matrix: AQUEOUS

Test Code: 8330-IRPMS-1C-A
Version: 3
QC Category: 8330-COE-A

S# NA		RL	Surrogate/SCS			Lab Control (LCS)			Lab Control (DCS)				Matrix Spike			
Modified Date:		25 JUL 98	24 OCT 95			24 OCT 95			24 OCT 95				20 OCT 94			
Component	CASN	ug/L	ug/L	LCL	UCL	ug/L	LCL	UCL	ug/L	LCL	UCL	RPD	ug/L	LCL	UCL	RPD
2,4-Dinitrofluorobenzene	70348	*	100	65	135	100	65	135	100	65	135	35				
HMX	2691410					50	65	135	50	65	135	35	50	65	135	35
1,3,5-Trinitrobenzene	99354					50	65	135	50	65	135	35	50	65	135	35
RDX	121824	5				50	65	135	50	65	135	35	50	65	135	35
1,3-Dinitrobenzene	99650	5.0				50	65	135	50	65	135	35	50	65	135	35
Nitrobenzene	98953	5.0				50	65	135	50	65	135	35	50	65	135	35
2,4,6-Trinitrotoluene	118967	5.0				50	65	135	50	65	135	35	50	65	135	35
Tetryl	479458	5.0				50	65	135	50	65	135	35	50	65	135	35
2,4-Dinitrotoluene	121142	5.0				50	65	135	50	65	135	35	50	65	135	35
2,6-Dinitrotoluene	606202	5.0				50	65	135	50	65	135	35	50	65	135	35
2-Am-DNT	35572782	5.0				50	65	135	50	65	135	35	50	65	135	35
4-Am-DNT	1946510	5.00				50	65	135	50	65	135	35	50	65	135	35
2-Nitrotoluene	88722	10.0				50	65	135	50	65	135	35	50	65	135	35
4-Nitrotoluene	99990	8.5				50	65	135	50	65	135	35	50	65	135	35
3-Nitrotoluene	99081	7.9				50	65	135	50	65	135	35	50	65	135	35

F-90

300551 300612

MASTER SHEET

Holding time Due: 7-29-98

PROJECT Due: 7-28-98

PROJECT NO. : 300592 300602

Page 1 of 1

INITIATED BY: B DANTON

DATE: 7-28-98

METHOD NO. : SUBP OP 0011 SMC

SAMPLE MATRIX: AQ

COMPOUND(S) : 8330 by HPLC/UV

COMPLETED BY:

DATE:

REVIEWED BY :

DATE:

APPROVED BY :

DATE:

STORAGE	Weigh	Fortification	Extract		Filter	Dilute		
WR1C WR1C WR1C W24h	770gm into 1L vol. flask BALQA051 BD 7-28-98	By BD 7-28-98 Witness [illegible] 7-28-98	1251.4 NACL +164 mL ACN Stir, Stand decant. +8mL ACN, Stir, Stand, decant BD 7-28-98	+85 mL SALT H2O Stir Stand, decant +2mL ACN Stir, Stand, decant	Bring to 5.0mL with ACN. +5ug Acetic Acid. Filter thru 25 min accu-disk BD 7-29-98	1 mL extract 3mL HPLC grade H2O BD 7-29-98		

Comments:

CAL ID/SAMPLE DATA	SAMPLE SIZE (gm)	FORTIFICATION - STANDARD DATA PPB	COMPOUND(S)	ALIQUOT	STD. CONC.	STD. NO.	(g/mL) Conc.	(g/mL) Dilution	
300592-MB	770						770/5	770/20	8330 Surrogate
28Jul98 13D	770	0.5	8330 mix	770	5.0ug/mL	E070898H	770/5	770/20	added to all
↓ 13 DD	770	0.5	↓	770	↓		770/5	770/20	40uL of 50ug/mL
300592-2	770						770/5	770/20	to yield 2.6ppb
300602-1	770						770/5	770/20	# E060898C
~~2X~~	770						770/5	770/20	
~~2MS~~	770	0.5	8330 mix	770	5.0ug/mL	E070898A	770/5	770/20	
✓ ~~2SD~~	770	0.5	↓	770	↓	↓	770/5	770/20	
300612-1	770						770/5	770/20	
300551-11	770						770/5	770/20	

EXACT COPY OF ORIGINAL

BOILER 5-26-94.

16-19