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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 03

July 02,1999

Prepared for:

SOUTHERN DIVISION
NAVAL FACILITIES ENGINEERING COMMAND
2155 EAGLE DRIVE
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U.S. EPA ID NUMBER [IN5 170 023 498]

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MK Program Manager	8/11/99 Date
MK Program Quality Control Manager	8/11/99 Date
US Navy Responsible Authority	9/14/99 Date
USEPA RCRA Project Coordinator	7-15-99 Date
USEPA Region 5 QAPP Coordinator	7-15-99 Date
Laboratory Representative	9-8-99 Date

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(NOTE: APPENDIX H IS IN A SEPARATE BINDER)

LIST OF RESERVED SECTIONS

These Sections will be submitted for approval at a later date as an Addendum to this QAPP.

Section 1.3.10.1 Current Status of ABG

Section 1.3.10.2 Current Status of Rockeye

Section 1.3.10.4 Current Status of Mine Fill B

APPENDIX G TOXICITY TEST PROCEDURE

SECTION 1
PROJECT DESCRIPTION

1.1 INTRODUCTION

1.1.1 Project Background

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

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

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

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

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

studies at several sites at NSWC Crane.

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

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

The RCRA Permit for NSWC Crane requires that RFI Phase I Environmental Monitoring Reports (EMRs) be submitted from many of the SWMUs at NSWC Crane. The purpose of the EMR is to identify and assess IRP Corrective Action SWMUs posing potential threat to human health or the environment due to contamination from past hazardous material operations. The EMRs for SWMUs #12/14 (Mine Fill A) and #13/14 (Mine Fill B) were completed in August 1992 (Halliburton NUS, 1992). A RFI for SWMU-10/15 (Rockeye Munitions Facility) was completed in July 1992 (ACOE, 1992a) and the RFIs for SWMU-03/10 (Ammunition Burning Ground) were completed in November 1992 (ACOE, 1992b) and September 1995 (ACOE, 1995).

In 1993, the U.S. Navy shifted emphasis from site investigations to actual cleanup activities. As a result of this decision, and to reduce or eliminate the potential for contaminant migration, NSWC Crane evaluated its 31 SWMUs to determine which ones would be amenable to accelerated cleanup actions, otherwise known as Interim Measures (IM). NSWC Crane submitted a proposal to the U.S. EPA to conduct IM cleanup actions at selected SWMUs. Four of those SWMUs have been identified for IM cleanup due to explosives contaminants in the soils and a fifth SWMU identified as the facility for treatment of the affected soil. Those SWMUs are as follows:

- SWMU-03/10, Ammunition Burning Ground (ABG)
- SWMU-10/15, Rockeye Munitions Facility (Rockeye)
- SWMU-12/14, Mine Fill A
- SWMU-13/14, Mine Fill B
- SWMU-33/00, Bioremediation Facility

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								
Pentaerythritol tetranitrate (PETN)	X	X	X	X	To be determined by U.S. EPA 1998	8330	0.25	~~0.25~~0.50
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	~~0.0100~~.020
Methyl ethyl ketone (2 - butanone)	XT	XT	XT		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	~~0.250~~.50
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~~0.020
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	~~0.250~~.50
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	~~0.0100~~.020
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								
Pentaerythritol 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	520
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	520
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 1-4c
REGULATORY LEVELS FOR TCLP ANALYSIS

TCLP Extraction	EPA #	Parameter	Reg. Level (mg/L)
Metals	D004	Arsenic	5.0
	D005	Barium	100.0
	D006	Cadmium	1.0
	D007	Chromium	5.0
	D008	Lead	5.0
	D009	Mercury	0.2
	D010	Selenium	1.0
	D011	Silver	5.0
GC/MS Volatiles	D018	Benzene	0.5
	D019	Carbon tetrachloride	0.5
	D021	Chlorobenzene	100.0
	D022	Chloroform	6.0
	D028	1,2 Dichloroethane	0.5
	D029	1,1 Dichoroethylene	0.7
	D035	Methyl ethyl ketone	200.0
	D039	Tetrachloroethylene	0.7
	D040	Trichloroethylene	0.5
	D043	Vinyl chloride	0.2
GC/MS Semivolatiles	D030	2,4 Dinitrotoluene	0.13
	D036	Nitrobenzene	2.0

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

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

4. Confirmation sampling (treated compost piles and excavations prior to backfilling).

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

1.1.3 QAPP Preparation Guidelines

This Quality Assurance Project Plan (QAPP) presents the organization, objectives, planned activities, and specific Quality Assurance/Quality Control (QA/QC) procedures associated with the Bioremediation Facility Full-Scale Operations. Sampling and data evaluation activities are summarized in Table 1-8. Specific protocols for sampling, sample handling and storage, chain-of-custody, and laboratory analyses will be described. All QA/QC procedures will be structured in accordance with applicable technical standards, certain U.S. Environmental Protection Agency's (U.S. EPA) requirements, regulations, guidance, and technical standards. This QAPP was prepared in accordance with U.S. EPA guidance manual entitled, "Region 5 Model RCRA Quality Assurance Project Plan," May, 1993 (U.S. EPA, 1993a).

1.2 SITE/FACILITY DESCRIPTION

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

1.2.1 Location

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

The ABG area is located in the eastern portion of NSWC Crane as shown in Figure 1-2. Access is by Highway H-462 via H-58, H-274 and H-463. The Rockeye Munitions Facility area is located in the northeastern portion of NSWC Crane at the intersection of Highways H-161 and H-45 as shown in Figure 1-2. Mine Fill A is located in the west-central portion of NSWC Crane situated along Highway H-17 as shown in Figure 1-2. Mine Fill B is located in the west-central portion of NSWC Crane situated along

Highway H-18 as shown in Figure 1-2.

1.2.2 Facility/Size and Borders

The ABG occupies approximately 20 acres and is located in a remote area near the east-center boundary of NSWC Crane. The site lies in the valley of Little Sulphur Creek (ACOE, 1992b).

Rockeye is a 40-acre tract in the north-central portion of NSWC Crane. It is situated on a flattened ridge crest which separates the headwater drainages of Sulphur Creek, First Creek, and Turkey Creek. The area is located on Highway H-45, approximately two miles south of North Gate #1 (ACOE, 1993a).

Mine Fill A consists of 29 buildings and is bordered to the north/northwest by Highway H-45 and Highway H-17 along its remaining perimeter. The entire site occupies approximately 70 acres (Halliburton NUS, 1992). Excavation will begin at Mine Fill A, around buildings 152, 153, 157, and 158 as shown in Appendix E of the Full-Scale Operations Plan (MK, 1998b).

Mine Fill B consists of 39 buildings and is situated along Highway H-18. The entire site occupies approximately 62 acres (Halliburton NUS, 1992).

1.2.3 Natural and Man-made Features

At the ABG a major drainage divide extends from southwest to the northeast along NSWC Crane Highway H-161. East of the divide, approximately two-thirds of the area drains to the south and southeast through Little Sulphur and Sulphur Creek. Ultimate drainage is to Indian Creek, the White River and then to the Wabash (ACE 1992b).

Drainage to the north and east of Rockeye goes to Sulphur Creek. Drainage to the south goes into (and is the origin of) Turkey Creek which flows into Boggy Creek. Drainage to the west goes into Greenwood Lake. Rockeye is a production facility and not a storage, treatment, or disposal site (ACOE, 1992a).

Buildings at Mine Fill A provide storage, receiving, and filling of munitions/bomb materials. Local facilities include Mine Fill B to the southwest, an inert storage area to the west, the pyrotechnics production area to the north, and the Supply Department, a restricted explosive area and high explosives magazines to the northeast (Halliburton NUS, 1992). Surface drainage is primarily off the eastern and western borders of Mine Fill A, via ditches and culverts. Surface discharge is forwarded to intermittent drainage ways which flow to Turkey Creek, which flows south-southwest to merge with Boggy Creek (Halliburton NUS, 1992).

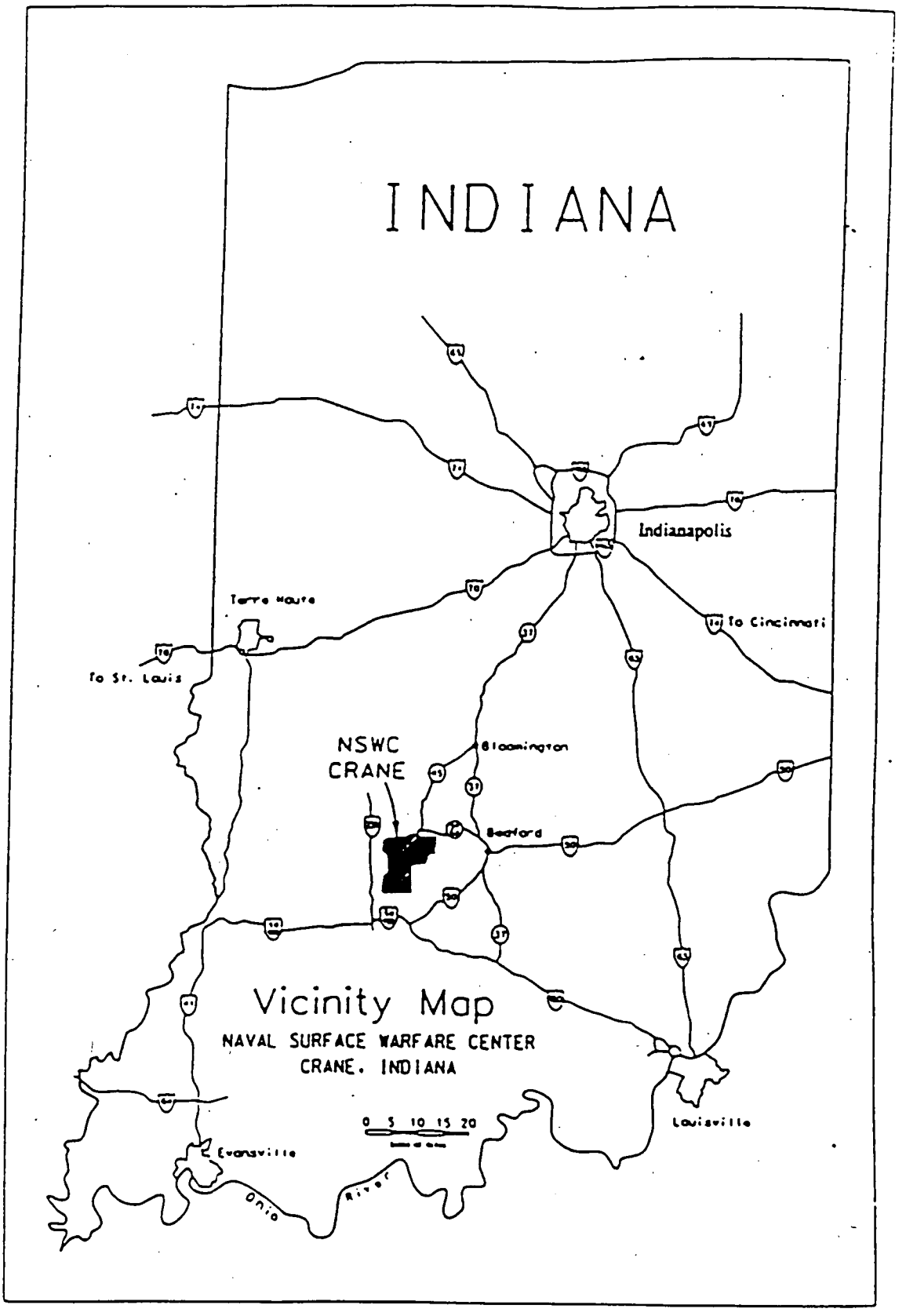
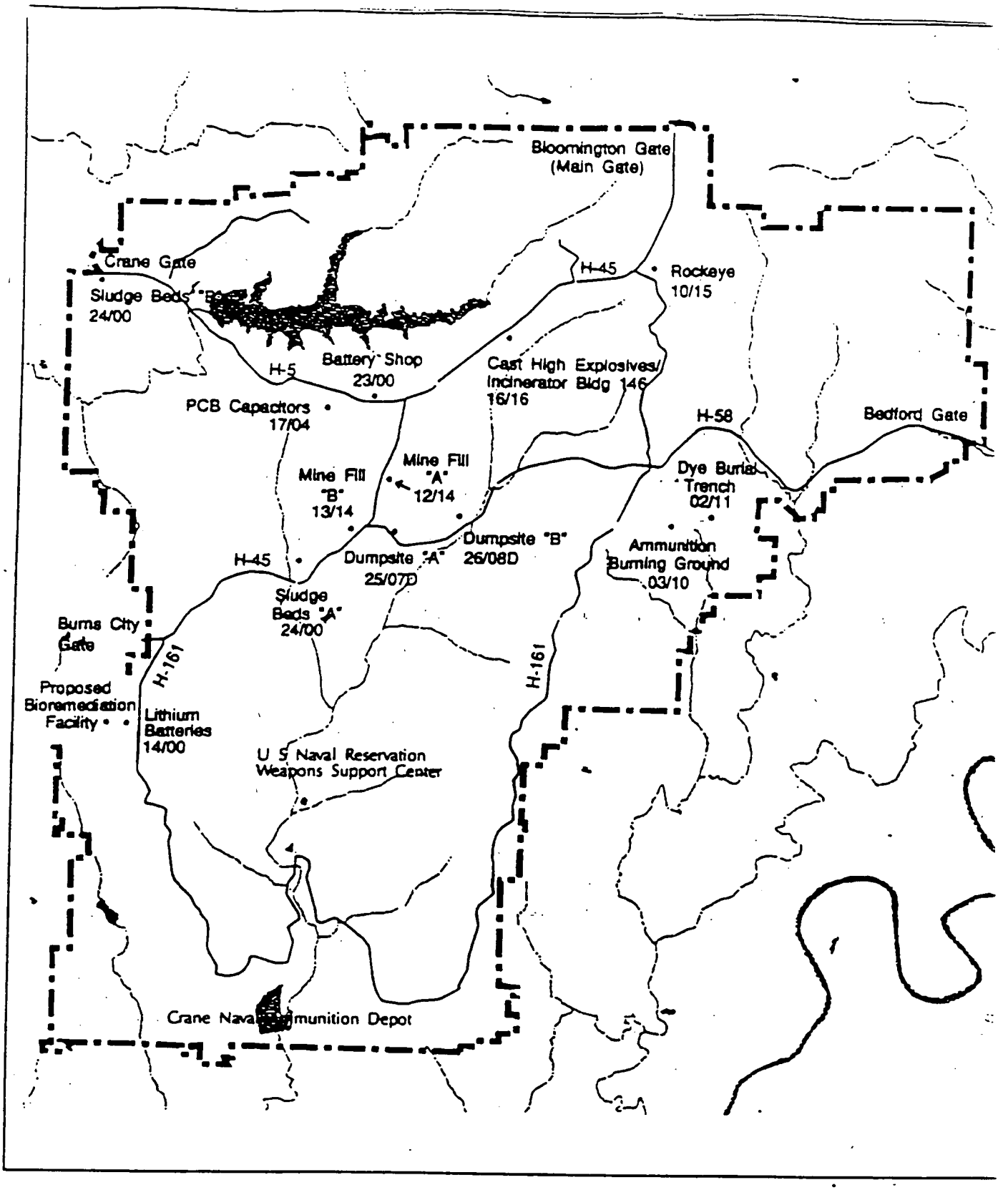


FIGURE 1-1 SITE LOCATION MAP



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

FIGURE 1-2 LOCATION OF SWMUs

MORRISON KNUDSEN CORPORATION

Mine Fill B was an operation similar to Mine Fill A. Nearby structures include magazines and loading and filling buildings to the west, bomb proof group and demolition area to the south, supply department to the east, and Mine Fill A to the northeast (Halliburton NUS, 1992). Surface drainage is routed via ditches and culverts to intermediate drainage-ways transcending slopes on the northwestern and southeastern boundaries of Mine Fill B. The majority of surface drainage is collected and drained towards the northwest and enters a southwest flowing intermittent tributary to Boggy Creek (Halliburton NUS, 1992).

1.2.4 Topography

The topography of ABG is rugged and the terrain heavily vegetated (ACOE, 1992b). Ground surface elevations in the area range from about 480 ft NVGD in the southeast to 845 ft along the major drainage divide (ACOE, 1992b). See Part IV of the August 1992 RFI (ACOE, 1992b) for information concerning the site's topography.

The topography of Rockeye is rugged and the terrain heavily vegetated (ACOE, 1993a). The Rockeye site was modified by the process of cut-and-fill to prepare the site for construction. Ground surface elevations in the area range from 610 ft NGVD in the valley of Sulphur Creek to approximately 860 ft NVGD along the divide immediately south of the facility. See Part IV of the December 1993 RFI (ACOE, 1993a) for information concerning the site's topography.

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

The topography of Mine Fill B is gently sloping from the northeast towards the southwest. Surface elevation varies from approximately 715 feet MSL in the northwest to approximately 657 feet MSL near the southwestern border. See Section 6.1.1.4 of the August 1992 EMR (Halliburton NUS, 1992) for information concerning the site's topography.

1.2.5 Local Geology

The ABG is in a bowl which opens to the southeast, high on the northwest, northeast and south and low in the middle where Little Sulphur Creek has incised its valley. Four geologic formations important to the hydrogeological investigation of the ABG comprise three aquifers, the Golconda-Haney limestone (the upper aquifer), the Big Clifty sandstone/Beech Creek limestone (the middle aquifer), and the Beaver Bend limestone (the lower aquifer). See Part IV of the August 1992 RFI (ACOE, 1992b) for information

concerning the site's geology.

The Pennsylvanian aged Raccoon Creek (probably Mansfield Formation) occupies the Rockeye area above approximately 680 ft elevation, including all of the ridge on which the Rockeye facility is situated. Several Mississippian-aged units are exposed in the tributary and main valleys of Sulphur Creek to the east and in the headwaters of First and Turkey Creeks to the west of the facility. See Part IV of the December 1993 RFI (ACOE, 1993a) for information concerning the site's geology.

Mine Fill A is covered with well-drained to moderately well-drained soil consisting of a broad range of grain sizes, depending on depth. Surface soils include a mixture of clay, silty clay and silt. Deeper soils include clay, sandy clay, silty sand and silty gravel. Depth to bedrock (Pennsylvanian Age Raccoon Group - sandstone, shale, limestone) ranges from 45 to over 65 inches, depending on earth moving activities in the area. See Section 5.1.1.6 of the August 1992 EMR (Halliburton NUS, 1992) for more information.

Mine Fill B is covered by Zanesville-Udorthents complex soil. Zanesville soil is commonly found on ridge tops in upland areas. Udorthents occur in areas which have been affected by earth moving activities. Surface soil include a mixture of clay, silt, and silty clay. Deeper soil include clay, sandy clay, silty sand and silty gravel. See Section 6.1.1.6 of the August 1992 EMR (Halliburton NUS, 1992) for more information.

1.3 SITE/FACILITY HISTORY

1.3.1 General History - NSWC Crane

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

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

In 1976, NAD Crane was renamed NSWC Crane. Its new mission was to provide support for ships and crafts equipment, shipboard weapons systems and assigned

ordnance items, and to perform additional functions as directed.

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

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

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

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

1.3.2 ABG History

Ordnance and ordnance-contaminated materials from NSWC Crane production areas have been taken to the ABG for treatment by burning since the 1940s. The ABG is used extensively for destroying unwanted materials contaminated with explosives, bare explosives, rocket motors, candles, flares, solvent, detonators, and fuse materials. Several separate burning areas are located within the site proper. The largest quantities of materials were destroyed from 1956 to 1960, when some 15,000 pounds per day of smokeless powder was destroyed. In the same period, about 46,000 pounds per day of high explosives were burned. The area is also used for flashing the residue from bombs and projectiles after they have been subjected to melt out or drill out operations for removal of the bulk of the explosives. A depression was constructed to burn the solvents and some other materials. This depression was lined with fire-proof bricks. This depression is the area of concern for trichloroethylene contamination. More details on site history may be found in Section 1.4 in the September 1995 RFI

(ACOE, 1995).

1.3.3 Rockeye History

Rockeye is a production facility and not a storage, treatment, or disposal site. Operations began in the mid-1950's as press-loading operation for 3-inch projectiles using Composition A-3 explosive (RDX and wax). In 1967-68, the facility was converted to a case-filling operation in order to produce the MK20 series anti-tank Rockeye cluster bomb. As part of the loading operation, the system generates a large volume of wastewater. The wastewater is collected in four sumps which are periodically pumped and the residue trucked to the ABG for disposal. Prior to 1978, explosives-contaminated waters for full sumps were discharged into the local streams. On the north side of the facility, the waters were released to a branch of Sulphur Creek, and on the south side the waters were released to Turkey Creek. In the spring of 1978, an activated carbon water treatment facility was brought into operation to purify the wastewater for recycled usage. In addition to the water treatment system, a scrubber system to remove contaminated particulates in the steam-fed tray wash area was installed. With the installation of the pollution abatement equipment, the release of explosive-contaminated waters has declined (ACOE, 1992a). More details on production may be found in Section 1.3.0 of the July 1992 RFI (ACOE, 1992a).

1.3.4 Mine Fill A History

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

1.3.5 Mine Fill B History

Operations at Mine Fill B were similar to those at Mine Fill A. Bomb loading operations were suspended at Mine Fill B in 1973. The site is currently used for renovation and rework of munition items. More details on production may be found in Section 6.4.1 of the August 1992 EMR (Halliburton NUS, 1992).

1.3.6 Past Data Collection Activities - ABG

The results of the Resource Conservation and Recovery Act (RCRA) Facility Investigation (RFI) Part 2, Phase III Soils Study for the ABG, SWMU-03/10 indicated

total xylene, ethylbenzene and toluene were detected in surface soil at concentrations ranging from 0.00061 to 0.0091 mg/kg. Trichloroethylene (TCE) was detected at a maximum concentration of 0.017 mg/kg. This TCE value falls below the risk-based concentration (RBC) of 3 mg/kg, for a soil to air inhalation scenario (USEPA, 1994a) and ~~does~~ did not exceed residential values for soil ingestion (58 mg/kg). Polynuclear aromatic hydrocarbons (PAHs) were detected in 9 of 33 samples collected. The highest PAH concentration detected was 0.37 ppm [benzo(b)fluoranthene]. All detected concentrations were below RBCs (ACOE, 1995).

In addition, eight explosives (HMX; RDX; TNT; TNB; 2,4-DNT; 2,6-DNT; 2A-DNT; and 4A-DNT) were detected in surface soils collected during the August, 1993 study. The maximum detected concentrations for HMX, RDX and TNT were 223 mg/kg, 1820 mg/kg and 136 mg/kg, respectively. Concentrations of metals detected during the study were compared to background levels for the site. Calcium, cadmium, copper, lead and zinc were found in surface soils at 100 times background levels. Magnesium, silver, tin and phosphorus were detected at concentrations 10 times above background levels (ACOE, 1995).

Based on the previous sampling data, an estimated 60,000 cubic yards of explosives-contaminated soil will be excavated at the ABG area. Additional site characterization and details of the excavation activities will be provided in Appendix E of the Final Full-Scale Operational Plan as an addendum at a later date, prior to excavation activities at ABG.

1.3.7 Past Data Collection Activities - Rockeye

The subsurface soil explosive concentrations range up to 42.7 mg/kg for individual constituents. Surface soil samples contained explosive concentrations ranging from non-detects to as high as 10,400 mg/kg (HMX). DNB, DNT, and TNB were found at concentrations below quantitation limits (the concentration reported was estimated) and tetryl was not detected at all. TNT was found in surface samples at a maximum concentration of 295 mg/kg and at a maximum level of 1.40 mg/kg in subsurface samples. An RDX concentration of 3,350 mg/kg was found in surface samples. HMX was found in more samples and at higher concentration than any other explosive compound analyzed. Subsurface soil samples contained HMX at concentrations above quantitation limits. HMX concentrations of 1,960 and 10,400 mg/kg were found in surface soil samples from two areas. The surface soils beneath the exhaust of a Rockeye Building 2734 ventilator contained concentrations of TNT, RDX, and HMX at 295, 3,350, and 10,400 mg/kg, respectively. These concentrations were the maximums for these compounds in the Rockeye soil samples (ACOE 1992a).

Comparison of the maximum metals concentrations of Rockeye subsurface soils to background subsurface soils does not appear to yield conclusive evidence as to whether or not a release of metals has occurred at Rockeye. Comparison of metals

analyses of Rockeye surface soils with background samples indicates that maximum concentrations in Rockeye surface soils were higher than the background, except for nickel and antimony (ACOE 1992a).

Based on the previous sampling data and locations, the areas below the ventilator exhausts at Buildings 2731 and 2734 have been selected as excavation sites for the IM cleanup discussed in this QAPP. In addition, the sumps, drainage ways, and drainage way outfalls will be excavated and treated during this IM cleanup.

An estimated 18,000 cubic yards of explosives-contaminated soil will be excavated at the Rockeye Munitions area and transported to the Bioremediation Facility for composting. Additional site characterization and details of the excavation activities will be provided in Appendix E of the Final Full-Scale Operational Plan as an addendum at a later date prior to excavation activities at Rockeye.

1.3.8 Past Data Collection Activities - Mine Fill A

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

Several soil borings were installed in the vicinity of Buildings 153 and 158 in 1985. Results are summarized in Table 1-6, with estimated locations provided in Appendix E of the Full-Scale Operations Plan (MK, 1998b). The samples were analyzed for TNT, RDX and HMX only, and results showed the highest levels of explosives are in the vicinity of Building 153. Further discussion of surface water, ground water and soil sampling results may be found in Section 5.3.2 of the August 1992 EMR (Halliburton NUS, 1992).

TABLE 1-6
SUMMARY OF PREVIOUS SOIL BORING RESULTS - MINE FILL A

Sample I.D./Location	TNT (ppm)	HMX (ppm)	RDX (ppm)
MFA-1/45 feet away from northeast corner of box bin at Building 153	69	292	2,620
MFA-2/at base of berm 10 feet south of box bin	3,790	1,340	15,300
MFA-3/10 feet out from end of drain pipe	8	908	8,150
MFA-4/10 feet down ditch from MFA-3	<3.6	64	374

TABLE 1-6
SUMMARY OF PREVIOUS SOIL BORING RESULTS - MINE FILL A

Sample I.D./Location	TNT (ppm)	HMX (ppm)	RDX (ppm)
MFA-5/28 feet east of Building 158, in ditch	64	99	906
MFA-7/two feet southeast of static pole south of Building 158, in run-off ditch	<3.6	<5.0	<7.2
MFA-8/near base of berm, 15 feet west of corner of box bin	5.2	52	349

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

Additional analytical results from recent sampling events prior to Bioremediation Facility Pilot-Scale Operations indicate levels of TNT, RDX, and HMX as high as 3,000, 14,000, and 1,200 mg/kg respectively, north of Building 153. Recent sampling has also been performed in areas west of Building 158. These analytical results show elevated levels of TNT, RDX, and HMX as high as 260, 830, and 100 mg/kg respectively, in the area west of Building 158. Areas around Buildings 152 and 157 are also identified as potentially contaminated soil sources. Visual inspections and additional field sampling shall be conducted to identify contaminated soil sources beyond the grids identified. A summary of the 1997 sample results with chains of custody are provided in the Full-Scale Operational Plan, Appendix E (MK, 1998b).

1.3.9 Past Data Collection Activities - Mine Fill B

Concentrations of individual explosives in soils ranged from undetected to 24,000 mg/kg (RDX). TNT and HMX were reported as high as 2410 mg/kg and 2020 mg/kg, respectively. Based on operational history, physical condition, location, waste characteristics, visual evidence, and analytical data, releases to groundwater, surface water, soil, and air have occurred (Halliburton NUS, 1992). Based on the previous sampling data and locations, the areas around Buildings 166, 167, 168, 171, 172, and 173 have been selected as excavation sites for soil interim measures cleanup.

An estimated 18,000 cubic yards of explosives-contaminated soil will be excavated at the Mine Fill B area and transported to the Bioremediation Facility for composting. Additional site characterization and details of the excavation activities will be provided in Appendix E of the Final Full-Scale Operational Plan as an addendum at a later date prior to excavation activities at Mine Fill B.

1.3.10 Current Status of SWMUs

1.3.10.1 Current Status of ABG

This Section will be submitted for approval at a later date as an Addendum to this QAPP.

1.3.10.2 Current Status of Rockeye

This Section will be submitted for approval at a later date as an Addendum to this QAPP.

1.3.10.3 Current Status of Mine Fill A

Mine Fill A is currently an active site. As of 1992, Mine Fill A was used for the production of 2,000-pound aerial bombs. Demilitarization activities are also conducted at Mine Fill A. Activities include the application of enamel and bituminous solvent coatings, rotary grit blasting, spray painting, propellant removal, and fuse and configuration changes. Ordnance compound particulate matter was exhausted through the ventilation system. Rinse and waste water containing TNT, Composition B, HBX-1, HBX-3, and H6 are potential sources. Chemical composition of these and other wastes generated at Mine Fill A are provided in Table 1-7.

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

Based on reports and documents reviewed for the site, and a current assessment of available information, concentrations of individual ordnance compounds in soils ranged from undetected to 15,300 mg/kg (RDX). It was not verified whether volatile organic compounds (VOCs) were tested or detected in Mine Fill A soils. Five paint booths are authorized to operate in Building 155, and are equipped with particulate control features. However, volatile organics samples will be collected during initial excavation site characterization, based on field observations, and if paint wastes were associated with Buildings 153 or 158.

Approximately 11,000 cubic yards of impacted soil within the Mine Fill A are targeted for interim measures cleanup. Based on previous soil boring results (Table 1-6), the areas around Building 153 show higher levels of explosives contamination than Building 158.

TABLE 1-7
SUMMARY OF CHEMICALS PRESENT AT MINE FILL A

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

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

1.3.10.4 Current Status of Mine Fill B

This Section will be submitted for approval at a later date as an Addendum to this QAPP.

1.4 PROJECT OBJECTIVES

1.4.1 Specific Objectives and Associated Tasks

For this full-scale operation, it will be necessary to gather sufficient information to evaluate the soil excavated for treatment and the effectiveness of treatment on the final composted soil. Associated data collection activities are summarized in Table 1-8 and as follows:

TABLE 1-8
DATA COLLECTION ACTIVITIES

TASK NUMBER	DATA COLLECTED	DATA USE	DATA QUALITY LEVEL
1. Pre-excavation Site Characterization	-SWMU-specific constituents per Section 1.4.2.1 -TCLP Metals (regulated list) -TCLP Volatiles (regulated list) -TCLP Semi-VOC (Compounds of concern)	-identify areas for excavation -determine "startup" levels of explosives to be treated -determine if SWMU-specific chemicals are ~~present~~greater than clean-up goals in soil to be treated -determine if soil is hazardous with respect to metals ~~and specific~~, VOCs and/or specific SVOCs of concern	-NEESA Level C QA/QC
2.Decon/wash water from Biofacility and Excavation Sites and Building sump water Sampling	-Explosives (8330) -BOD, COD, TSS, Ammonia-N, Oil and Grease -Metals (cadmium, chromium, copper, lead, nickel, zinc, silver, mercury) -Total cyanide	-results used for discharge to on-site treatment system -determine levels of explosives entering treatment system -metals for informational (loading) purposes	-NEESA Level C QA/QC
3. Storm-Water Retention Pond Sampling	-NPDES parameters (see Table 4-1)	-determine if NPDES criteria are met for discharge to a permitted outfall	-NEESA Level C QA/QC

TABLE 1-8
DATA COLLECTION ACTIVITIES

TASK NUMBER	DATA COLLECTED	DATA USE	DATA QUALITY LEVEL
4. Windrow Process Sampling (plus Confirmation Sampling - At end of cycle, estimated to be Day 28)	-Explosives, pH, %moisture, temperature, oxygen (field tests) -SWMU-specific constituents per Section 1.4.2.1 -Explosives (8330)	-measure the effectiveness and progress of the compost cycle (explosives~~, pH, %moisture, temperature, oxygen~~ will be tested at day 0, 7, 14, 21, and end of cycle; pH, %moisture, temperature, oxygen will be tested daily; SWMU-specific constituents will be tested at day 0 and end of cycle, if found at the excavation sites in excess of the remedial goals listed in Table 1-2) -determine if process goals have been met with respect to explosives	Qualitative for field tests -NEESA Level C QA/QC for off site lab analysis
5. Post-Excavation Sampling	-SWMU-specific constituents per Section 1.4.2.1.	-determine the levels of explosives and SWMU-specific contaminants remaining in soil after the excavation is complete and prior to backfilling.	-NEESA Level C QA/QC

Notes:
1. Methods for parameters listed under "data collected" are off-site analytical methods, unless otherwise noted.

Data Collection Task 1 - Initial Excavation Site Characterization

Previous data collected at the excavation sites (ABG, Rockeye, Mine Fill A, and Mine Fill B) indicates that high levels of explosives contamination were detected. Although explosives were detected in high concentrations, it is not clear if soil in these SWMU sites is non-hazardous, with respect to 40 CFR 261. The full-scale phase will focus on explosives and site-specific chemicals listed in Section 1.4.2.1 of this QAPP.

This task has three analytical objectives:

1. Analyze the excavated soil for explosives. This data will be used to identify areas to be excavated for Full-Scale Operations. This data will also be used as "entry " concentrations for composting.

2. Analyze the soil for SWMU-specific contaminants to determine if other ~~waste products~~target compounds are present. Certain observations will dictate a need for VOCs testing. Observations and criteria include visibly stained soils, unusual odors, and PID readings exceeding resident background levels. If one or more of these criteria is discovered, additional samples for VOCs will be collected. Analysis will be approved by the Project Manager and U.S. Navy designee, as required. It is anticipated that "low level" (if any) VOC contamination will be present.

3. Analyze the soil to determine if it is hazardous with respect to metals~~ and~~, VOCs, and semivolatiles. Soil will not be accepted into the Biofacility for treatment if it is hazardous with respect to these constituents. Toxicity characteristic levels are shown in Table 1-4C

Based on generator knowledge and process operations at a particular SWMU additional analytical testing of constituents may be required to ensure that solvent-contaminated soil or other listed or characteristic wastes are not transported to the Bioremediation Facility for composting. If metals and/or organics (VOCs and semivolatiles) are present in soil at a particular SWMU, the soil will be analyzed for toxicity characteristic leaching procedure (TCLP) metals and/or organics. However, metals and organics will be analyzed for total metals and total organics and the results based on the correction factor of 20 will be used to check if the values are below the TCLP regulatory limits as specified in 40 CFR Part 261 (see Table 1-4c). If the total analyses results with the correction factor indicate values are higher than the regulatory limits, then TCLP analysis will be requested to verify the total metals and/or organics results.

Additionally, some of the SWMU-specific constituent analysis (e.g., SVOCs, PCBs, Dioxins, etc.) will be performed only at areas within that SWMU known to be impacted by these constituents. Past operations and generator knowledge of the process at a particular SWMU will be used to define areas requiring these analysis. These areas will be identified in the SWMU-specific Soil Excavation Plan (Appendix E of the Full-Scale Operational Plan) to be issued to U.S. EPA for review and approval at a later date.

A grid system specified in Appendix E of the Full-Scale Operations Plan will be established for soil sampling. The grid layout and boundaries will be affected by the presence of buildings, utilities, etc. Grid borders will be flagged in the field, and areas will be chosen which will be accessible by heavy equipment (i.e. individual grid blocks will not be placed in the midst of heavy utilities or surface structures). Grid blocks are not expected to be perfectly square (i.e. those in the berm).

Soil samples will be collected from each grid block or sampling area. Five grab samples will be collected: one from the center and one from near each of the four corners of each grid block. Grab samples will be composited using a procedure which

will provide a more representative sample, as explosives contamination is known to vary over short distances (U.S. EPA, 1996a). A total of two composites, one for the surface to 12-inch depth and one for the two to three-foot depth, will be collected per grid block. Each composite sample will be analyzed for SWMU-specific contaminants. A minimum of one sample for SWMU-specific volatile organics analysis will be collected from the center of each grid block at the 12 to 18 inch depth and will not be composited.

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

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

Decontamination procedures described in Section 7.0 of the Full-Scale Operational Plan (MK, 1998b) will yield a quantity of water which may be used for windrow moisture addition. If found unsuitable for compost moisture addition, the rock wash and other decontamination fluids will be disposed of as specified in the Full-Scale Operational Plan (MK, 1998b).

Data Collection Task 3 - Storm-Water Retention Pond Sampling

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

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

Data Collection Task 4 - Process Sampling

Process parameters which will be tested on-site include pH, temperature, oxygen, and % moisture. These parameters are further discussed in Section 6.0 of the Full-Scale Operational Plan (MK, 1998b).

Explosives (full Method 8330 list) will be analyzed at Day 0 and at the end of the cycle (estimated to be 28 days). Off-site analysis for other SWMU-specific contaminants in the compost material will only be required if they are found above the cleanup criteria in the initial soil characterization sample results.

Confirmation sampling will not be completed unless concentrations of TNT and RDX are at or below cleanup levels based on Field Screening results. Confirmation sample

results will be compared to those levels in Table 1-2 to determine if explosives are below cleanup levels.

If treated soil does not meet explosives cleanup levels, one of the options discussed in Section 5.6 of the Full-Scale Operations Plan (MK, 1998b) will be executed as directed by NSWC Crane EPD.

Data Collection Task 5 - In-process Excavation Sampling

Grids, as specified in the Soil Excavation Plan, Appendix E of the Full-Scale Operations Plan (MK, 1998b), will be established for soil sampling of the excavation sidewalls and bases at each SWMU for the purpose of assessing horizontal and vertical extent of contamination, based on results of TNT and RDX field screening. As the contaminated areas are excavated, delineated by horizontal field screening, additional field screening will be used to determine the vertical extent of contamination. Once results indicate concentrations in the excavation area soil are below the established cleanup levels (TNT/RDX), confirmation soil samples will be collected at the intervals specified in the Soil Excavation Plan, Appendix E of the Full-Scale Operations Plan (MK, 1998b) and submitted for off-site laboratory analysis.

Data Collection Task 6 - Post-Excavation Sampling

Once soil has been removed, the excavation will be sampled for confirmation of attaining cleanup levels.

A sampling grid will be laid out in the base of the excavation(s), with a nodal spacing as specified in Appendix E of the Full-Scale Operations Plan (MK, 1998b). Samples will be collected six inches below ground surface from each grid block (base) and analyzed for SWMU-specific compounds. Sidewalls will be sampled every 20 feet and will not be gridded. Five grab samples will be collected: one from the center and one from near each of the four corners of the grid block. Grab samples will be composited using a procedure which will provide a more representative sample, as explosives contamination is known to vary over short distances (U.S. EPA, 1996a). The composite sample will be analyzed for SWMU-specific compounds. A minimum of one sample for SWMU-specific volatile organics analysis will be collected from the center of each grid block and will not be composited.

1.4.2 Project ~~Target~~SWMU-Specific Constituents and Laboratory Data Use

1.4.2.1 Target Constituents

Three primary target constituents have been identified for the Full-Scale operations: TNT, RDX and HMX. These parameters are present in many of the waste streams and have been detected in soil and surface water.

The constituents of concern for Mine Fill A (SWMU-12/14) are listed below. They are derived from the Resource Conservation and Recovery Act (RCRA) Facility Investigation (RFI) Phase I Environmental Monitoring Report for Mine Fill A (Halliburton NUS, 1992).

Explosives: Penta erylthritol tetranitrate (PETN); TNT; RDX; HMX; Tetryl; trinitrobenzene (TNB); 1,3-dinitrobenzene (DNB); nitrobenzene (NB); 4-amino-2,6-dinitrotoluene (4-Am-DNT); 2-Amino 4,6-DNT (2-Am-DNT); 2,4-dinitrotoluene (DNT); 2,6-DNT; 2-nitrotoluene (NT); 3-NT; and 4-NT.

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

Metals: Aluminum; barium; cadmium; chromium (total); mercury; and lead.

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: Dichloromethane (methylene chloride); acetone; methyl ethyl ketone (2-butanone); methyl isobutyl ketone; toluene; and xylene (total).

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.

Other Constituents: Calcium chloride, ammonium nitrate, N-N' Ethylene bisstearamide, bituminous asphalt, micro-crystalline wax, ethanol, isopropanol, methanol, and naptha may also be found at Mine Fill A, but are not being evaluated in this study.

The constituents of concern for Mine Fill B (SWMU-13/14) are listed below. They are derived from the RFI Phase I Environmental Monitoring Report for Mine Fill B [Halliburton NUS, 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; barium; cadmium; chromium (total); mercury; and lead.

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: Dichloromethane (methylene chloride); acetone; methyl ethyl ketone (2-butanone); methyl isobutyl ketone; toluene; and xylene (total).

Note: The following list of VOCs will be evaluated for waste characterization of Toxicity Characteristic (TC): Methyl Ethyl Ketone (MEK).

PCBs: Aroclor 1242; Aroclor 1254; and Aroclor 1260.

Other Constituents: Calcium chloride, ammonium nitrate, ethanol, isopropanol, methanol, and naptha may also be found at Mine Fill B, but are not being evaluated in this study.

The constituents of concern for ABG (SWMU-03/10) are listed below. They are derived from the Part 2 RFI Phase III Soils Report for ABG [ACOE, 1995] and Current Contamination Conditions Risk Assessment [BRE, 1997].

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; chromium (total); cobalt; copper; lead; manganese; mercury; nickel; ~~silver~~; thallium; vanadium; beryllium; cyanide; 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, Silver.

VOCs: Acetone, 1,1-dichloroethylene; 1,2-dichloroethylene, trichloethylene (TCE); 1,1,1 Trichloroethane; 1,1,2 Trichloroethane; Dichloromethane; MEK; 1,1,2,2-tetrachloroethane; ~~trans-1,2-dichloroethylene~~; vinyl chloride; chloroethane; chloroform; and tetrachloroethylene.

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: Bis(2-ethylhexyl) phthalate; di-n-butyl phthalate; N-nitroso-di-n-phenylamine; 4-nitrophenol; benzo(a)anthracene; chrysene; and

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 1-9
PROCESS MONITORING DATA COLLECTION TASKS

TASK	DATA COLLECTED	DATA USE	DATA QUALITY LEVEL
Oxygen level monitoring	oxygen, %	monitor the effectiveness and progress of the compost cycle	field level
Temperature monitoring	temperature, °C		field level
Moisture level monitoring	moisture, % WHC		field level
pH level monitoring	pH		field level
Field level data is characterized by the use of portable instruments which can provide real-time data. Instrument calibration and correct data interpretation are required for data to be acceptable.			

Oxygen Level Monitoring

Oxygen levels will be monitored to ensure aerobic conditions are present in the windrows. Oxygen measurements will be collected each day prior to and following turning of the windrow.

Temperature Monitoring

Windrow temperature will be the primary indicator of successful operation of composting operations. Windrow temperature measurements will be collected each day prior to and following turning of the windrow.

Oxygen Level Monitoring

Oxygen levels will be monitored to ensure aerobic conditions are present in the windrows. Oxygen measurements will be collected each day prior to and following turning of the windrow.

Temperature Monitoring

Windrow temperature will be the primary indicator of successful operation of composting operations. Windrow temperature measurements will be collected each day prior to and following turning of the windrow.

Moisture Level Monitoring

Moisture level will be monitored to ensure optimum operating conditions for microbial growth and contaminant degradation. Moisture level measurements will be collected three times per week prior to windrow turning. If additional moisture is added during windrow turning, a moisture level measurement will be collected following turning of the

windrow.

pH Level Monitoring

pH level measurements will be collected once per week. pH data will be recorded but will not be used to change operating conditions.

1.4.4 Data Quality Objectives

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

Level C of NEESA is defined as a site near a populated area, not on the NPL, and not likely to be undergoing litigation, but includes review and approval of the Laboratory QA and Site Work Plan. It allows the use of non-CLP methods but all methods must be EPA method or equivalent. The lab must successfully analyze a performance sample, undergo an audit, correct deficiencies, and provide monthly progress reports (MPRs) on QA.

The intended data quality objectives for precision, accuracy and completeness for laboratory data are summarized in Tables 3-1 through 3-7.

The overall objective of collecting field measurements of process parameters is to obtain data of sufficient quality and quantity to determine that acceptable operating conditions are maintained in the windrows. The specific objective of each process monitoring sampling event is to collect representative data for the purpose of evaluating the variance in each process parameter (oxygen, temperature, moisture, pH) among samples from different locations within a single windrow.

The objective of the field test kit monitoring for RDX and TNT will be to assist in the delineation of approximate excavation limits and the process monitoring of explosive degradation within the compost piles to determine when final confirmation samples should be collected. An off-site laboratory will be used for the final confirmation analysis in both cases. Since the primary purpose of the test kits will be to approximate concentrations to help refine when analytical samples should be collected for off-site laboratory analysis, the accuracy of the measurement is not critical. A comparison of soil field test kit measurement was performed for soil samples collected as part of the pilot-scale operations. A summary of this comparison including all data is provided in Appendix H. Review of results of these tests show that the field test kits are acceptable

for use for the intended objectives. A similar comparison of the results for the compost samples will be performed during the full-scale operation.

The analytical level of process monitoring data will be field level for in-situ field measurements, as shown in Table 1-9. Measurement methods and acceptable reporting limits for the various field measurement parameters are presented in Table 1-10. The data quality objectives for precision, accuracy and completeness are described in Section 5.

1.5 SAMPLE LOCATIONS

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

TABLE 1-10
FIELD MEASUREMENT PARAMETERS AND ASSOCIATED MEASUREMENT RANGES AND ACCURACY ~~RANGES~~CRITERIA

PARAMETER	MEASUREMENT METHOD	MEASUREMENT RANGES AND ACCURACY ~~RANGES~~CRITERIA
Oxygen	oxygen probe	range = 0 - 21% in 1% increments
Temperature	hand-held compost temperature probe	range = 0 to 93°C in 1° increments or 0 to 200° F in 1° increments
Moisture Level	sample weighing and drying	weights reported to the tenth of a gram
pH	pH meter	range = 4.0 to 10.0; pH reported to the tenth of a pH unit
TNT and RDX	colorimetric	range = 1 to 30 ug/g; 8% RSD

1.5.1 Sample Network by Task and Matrix

Analytical constituents per task are summarized in Table 1-11. This is a summary of parameters relating to the full-scale operations. Sample totals are provided in Table 4-6, Section 4.0.

1.5.2 Excavation Site Maps of Sampling Locations

Sample location maps will be generated prior to mobilization to the SWMUs. Sampling areas for Mine Fill A are discussed in Section 4.0 of this QAPP. Depending on the nature of encountered field conditions some of these locations may be changed.

Sampling locations will be finalized/approved by the Project Manager or designee, as overseen by the Navy, EPA and/or IDEM representatives.

1.5.3 Process Monitoring Measurement and Sample Locations and Frequency

Locations for process monitoring measurements and samples are described in Section 4.0 and Field SOP QAPP- 4.0, in Appendix A of this QAPP. A summary of the process monitoring schedule is presented in Table 1-12.

TABLE 1-11
SUMMARY OF SAMPLING PARAMETERS PER TASK

Task	Analytical Method Requested	Parameters to be Reported
1. Initial excavation site (soil) Characterization [NOTE: Parameters to be analyzed will be based on the chemicals of concern at a particular SWMU site per Section 1.4.2.1.]	-Site-specific VOCs (Method 8260) -TCLP VOCs (TCLP regulated list using Method 8260) (NOTE: one sample will be collected using EnCore samplers for VOCs analysis, covering both lists) -Explosives (Method 8330) -SWMU-specific metals (6010; 7471 - mercury) -TCLP-metals (1311, 6010, 7470) -SVOCs (Method 8270) -PCBs (Method 8081) -Dioxins (Method 8290)	-per Section 1.4.2.1 -benzene, carbon tetrachloride, chloroform, chlorobenzene, 1,2-dichloroethane, 1,1-dichloroethene, methyl ethyl ketone, tetrachloroethene, trichloroethene, vinyl chloride -full list for explosives -per Section 1.4.2.1 -regulated list only -per Section 1.4.2.1 -per Section 1.4.2.1 -per Section 1.4.2.1
2. Equipment Wash Water/ Decontamination Water Sampling	-Explosives (Method 8330) -POTW requirements -RCRA metals	-full Method 8330 list -dependent on POTW -Methods 6010 and 7470
3. Storm-Water Retention Pond Sampling	-TSS (EPA 160.2) -COD (EPA 410.4) -BOD (EPA 405.1) -Oil and Grease (EPA 413.1) -Nitrate (EPA 300.0) -Ammonia-N (EPA 350.2)	list as shown

TABLE 1-11 (continued)
SUMMARY OF SAMPLING PARAMETERS PER TASK

Task	Analytical Requested	Parameters to be Reported
4. Process Sampling and Confirmation Sampling (compost)	-Explosives (field test kits) (Operational on Days 7, 14 and 21) -moisture (WHC), oxygen, and pH per SOP QAPP-4.0	~~-full list for explosives~~ -TNT and RDX -moisture, oxygen, and pH
	[Note: The following analysis will be performed at Day 0 and at end of cycle.] Explosives (Method 8330)	-full list for explosives
	[NOTE: The following analysis will be performed at Day 0 and at end of cycle only if parameters are determined to be in excess of the cleanup goals (Table 1-2) at the initial excavation characterization samples] -SWMU-specific metals (6010) -TCLP-metals (1311, 6010, 7470) -SVOCs (Method 8270) -PCBs (Method 8081) -Dioxins (Method 8290)	 -per Section 1.4.2.1 -regulated list only -per Section 1.4.2.1 -per Section 1.4.2.1 -per Section 1.4.2.1

TABLE 1-11 (continued)
SUMMARY OF SAMPLING PARAMETERS PER TASK

Task	Analytical Method Requested	Parameters to be Reported
5. Sampling- Excavations (Soil) [NOTE: Parameters to be analyzed will be based on the chemicals of concern at a particular SWMU site per Section 1.4.2.1.]	-Site-specific VOCs (Method ~~8240~~8260) -TCLP VOCs (TCLP regulated list using Method ~~8240~~ 8260) (NOTE: one sample will be collected using EnCore samplers for VOCs analysis, covering both lists) -Explosives (Method 8330) -SWMU-specific metals (6010) -TCLP-metals (1311, 6010, 7470) -SVOCs (Method 8270) -PCBs (Method 8081) -Dioxins (Method 8290)	-per Section 1.4.2.1 -benzene, carbon tetrachloride, chloroform, chlorobenzene, 1,2-dichloroethane, 1,1-dichloroethene, methyl ethyl ketone, tetrachloroethene, trichloroethene, vinyl chloride -full list for explosives -per Section 1.4.2.1 -regulated list only -per Section 1.4.2.1 -per Section 1.4.2.1 -per Section 1.4.2.1

TABLE 1-12
PROCESS MONITORING MEASUREMENT AND SAMPLING SCHEDULE

PARAMETER	SAMPLING/ ANALYTICAL METHOD	*NUMBER OF SAMPLES PER SAMPLING EVENT	VOLUME REQUIRED PER SAMPLE	SAMPLING SCHEDULE
Oxygen	Field SOP QAPP-4.0	6 per cross-section (30 total)	in-situ measurement per Field SOP	Daily
Temperature	Field SOP QAPP-4.0	6 per cross-section (30 total)	in-situ measurement per Field SOP	Daily
Moisture Level	Field SOP QAPP-4.0	~~3~~1 per cross section (~~15~~5 total)	ex-situ measurement per Field SOP	Three times per week and after moisture added to any pile
pH	Field SOP QAPP-4.0	1 per cross section (5 total)	ex-situ measurement per Field SOP	Once per week
TNT	Field SOP QAPP-5.0	1 composite per cross section (from ~~per~~ location 1, 2 & 3) (~~35~~ total)	measurement per field SOP	Day 7, 14 & 21
RDX	Field SOP QAPP-5.0	1 composite per cross section (from ~~per~~ location 1, 2 & 3) (~~35~~ total)	measurement per field SOP	Day 7, 14 & 21

*The number of samples per event may be reduced as the windrow length is shortened due to volume reduction.

SECTION 2
PROJECT ORGANIZATION AND RESPONSIBILITY

2.1 PROJECT ORGANIZATION CHART

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

2.2 MANAGEMENT RESPONSIBILITIES

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

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

Southern Division Project Manager

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

- Define project objectives and develop a detailed work plan schedule;
- Establish project policy and procedures to address the specific needs of the project as a whole, as well as the objectives of each task;
- Acquire and apply technical and corporate resources as needed to ensure performance within budget and schedule constraints;
- Orient all field leaders and support staff concerning the project's special considerations;
- Monitor and direct the field leaders;
- Develop and meet ongoing project and/or task staffing requirements,

including mechanisms to review and evaluate each task product;

- Review the work performed on each task to ensure its quality, responsiveness, and timeliness;
- Review and analyze overall task performance with respect to planned requirements and authorizations;
- Approve all reports (deliverables) before their submission to U.S. EPA Region 5;
- Ultimately be responsible for the preparation and quality of interim and final reports; and
- Represent the project team at meetings and public hearings.

MK Program Management Office

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

2.3 QUALITY ASSURANCE RESPONSIBILITIES

MK Program QA Manager

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

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

- Reviewing analytical laboratory qualifications.

MK Site Quality Control Supervisor

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

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

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

- Conducting external Performance and System Audits of the off-site analytical Laboratory; and
- Reviewing and evaluating field and laboratory procedures.

2.4 LABORATORY RESPONSIBILITIES

MK Project Chemist

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

- Review of analytical laboratory qualifications as specified in Section 2.6;
- Coordinating laboratory analyses requirements with field personnel;
- Review of preliminary analytical data;
- Maintenance of final evidence files;
- Review of analytical reports;
- Approving final analytical reports prior to submission to Southern Division; and
- Overseeing data verification and data validation performed by MK Staff.

- Reviewing analytical laboratory qualifications.

MK Site Quality Control Supervisor

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

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

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

- Conducting external Performance and System Audits of the off-site analytical Laboratory; and
- Reviewing and evaluating field and laboratory procedures.

2.4 LABORATORY RESPONSIBILITIES

MK Project Chemist

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

- Review of analytical laboratory qualifications as specified in Section 2.6;
- Coordinating laboratory analyses requirements with field personnel;
- Review of preliminary analytical data;
- Maintenance of final evidence files;
- Review of analytical reports;
- Approving final analytical reports prior to submission to Southern Division; and
- Overseeing data verification and data validation.

Laboratory Project Manager

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

- Providing technical support for analytical method procedures and discrepancies;
- Ensuring all resources of the laboratory are available on an as-required basis; and
- Reviewing final analytical reports.

Laboratory Operations Manager

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

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

Laboratory Quality Assurance Officer

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

- Oversee laboratory quality assurance;
- Oversee QA/QC documentation;
- Conduct detailed review and verification of analytical data in reports prior to submission to MK;

- Determine whether to implement laboratory corrective actions, if required;
- Define appropriate laboratory QA procedures; and
- Prepare laboratory Standard Operation Procedures.

Laboratory Sample Custodian

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

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

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

Laboratory Technical Staff

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

2.5 FIELD RESPONSIBILITIES

Site Project Manager

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

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

Site QC Supervisor

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

Site Safety and Health Officer

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

Contract Administration

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

Field Engineer

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

Superintendent

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

2.6 LABORATORY QUALIFICATION REQUIREMENTS

2.6.1 Analytical Chemistry Laboratories

To support the analytical chemistry needs of the District II RAC program, MK will be responsible for the selection of qualified laboratories capable of performing quality analysis and data. Because the quality of analytical chemistry data must be defensible, only laboratories meeting certain minimum qualification requirements will be utilized. MK will utilize analytical laboratories that have previously performed or are currently performing analysis in support of the U.S. Navy's Installation Restoration Program (IRP) per NEESA 20.2-047B (NEESA, 1988). Laboratories will have undergone the laboratory approval process as defined in the subject NEESA document for the scope of work performed under the IRP. Because the NEESA approval process is project specific and not global, previous NEESA evaluations and approvals are not applicable to work performed on the District II RAC. However, they do provide some indication of the laboratory's experience working under the U.S. Navy's NEESA analytical requirements. Laboratories will also have undergone U.S. EPA Region 5 RCRA approval. Approval of this QAPP constitutes U.S. EPA Region 5 approval of the proposed laboratory and applicable SOPs for this project only.

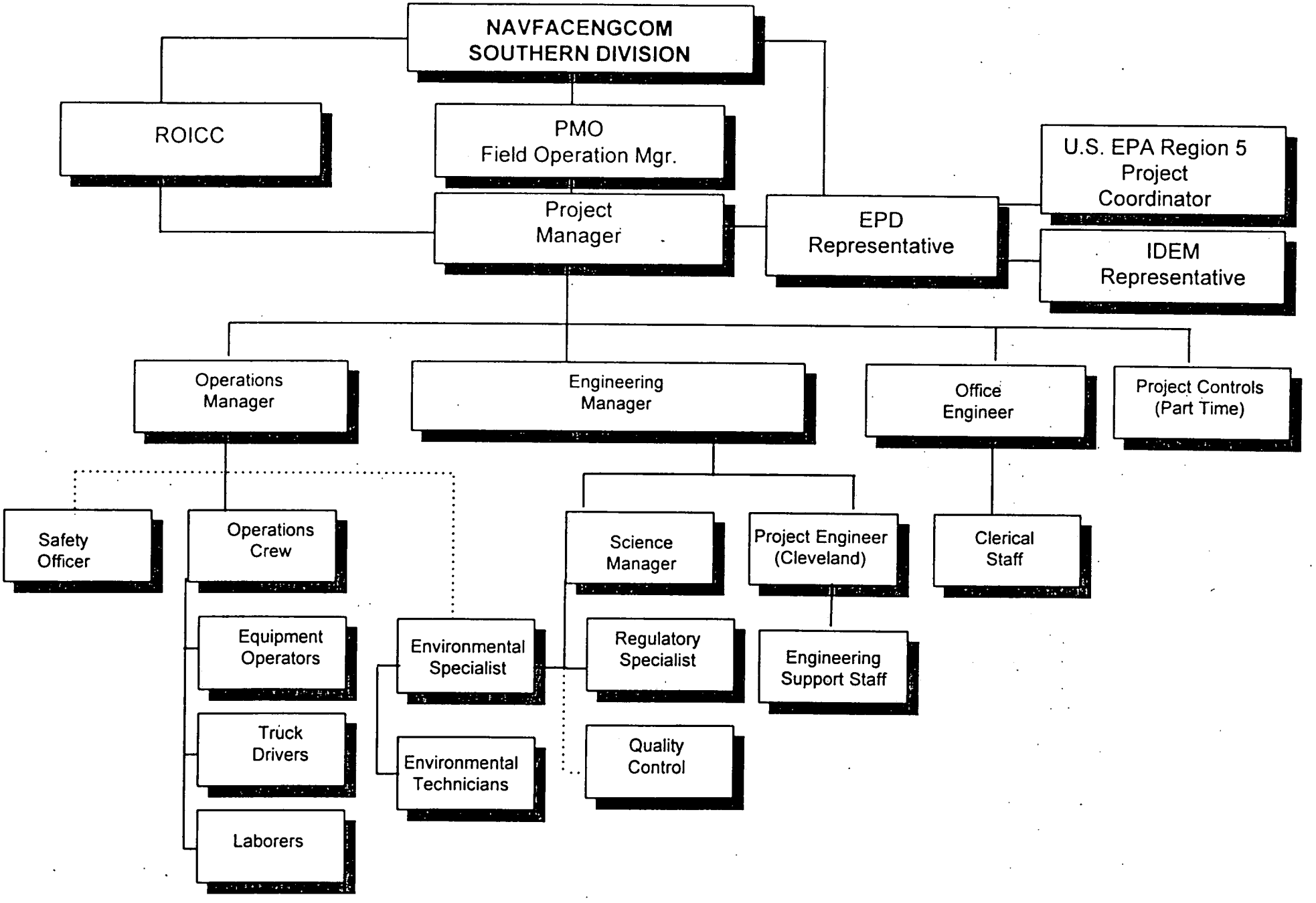
A QA systems audit of the laboratory may be scheduled to verify implementation of details of the laboratory's QAPP. The performance of an audit is based on the volume of work to be performed by the laboratory, and other factors, such as previous use of the laboratory. Any necessary corrective action identified as a result of an audit will be documented, and the laboratory required to respond to MK, stating the measures to be taken to correct the condition. The PQCM, as defined in Section 2.3, and the Project Chemist will work closely to ensure that any laboratory corrective action is adequate to provide the level of quality required on the District II RAC.

The QAPP provides the detailed requirements for analytical sampling, from collection in the field to analysis in the laboratory. Additionally, specific requirements for data deliverables are included in this QAPP.

2.6.2 Test Results

Laboratory test results will be signed by a testing laboratory ~~representative~~, project manager or designee authorized to sign certified test results. The laboratory will furnish all test results to the MK Project Chemist or designee. All test results will contain the following, as a minimum:

- A reference to applicable contract requirements, tests, or analytical procedures used;
- Actual test results; and
- A statement that the item tested or analyzed conforms or fails to conform to specified QC requirements.


NAVFACENGCOM
SOUTHERN DIVISION
ROICC
PMO
Field Operation Mgr.
Project
Manager
EPD
Representative
U.S. EPA Region 5
Project
Coordinator
IDEM
Representative
Operations
Manager
Engineering
Manager
Office
Engineer
Project Controls
(Part Time)
Safety
Officer
Operations
Crew
Equipment
Operators
Truck
Drivers
Laborers
Environmental
Specialist
Environmental
Technicians
Science
Manager
Regulatory
Specialist
Quality
Control
Project Engineer
(Cleveland)
Engineering
Support Staff
Clerical
Staff

SECTION 3
QUALITY ASSURANCE OBJECTIVES FOR MEASUREMENT DATA

The overall QA objective for this project is to develop and implement procedures for field sampling, chain-of-custody, laboratory analysis, and reporting that will provide results which are legally defensible in a court of law. Specific procedures for sampling, chain-of-custody, laboratory instrument calibration, laboratory analysis, reporting of data, internal quality control, audits, preventive maintenance of field equipment, and corrective action are described or referenced in other sections of this QAPP. Laboratory reporting and/or detection limits for each method in Tables 1-1 through 1-5 are provided in Appendix B. General quality control limits for accuracy, precision and completeness are provided in Tables 3-1 through ~~3-7~~3-8.

3.1 PRECISION

3.1.1 Definition

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

3.1.2 Field Precision Objectives

Field duplicate data will be evaluated to determine potential variability introduced by soil heterogeneity and sampling technique. This evaluation will be performed during data verification, as defined in Section 9.2.2, and summarized in the data assessment. One duplicate sample or reading will be analyzed for at least every 20 samples to qualitatively evaluate precision.

3.1.3 Laboratory Precision Objectives

Precision in the laboratory is assessed through the calculation of RPD and RSD for three or more replicate samples. The equations to be used for precision in this project can be found in Section 12. Precision control limits are established by SW-846 methods. Limits for confirmation soil sample precision are summarized in Tables 3-1 through 3-7.

3.2 ACCURACY

3.2.1 Definition

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

3.2.2 Field Accuracy Objectives

For laboratory data evaluation, field blanks, trip blanks and equipment rinsate blank data will be evaluated to identify potential sources of error introduced during sampling. This evaluation will be performed during data validation and summarized in the data assessment. Accuracy for laboratory samples is also assessed through adherence to all sample handling, preservation and holding times. Accuracy for field measurements will be ensured by adhering to established Field SOPs found in Appendix A of this QAPP.

3.2.3 Laboratory Accuracy Objectives

Laboratory accuracy is assessed through the analysis of standard matrix spikes, sample matrix spikes (MS) or standard reference materials (SRM) and the determination of percent recoveries. The equation to be used for accuracy in this project can be found in Section 12. The accuracy control limits are also summarized in Tables 3-1 through 3-7, and may vary based on laboratory performance.

3.3 COMPLETENESS

3.3.1 Definition

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

3.3.2 Laboratory Completeness Objectives

Laboratory completeness is a measure of the amount of valid measurements obtained from all measurements taken in the project. The equation for completeness is presented in Section 12. The laboratory completeness goal for Level C data is 90 percent, which is considered to be attainable using SW-846 and other EPA-approved

analytical methods.

3.3.3 Field Completeness Objectives

Field completeness for process monitoring data will be 85%. This goal reflects the ability to collect additional samples or measurements, if necessary, to complete the data set.

3.4 REPRESENTATIVENESS

3.4.1 Definition

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

3.4.2 Measures to Ensure Representativeness of Field Data

Representativeness is a qualitative parameter which is controlled by the proper design of the sampling program. Representativeness is addressed by describing sampling techniques and the rationale used to select sampling locations. It will be assessed through the use of samples collected in a manner such that they are equally representative of a given point in space or time. Representativeness will be satisfied by ensuring that this QAPP is followed and that proper sampling techniques and sample preservation procedures are followed as demonstrated in Section 4.0 of QAPP.

3.4.3 Measures to Ensure Representativeness of Laboratory Data

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

3.5 COMPARABILITY

3.5.1 Definition

Comparability is a qualitative parameter expressing the confidence with which one data set can be compared with another as in comparison of data from differing time intervals and to the Pilot-Scale Study.

3.5.2 Measures to Ensure Comparability of Field Data

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

3.5.3 Measures to Ensure Comparability of Laboratory Data

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

3.6 LEVEL OF QUALITY CONTROL EFFORT

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

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

One Field Blank sample will be collected from each source of tap water every month used for equipment decontamination.

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

MS/MSD samples are investigative samples. Soil MS/MSD samples ~~may require extra volume~~ for ~~VOCs or~~ extractable organics may require extra volume. However, ~~aqueous~~soil MS/MSD samples must be collected with two additional EnCore samplers to ensure sufficient volume for VOCs. Aqueous MS/MSD samples must be collected at triple the volume for VOCs and double the volume for extractable organics. One MS/MSD sample will be collected/designated for every 20 or fewer investigative

samples per sample matrix (*i.e.*, water, soil).

Field duplicates will be collected at a frequency of one sample per 20 ~~regular~~investigative samples collected, depending on the data use and as dictated in Table 3-1. The general level of the QC effort will be one field duplicate for every 20 or fewer investigative samples per sampling interval for compost sampling. One volatile organic analysis (VOA) trip blank, consisting of distilled/deionized water, will be included along with each shipment of aqueous VOA samples.

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

TABLE 3-1
QA OBJECTIVES FOR
PRECISION, ACCURACY AND COMPLETENESS
VOLATILE 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 = ~~0-30~~ 40 %	RPD = 0 - 35%
	Water	Laboratory Control Sample/Duplicate	One per 20 rinse events	RPD = ~~0-30~~ 40 %	RPD = 0 - 35%
	Soil, compost	Laboratory MS/MSD	One per 20 samples (one per analytical batch)	RPD = ~~0-40~~ 45 %	RPD = 0 - 35%
	Soil, compost	Laboratory Control Sample/Duplicate	One per 20 samples (one per analytical batch)	RPD = ~~0-40~~ 45 %	RPD = 0 - 35%
Accuracy	Water	Surrogate	All samples and standards	~~See Appendix G, Page G2-21~~Recovery = 86-118 %	Recovery = 75 - 121 %
	Water	Laboratory Control Sample/Duplicate	One per 20 samples (one per analytical batch)	~~See Appendix G, Page G2-17~~Recovery = 23-185 %	Recovery = 70 - 130 %
	Water	Laboratory MS/MSD	One per 20 samples (one per analytical batch)	~~See Appendix G, Page G2-17~~Recovery = 23-185 %	Recovery = 70 - 130 %
	Soil, Compost	Surrogate	All samples and standards	~~See Appendix G, Page G2-21~~Recovery = 74-120 %	Recovery = 75 - 120 %
	Soil, Compost	Laboratory Control Sample/Duplicate	One per 20 samples (one per analytical batch)	~~See Appendix G, Page G2-17~~Recovery = 50-151 %	Recovery = 68 - 130 %
	Soil, Compost	Laboratory MS/MSD	One per 20 samples (one per analytical batch)	~~See Appendix G, Page G2-17~~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 C, Page G2-222~~RPD = 0-65 %	RPD = 0 - 30%
	Water	Laboratory Control Sample/Duplicate	One per 20 samples (one per analytical batch)	~~See Appendix C, Page G2-222~~RPD = 0-65 %	RPD = 0 - 30%
	Soil, Compost	Laboratory MS/MSD	One per 20 samples (one per analytical batch)	~~See Appendix C, 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 C, Page G2-222~~RPD = 0-19 %	RPD = 0 - 40%
Accuracy	Water	Surrogate	All samples and standards	~~See Appendix C, Page G2-222~~Recovery = 10-141 %	Recovery = 10 - 141%
	Water	Laboratory Control Sample/Duplicate	One per 20 samples (one per analytical batch)	~~See Appendix C, Page G2-222~~Recovery = 10-135 %	Recovery = 10 - 150%
	Water	Laboratory MS/MSD	One per 20 samples (one per analytical batch)	~~See Appendix C, Page G2-222~~Recovery = 10-135 %	Recovery = 10 - 150%
	Soil, Compost	Surrogate	All samples and standards	~~See Appendix C, 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 C, Page G2-222~~Recovery = 34-115 %	Recovery = 10 - 150%
	Soil, Compost	Laboratory MS/MSD	One per 20 samples (one per analytical batch)	~~See Appendix C, 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 C, Page G2-169~~RPD = 0-80 %
	Soil-, Compost	Laboratory Control Sample/Duplicate	One per 20 samples (one per analytical batch)	~~See Appendix C, Page G2-169~~RPD = 0-80 %
Accuracy	Water	Laboratory Control Sample/Duplicate	One per 20 samples (one per analytical batch)	~~See Appendix C, Page G2-169~~Recovery = 24-140 %
	Soil, Compost	Laboratory Control Sample/Duplicate	One per 20 samples (one per analytical batch)	~~See Appendix C, 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 3-8
QA OBJECTIVES FOR
PRECISION, ACCURACY AND COMPLETENESS
EXPLOSIVES (Field Test Kit)

Data Quality Parameter	Matrix	Method of Determination	Frequency	QC Limits
Precision	Soil	Sample Duplicate	One per 20 samples	RPD = 0-45 %
	Soil	Field Check Standard/Duplicate	One per 20 samples	RPD = 0-45 %
Accuracy	Soil	Field Check Standard	One per 20 samples	Recovery - 77-122 %
Completeness	Soil	Valid Samples	All Samples	90 %

SECTION 4
SAMPLING PROCEDURES

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

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

These tasks and associated objectives are described in Section 1.4.

QA/QC samples are described in Section 4.6. Decontamination procedures are provided in Section 4.7. Sample packaging and shipping information is provided in Section 4.8.

Each of the sampling tasks listed above will be completed as applicable. Soils will be excavated and treated.

Confirmation sampling in the excavation(s) for the purpose of closure will be completed. The excavation will be sampled for SWMU-specific parameters listed in Section 1.4.2.1 prior to being-backfilled. This data will assist in delineating further excavation at the SWMUs.

4.1 INITIAL EXCAVATION SITE CHARACTERIZATION PROCEDURES

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

Objective 1: Identify areas for excavation. Analyze the soils data will also be used as "entry " concentrations for composting.

Objective 2: Determine if SWMU-specific ~~waste products~~chemicals are present in the soil.

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

constituents.

Note that in this section and in Field SOP QAPP- 1.0 in Appendix A of this QAPP, "VOCs analysis" indicates the soil will be tested for SWMU-specific VOCs and TCLP list VOCs. Only one sample (three EnCore samplers) will be collected for analysis of both sets of VOCs.

Sampling Areas

Grid layout and boundaries will be affected by the presence of buildings, utilities, etc. Grid borders will be flagged in the field and areas will be chosen which will be accessible by heavy equipment (i.e. individual grid blocks will not be placed in the midst of heavy utilities or surface structures). Grid blocks are not expected to be perfectly square (i.e. those in the berm). The Soil Excavation Plan, Appendix E of the Full-Scale Operations Plan (MK, 1998b) shows the layout of the grid blocks.

Soil Sampling

Soil samples will be collected from each grid block or sampling area using the procedure provided in Field SOP QAPP-1.0 in Appendix A, which is highlighted as follows:

For each grid block:

Volatile Organics Sampling

At a minimum, one sample for VOCs analysis will be collected from the center of the grid block using an EnCore sampler.

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

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

Grab samples for other SWMU-specific parameters will be held separately.

Sampling for other SWMU-Specific Parameters

Four grab samples will be collected from each corner of the grid block, as shown in the Soil Excavation Plan, Appendix E of the Full-Scale Operations Plan (MK, 1998b), for the 0 to 12-inch depth and 2- to 3-foot depth.

For each depth:

The four grab samples plus the one from the center of the grid block will be composited for the required analyses (Table 1-11). The grabs will be combined to form one composite sample as follows:

> Place each grab sample into ~~an aluminum pie pan or cookie sheet, after removing~~a stainless steel bowl and remove vegetation and ~~coarse fractions~~large rocks. ~~Break~~Homogenize samples by breaking up the soil using ~~gloved hands, and remove large rocks~~a pestle or spoon. ~~(the soil may also be sieved)~~. ~~Allow~~Thoroughly mix and homogenize the soil ~~to air dry~~in the bowl. ~~Cover the pie pan with a second pie pan and shake and swirl vigorously to disperse and homogenize the soil.~~ Cone and quarter the soil in the ~~pie pan~~bowl. Collect an equal portion from each quarter and composite to fill an 8-ounce jar for explosives analysis.
>
> This process will provide a more representative sample as explosives contamination is known to vary over short distances (Crockett and others, 1996). Combine the remaining soil together for other analysis

As a result, for each grid block, a minimum of two composite samples will be collected for SWMU-specific compounds. A minimum of one sample will be collected for VOC analysis from the center of the grid block, from a depth of 12 to 18 inches.

Marking Grid Blocks for Excavation

Analytical results will be used to mark grid blocks or sampling areas as requiring excavation. If a grid block or sampling area shows contamination (off-site laboratory results TNT or RDX shows greater than cleanup goals in Table 1-2 of the Full-Scale Operations Plan) to a depth of three feet, the grid block or area will be marked for upto three feet of excavation. If a grid block or area shows contamination at the one-foot depth, the block or area will be marked for one foot of excavation. If a grid block shows no contamination at the one-foot and the three-foot depth, the grid block shall not be marked for excavation. Additional grid blocks will be sampled as necessary to allow excavation of the required volume of soil.

4.2 EQUIPMENT WASH WATER/DECONTAMINATION WATER SAMPLING

Decontamination water generated at the excavation staging area and at the Biofacility will be collected and stored in a tank. This water will be used for windrow moisture addition. Any solids which collect in the sumps at the Biofacility will be added to the compost during the initial windrow mixing only.

Decontamination water stored in a tank will not be sampled if used in the treatment process~~,~~ or ~~will be sampled for explosives, RCRA metals and POTW requirements if~~ shipped to an on-site treatment facility. However, it will be sampled for parameters listed in Table 1-5 if discharged to the POTW system. Sampling procedures are provided in Field SOP QAPP- 2.0 in Appendix A.

The samples will be labeled and stored in a cooler with ice and kept at approximately 4 ± 2 degrees Celsius. Sample documentation, packaging, and shipping procedures will be followed as described in Section 4.8 and Section 5.0, Custody Procedures.

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

4.3 STORM-WATER RETENTION POND SAMPLING

Surface run-off or storm-water present on the Biofacility asphalt-paved areas is collected in two ponds, #1 and #2. The water in the retention ponds will be sampled to determine if NPDES discharge requirements are met. Sampling procedures are provided in Field SOP QAPP- 2.0 in Appendix A. Samples will be tested for NPDES parameters listed in Table 4-1.

TABLE 4-1
NPDES DISCHARGE LIMITATIONS[1]

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

TABLE 4-1 (continued)
NPDES DISCHARGE LIMITATIONS[1]

Notes:

1. These values were obtained from the NPDES permit no. IN 0021539 (highest levels permitted).
2. Field pH will be measured using a Horbia Twin B-Z13 or a Hach Model EC10 pH meter (290). Calibration data and model information will be recorded in the field logbook, including results.

N/A = not available

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

A NSWC Crane representative may perform the storm/run-off water sampling in lieu of the above, based on the approved IDEM NPDES requirements.

4.4 PROCESS SAMPLING

The composting process is anticipated to take approximately 28 days as dictated in the Full-Scale Operational Plan (MK, 1998b). Each windrow will be approximately 250 feet long, 7 feet high and 20 feet wide. Sampling locations are displayed in Figure 4-3. Cross-section at column numbers 3, 5, 7, 9, and 11 will be sampled throughout the process for various parameters to be analyzed by an off-site laboratory. The schedule for sampling is shown in Table 4-2.

Once Field Screening results have shown that explosive concentrations of TNT and RDX have been reduced below cleanup levels, a confirmation sample will be collected. Confirmation samples are anticipated to be collected on Day 28 of processing. However, should processing be extended or shortened, confirmation samples will be collected at the end of the compost cycle. Confirmation samples will be collected using procedures in Field SOP QAPP- 3.0 in Appendix A.

TABLE 4-2
WINDROW/COMPOST SAMPLING SCHEDULE

Parameter	Analytical Method	Number of Samples per Sampling Event	Volume Required per sample	Schedule
Explosives field screening kits	SOP QAPP-5.0 (SW 846-8510 & 8515)	1 composite per cross-section of samples from locations 1, 2, & 3	20 grams per analysis	Days 7, 14, & 21
Explosives (full list)	8330	3 per cross-section (15 total)	8 ounces	Day 0 and at end of cycle
The following analysis will be performed at Day 0 and at end of cycle only if parameters are determined to be in excess of the cleanup goals (Table 1-2) at the initial excavation characterization samples				
TCLP-metals	1311, 6010, 7470	3 per cross-section (15 total)	8 ounces (Both analysis may be done using 8 ounces)	Day 0 and at end of cycle
Metals	6010	3 per cross-section (15 total)		Day 0 and at end of cycle
SVOCs	8270	3 per cross-section (15 total)	8 ounces	Day 0 and at end of cycle
PCBs	8081	3 per cross-section (15 total)	8 ounces	Day 0 and at end of cycle
Dioxins	8290	3 per cross-section (15 total)	8 ounces	Day 0 and at end of cycle

4.4.1 Other Process Parameters

Field testing equipment will consist of, oxygen and temperature direct reading probes. Oxygen and temperature will be measured by pushing the probes into the center of each sampling sector of the compost piles and allowing the monitor to stabilize. Compost pH will be determined by measuring the pH level in a solution of deionized water and a compost sample. Soil moisture will be measured by weighing the sample, drying it, reweighing it and calculating the percent moisture. Frequency of pH, oxygen, temperature and soil moisture measurements are described in Section 7.0 of the Full-Scale Operational Plan (MK, 1998b) and in the Field SOP QAPP- 4.0 in Appendix A of this QAPP.

4.4.2 Air Monitoring

Air monitoring will be performed as described in the Site Safety and Health Plan, Appendix A of the Full-Scale Operational Plan (MK, 1998b).

4.5 EXCAVATION SAMPLING PROCEDURE

4.5.1 In-process Excavation Sampling

Field test kits for explosives will be used to help delineate the horizontal and vertical extent of contamination during excavation activities, followed by confirmation sampling and laboratory analysis. After the initial excavation activities are complete in each grid, the horizontal and vertical extent of remaining contaminants will be determined by using colorometric field test kits for TNT and RDX. A step by step procedure for field screening is provided in Field SOP QAPP- 5.0 in Appendix A.

A sampling grid will be laid out along the base of the excavation(s), using a nodal spacing specified in Appendix E of the Full-Scale Operational Plan (MK, 1998b). Each grid block will be sampled using Field SOP QAPP- 1.0 in Appendix A. Discrete samples will be collected from the four corners and center of the grid block, from surface to six inches below ground surface within the excavation, using ~~a drive sampler~~an auger, composited, and analyzed for TNT and RDX per Field SOP QAPP-5.0 in Appendix A.

Pin flags will be placed every 20 feet along sidewalls greater than one foot in depth. Samples will be collected at each pin flag location, using a drive sampler. One sample will be collected from 0-6 inches into the sidewall surface at the midpoint between the surface and bottom of excavation. The sample will be analyzed for TNT and RDX (field test kits).

4.5.2 Post-excavation Soil Sampling

After the soil is excavated to a depth where field screening indicate acceptable concentrations of explosives, post-excavation soil samples will be collected for analysis by a laboratory for all constituents as shown in Table 1-1.

Once soil has been removed, the excavation will be sampled and covered. After analytical results are obtained and determined to meet the cleanup levels, the excavation will be backfilled as described in the Full-Scale Operations Plan (MK, 1998b).

The purpose of sampling the excavation is to determine the level of contamination remaining, with respect to explosives and SWMU-specific contaminants. A sampling

grid will be laid out along the base of the excavation(s), using a nodal spacing specified in Appendix E of the Full-Scale Operational Plan (MK, 1998b). Each grid block will be sampled using procedures described in the QAPP for pre-excavation sampling. However, only one composite sample and one grab sample (for VOCs) will be collected from each grid block. The composite sample will be analyzed for SWMU-specific parameters (Table 1-11), and will be collected from the surface to six-inch interval in a manner similar to pre-excavation composite samples. The grab sample will be analyzed for site-specific VOCs, and will be collected from the center of the grid block, ~~120~~ to ~~186~~ inches below excavated ground surface, using ~~a drive sampler~~three EnCore samplers.

Pin flags will be placed every 20 feet along sidewalls greater than one foot in depth. Samples will be collected at each pin flag location, using a drive sampler. One sample will be collected from 0-6 inches into the sidewall surface at the midpoint between the surface and bottom of excavation. The sample will be analyzed for SWMU-specific parameters (Table 1-11).

4.6 QC SAMPLES

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

4.6.1 Trip Blanks

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

4.6.2 Equipment Rinsate Blanks

Equipment rinsate blanks (or rinsate blanks) are collected to ensure that sampling equipment is clean and that the potential for cross-contamination has been minimized or prevented. Rinsate blanks are collected from the final reagent grade water rinse used in sample equipment decontamination. Rinsate blanks will be collected and analyzed at a frequency of one per 20 rinses, or at a frequency listed with the sampling procedures, whichever is more stringent. A reduction in the frequency of rinsate blanks may be deemed justifiable by the Project Chemist based on review of analytical results which would need to show no discernable evidence of introduction of cross-contamination due to inadequate equipment cleansing with respect to analysis type.

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

4.6.3 Field Blanks

Field blanks consist of the source water used in decontamination and steam cleaning. At a minimum, one field blank from each event and each source of water must be collected and analyzed for the same parameters as the regular samples (NEESA, 1988).

NEESA defines an event as the time period a sample crew is on site. Although the crew is onsite continuously, routine VOA analysis is advisable. As a result, samples of decontamination water used, including the potable water used during treatment, will be collected at a frequency of 1 per source per month, as shown in Table 4-3.

4.6.4 Field Duplicate Samples

Field duplicate samples will be collected to allow determination of analytical and sampling precision. One duplicate sample per 20 samples will be collected and submitted for the same analysis as the regular samples. However, one duplicate per 10 samples for explosives will be collected and submitted for analysis. The duplicate sample will be collected simultaneously with the true sample. The sample will be collected with the same sampling equipment at the same location as the regular sample, but it will be labeled with the next consecutive sample number.

TABLE 4-3
SUMMARY OF SAMPLING AND ANALYSIS PROGRAM

Task	Matrix	Parameters	Frequency		QA/QC Samples			
			RS	FD	FB	RB	TB	MS/MSD
1. Initial Excavation Site Characterization	Soil	SWMU-specific parameters per Section 1.4.2.1	Two composites per grid block and one grab for VOCs	1 per 20 samples (1 per 10 for explosives)	1 per month	1 per 20 rinses	1 per VOA Sample Cooler	1 per 20 samples
2. Equipment Wash Water/ Decon Water; Building Sump Water [NOTE: No testing is required if disposal to one of the on-site waste treatment facilities or used for compost moisture addition]	Water	Per Table 1-5	1 per 20 decon or tank	1 per 20 samples	1 per month	0	1 per VOA Sample Cooler	1 per 20 samples
3. Storm-Water Retention Pond Sampling	Water	NPDES parameters (listed in Table 4-1)	1 per rain event	1 per 20 samples	1 per month	0	0	1 per 20 samples
4a. Compost (Process) Sampling (includes confirmation sampling)	Compost	Explosives (Day 0 and at end of cycle)	One per location per cross-section (15 per pile per event)	1 per ~~pile per event~~10 samples	1 per month	1 per 20 rinses	0	1 per 20 samples

TABLE 4-3
SUMMARY OF SAMPLING AND ANALYSIS PROGRAM

Task	Matrix	Parameters	Frequency		QA/QC Samples			
			RS	FD	FB	RB	TB	MS/MSD
4b. Compost (Process) Sampling (includes confirmation sampling)	Compost	Other SWMU-specific parameters per Section 1.4.2.1 only if found in the characterization samples (Day 0 and at end of cycle)	One per location per cross-section (15 per pile per event)	1 per pile per event (1 per 10 for explosives	1 per month	1 per 20 rinses	1 per VOA Sample Cooler	1 per 20 samples
5. Excavation Soil Sampling	Soil	SWMU-specific parameters per Section 1.4.2.1	Base: One composite per grid block and one grab for VOCs Sidewall: One composite per grid block and one grab for VOCs	1 per 20 samples (1 per 10 for explosives	1 per month	1 per 20 rinses	1 per VOA Sample Cooler	1 per 20 samples

Notes:
FB = field blank
RB = rinsate blank or equipment rinsate blank
TB = trip blank (will be prepared by laboratory and tested for VOCs only)
MS/MSD = matrix spike and matrix spike duplicate (collected at a frequency of 1 per 20 samples collected)
RS = regular sample
FD = field duplicate

A cooler temperature blank consisting of tap water will be shipped with each cooler containing samples that require temperature preservation

4.6.5 Matrix Spike Samples

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

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

4.7 DECONTAMINATION PROCEDURES

4.7.1 Sample Bottles

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

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

> *Metals*: Detergent wash, tap water rinse, 1:1 HNO_3 rinse, tap water rinse, 1:1 HCL rinse, final reagent grade water rinse.
>
> *Volatiles* (40 ml glass vials and septa): Non-phosphate detergent wash, tap water rinse, reagent grade water wash, dry (glassware only) in a muffle furnace at 105 degrees Celsius for one hour.
>
> *Explosives:* Non-phosphate detergent wash, tap water rinse, deionized water rinse, methanol rinse, final reagent grade water rinse.

4.7.2 Sample Equipment Decontamination

Decontamination of construction and field equipment will take place in a designated area using procedures described in Section 7.0 of the Full-Scale Operational Plan (MK,

1998b). All non-disposable sampling equipment, including stainless steel trowels and spoons, will be decontaminated according to the following procedure and sequence, depending on the contaminants involved:

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

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

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

4.8 SAMPLE PACKAGING AND SHIPPING

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

Packaging and shipping procedures will vary depending upon sample media, potential contaminant concentration, preservation technique, and sample container. General procedures are provided in Field SOP QAPP- 6.0 in Appendix A. A waterproof ice chest or cooler is suitable for packaging and shipping samples not considered "Dangerous Goods" by the U.S. Department of Transportation (USDOT). The person packaging the samples is responsible for ensuring that the cooler is in suitable condition for shipping. In the event a concern arises regarding whether a sample shipment is a USDOT "Dangerous Good" or is potentially in violation of international transportation regulations, the Project Manager will be consulted. Sample packaging should ensure that the chance of breakage is minimized and that, in the event a sample container breaks or leaks, it will not impact the integrity of other samples in the shipment. Also, samples should be protected from ice melt. Each sample container should be wrapped in packing material such as bubble wrap or comparable shock-absorbing materials when shipping glass containers to avoid breakage.

TABLE 4-4
ANALYTICAL METHOD REQUIREMENTS

Analytes	Analytical Method[1]	Sample Container[2]	Preservation	Holding Time
Initial Excavation Site Characterization Analyses (soil) , Compost Solids, Soil in Excavation				
SWMU-specific VOCs per Section 1.4.2.1	8260	~~1 4-ounce glass~~3 EnCore samplers and one 2-ounce jar ~~with Teflon-lined lid or brass sleeve (pack tightly!)~~ ~~———~~for moisture determination	Ice to 4°C	14 days
VOCs (TCLP list)	~~1311/8260~~8260			
Explosives	8330 (full list)	1 8-ounce glass jar with Teflon-lined lid or brass sleeve	Ice to 4°C	14 days for extraction; analyze within 40 days of extraction
SWMU-specific total metals per Section 1.4.2.1	6010	1 8-ounce glass jar with Teflon-lined lid or brass sleeve	Ice to 4°C	180 days
TCLP metals	~~1311,~~ 6010, 7470	1 8-ounce glass jar with Teflon-lined lid or brass sleeve	Ice to 4°C	mercury; field collection to extraction 28 days; from extraction to analysis 28 days; other metals: 180 days from extraction to analysis
SWMU-specific SVOCs per Section 1.4.2.1	8270	1 8-ounce glass jar	Ice to 4°C	14 days for extraction; analyze within 40 days of extraction
SWMU-specific PCBs per Section 1.4.2.1	~~8081~~8082	1 8-ounce glass jar	Ice to 4°C	14 days for extraction; analyze within 40 days of extraction

TABLE 4-4
ANALYTICAL METHOD REQUIREMENTS

Analytes	Analytical Method[1]	Sample Container[2]	Preservation	Holding Time
Dioxins	8290	1 8-ounce glass jar	Ice to 4°C	14 days for extraction; analyze within 40 days of extraction

TABLE 4-4
ANALYTICAL METHOD REQUIREMENTS

Analytes	Analytical Method[1]	Sample Container[2]	Preservation	Holding Time
Water Analyses (decontamination water, storm-water retention pond)				
Explosives	8330 (full list)	2 1-L amber glass jars	Ice to 4°C	7 days; analyzed within 40 days after extraction
total suspended solids (TSS)	EPA 160.2	1 500-mL plastic	Ice to 4°C	7 days
chemical oxygen demand (COD)	EPA 410.1, 410.2	1 250-mL plastic	H_2SO_4 to pH < 2, Ice to 4°C	28 days
biochemical oxygen demand (5-day BOD)	EPA 405.1	1 1-L amber glass jar	Ice to 4°C	**48 hours**
Oil and Grease	EPA 413.1	2 1-L amber glass jars	HCL to pH < 2; Ice to 4°C	28 days
Nitrate	EPA 300.0	1 500-mL plastic	Ice to 4°C	**48 hours**
Ammonia-N	EPA 350.2	1 500-mL plastic	H_2SO_4 to pH < 2, Ice to 4°C	28 days
Additional Water Analyses (blanks)				
Volatile Organics	8260	3 40-mL glass vials	HCL to pH < 2; Ice to 4°C	14 days
Explosives	8330 (full list)	2 1-L amber glass jars	Ice to 4°C	7 days; analyze within 40 days after extraction
RCRA metals	6010	1 500-mL plastic	HNO_3 to pH < 2; Ice to 4°C	180 days

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

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

Adequate ice, contained in double Ziploc™-type bags or equivalent, must be included with each cooler shipment so that the contents are maintained at 4 ± 2 degrees Celsius until receipt at the laboratory. A temperature blank will be shipped with each cooler to verify the temperature.

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

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

4.9 PROCESS MEASUREMENTS

Process monitoring field measurements and samples will be collected from the sampling locations shown in Figure 1-1 in Field SOP QAPP- 4.0 in Appendix A. Sample locations other than those that are pre-designated will be clearly identified in the field logbook. A summary of the process monitoring program is provided in Table 4-5.

TABLE 4-5
SUMMARY OF PROCESS MONITORING PROGRAM

TASK	PARAMETERS	SAMPLING FREQUENCY	FIELD DUPLICATES
Oxygen level monitoring	oxygen	6 per cross-section (30 per event)	at least one per event
Temperature Monitoring	temperature	6 per cross-section (30 per event)	at least one per event
Moisture Level Monitoring	moisture	1 per cross section (5 per event)	at least one per two events
pH Level Monitoring	pH	1 per cross section (5 per event)	at least one per 5 events

Field measurements will be collected only with kits, instruments, tools, gauges, or other measuring devices that are controlled, calibrated, adjusted and maintained in accordance with the maintenance and calibration requirements described in Field SOP QAPP- 4.0 in Appendix A.

4.9.1 QA/QC Measurements and Samples

QA/QC measurements and samples will be collected to ensure that representative and reproducible data are obtained. QA/QC measurements associated with process monitoring field measurements are described below.

Field duplicate data will be evaluated to determine potential variability introduced by sampling or measurement techniques. The precision of field duplicate measurements will be determined by calculating the relative percent difference (RPD) between the original and the duplicate measurement. The calculation for RPD is provided in Section 12.0 of this QAPP.

A second duplicate reading for temperature shall be taken if the initial reading and the first duplicate reading differ by more than 5°C. A second duplicate reading for oxygen will be taken if the initial reading or first duplicate reading is less than 10% and the readings differ by 4 or more percent oxygen. The second duplicate readings shall be recorded in the comments section of the Windrow Process Data - Daily Log in Field SOP QAPP- 4.0 in Appendix A.

If the second duplicate has percent differences which vary from both of the other two measurements, the measurement device will be recalibrated per the manufacturer's guidelines, and the procedure will be reviewed to ensure compliance with established techniques

4.9.2 Decontamination Procedures

Procedures for decontamination of large equipment, sampling equipment, and personnel are described in Section 7.0 of the Full-Scale Operational Plan (MK, 1998b).

4.9.3 Field Method Audits

Field methods may be audited at any time during the Full-Scale Operations by the U.S. Navy and the U.S. EPA.

SECTION 5
CUSTODY PROCEDURES

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

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

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

5.1 FIELD CUSTODY PROCEDURES

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

Logbooks will be bound field survey books or notebooks. Logbooks will be assigned to field personnel, but will be stored in the document control center when not in use. Each logbook will be identified by the type of activity (i.e., operations, safety, and environmental).

The title page of each logbook will contain the following:

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

Entries into the logbook will contain a variety of information. At the beginning of each entry, the date, start time, weather, names of all sampling team members present, level of personal protection being used, and the signature of the person making the entry will

be entered.

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

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

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

5.1.1 Field Documentation

Logbooks and forms will be used to document all field activities. Information to be entered in the field logbook includes at a minimum:

<u>Operations Logbook:</u>

- Individuals on site
- Operational activities/impacts
- Production information
- Visitors

<u>Safety Logbook:</u>

- Weather conditions
- PPE
- Injuries/incidents/accidents
- Results of air monitoring and personal protective equipment levels

<u>Environmental Logbook:</u>

- Sample identification

- Date and time of sample collection
- Requested analyses, type and number of sample containers, and preservatives
- Results of field screening measurements
- Sample locations and depth (include sketches as necessary)
- Type of sample collected (pond water, decontamination water, soil, grab, etc.)
- Details of QA/QC samples

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

5.1.2 Sample Identification

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

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

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

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

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

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

5.1.2.1 Excavation Site Sample Identification

The sample identification number for excavation site samples consists of up to four sets of characters and/or letters. The first set of three characters represent the excavation site; the second set of three characters represent the excavation site sample type; the next three numbers represent unique sample number; and the last two characters (FD) will be used to designate field duplicate of the field sample. Document all field duplicates with their associated samples and locations in the field logbook. NOTE: The designation FD will be used only when a field duplicate is collected. A generic sample name would be:

XXX-XXX-NNN-FD

Excavation Site

- MFA - Mine Fill A
- MFB - Mine Fill B
- RKY - Rockeye
- ABG - Ammunition Burning Ground

Excavation Site Sample Type

- ICS - Initial Characterization Soil (sample) (pre-excavation - Task 1)
- IES - In-process Excavation Soil (sample) (Task 5)
- PES - Post-Excavation Soil (sample) (Task 6)

QC Sample Identifiers (Excavation Site Samples)

The following identification system will be used for numbering QC samples associated with excavation sites:

XXX-XXX-XX-NNNNNN

The first set of three characters represent the excavation site; the second set of three characters represent the excavation site sample type; the next two characters represent field QC designation (TB - Trip Blank; FB - Field Blank; RB - Rinsate Blank); and the last six numbers represent the month, date, and year of QC sample (e.g., 021598 represents February 15, 1998). Document all QC samples with their associated samples in the field logbook.

5.1.2.2 Non-Compost Sample Identification

The sample identification number for non-compost samples consists of up to three sets of characters and/or letters. The first set of three characters represent the type of sample; the second set of three numbers represent unique sample number; and the last two characters (FD) will be used to designate field duplicate of the field sample.

Document all field duplicates with their associated samples and locations in the field logbook. NOTE: The designation FD will be used only when a field duplicate is collected. A generic sample name would be:

XXX-NNN-FD

Non-Compost Samples Type

Samples collected will consist of one of the following types:

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

QC Sample Identifiers (Non-Compost Samples)

The following identification system will be used for numbering QC samples associated with non-compost samples:

XXX-XX-NNNNNN

The first set of three characters represent the non-compost sample type; the next two characters represent field QC designation (TB - Trip Blank; FB - Field Blank; RB - Rinsate Blank); and the last six numbers represent the month, date, and year of QC sample (e.g., 021598 represents February 15, 1998). ~~Document all QC samples with their associated samples in the field logbook.~~

~~5.1.2.2 Biofacility (Compost) Sample Identification~~

~~The sample identification number~~Samples collected for ~~Biofacility (Compost) samples consists of up to seven sets of characters and/or letters~~MS/MSD purposes will contain the same sample identifier as the corresponding sample. Document all QC samples with their associated samples in the field logbook.

5.1.2.3 Biofacility (Compost) Sample Identification

The sample identification number for Biofacility (Compost) samples consists of up to

seven sets of characters and/or letters. The first set of three characters ("BIO") represent that the sample is collected from Biofacility; the second set of one character (N, S, M) represents the building designation - north, south or middle building respectively; the next three numbers represent unique compost pile number within the building; the next two numbers will be used to designate the day on which the sample was collected (i.e, 00, 07, 14, 21, 28, etc.); the next two numbers will be used to designate the column number representing the cross-sections (i.e., 03, 05, 07, 09, and 11); the next number represents the location (i.e., 1, 2, or 3) within the cross-section; and the last two characters (FD) will be used to designate field duplicate of the field sample. Document all field duplicates with their associated samples and locations in the field logbook. NOTE: The designation FD will be used only when a field duplicate is collected. A generic sample name would be:

BIO-X-NNN-NN-NN-N-FD

QC Sample Identifiers (Compost Samples)

The following identification system will be used for numbering QC samples associated with compost samples:

BIO-XX-NNNNNN

The first set of three characters ("BIO") represent that the sample is collected from the Biofacility; the next two characters represent field QC designation (TB - Trip Blank; FB - Field Blank; RB - Rinsate Blank); and the last six numbers represent the month, date, and year of QC sample (e.g., 021598 represents February 15, 1998). Document all QC samples with their associated samples in the field logbook.

5.1.3 Chain-of-Custody Procedures

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

The subcontracted laboratories will not accept samples collected by MK for analysis without a completed COC form. The COC form shall be initiated in the field by the person collecting the sample, for each sample. Each COC form will be assigned a unique number based on the SWMU designation (i.e., samples collected from Mine Fill A will start with MFA-001; similarly samples collected from Biofacility operations will start with BIO-001). If sampling is conducted at the Biofacility and Mine Fill A, two COC forms, each with a unique COC number, will be used: one for Mine Fill A and one for the Biofacility. Each sample will be assigned a unique identification number, as

described in Section 5.1.2, and that number will be entered on the COC form. In order to be complete, project identification, date and time of sample collection, sample location, requested analyses, turnaround time, and any special instructions must be included on the COC, along with each sample identification. Custody will be relinquished by using the signature blocks at the bottom of the custody form. The original COC form will accompany the shipment, and a copy will be retained by the PM or designee. A signed COC form will be obtained from the laboratory custodian after the samples have been received and their condition checked.

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

5.1.4 Handling and Shipping

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

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

5.2 LABORATORY CUSTODY PROCEDURES

Laboratory custody procedures for sample receiving and log-in; sample storage and

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

5.3 FINAL EVIDENCE FILES

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

The final evidence file will include at a minimum:

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

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

The U.S. Navy will take possession of final evidence files at the conclusion of the Contract or as required by the U.S. Navy. The Navy, or designated representatiave, will maintain the files for a period of ten years following the termination of the Project. Afterward, prior to disposal, the U.S. Navy, or designated representatiave, will provide written notification to the U.S. EPA

SECTION 6
CALIBRATION PROCEDURES AND FREQUENCY

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

6.1 FIELD INSTRUMENT CALIBRATION

Field instruments will be calibrated in accordance with manufacturer's instructions and Field SOP QAPP- 4.0 in Appendix A. Instruments, including the PID, pH meter and temperature/oxygen probe will be calibrated daily, prior to use. Calibration data will be documented in the logbook or on the appropriate forms provided in the Field SOPs. Operation and calibration of the PID instrument is discussed in the Site Safety and Health Plan, Appendix A of the Full-Scale Operational Plan (MK, 1998b).

6.2 LABORATORY INSTRUMENT CALIBRATION

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

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

Calibration information is provided in the laboratory SOPs included in Appendix C and summarized in Table 6-1.

TABLE 6-1
CALIBRATION CRITERIA - LABORATORY METHODS

Parameter	SOP Number	Section Number (in SOP)	Page Number Appendix C-
VOCs	SWL-OV-303	Section 8.0	C2-8
Explosives	SWL-OL-200	Section 7.4	C2-38
Metals	SWL-IN-205	Section 7.4	C2-87
Mercury	SWL-IN-207	Section 8.5	C2-93
Total Cyanide	SWL-IN-	Section IV	C2-347
SVOCs	SWL-OS-500	Section 10.7	C2-216
PCBs	SWL-OP-412	Section 8.4	C2-252
Dioxins	SWL-OD-205	Section 8.8	C2-160
Oil and grease	SWL-OG-105	Section 7.2	C2-332
Nitrates	SWL-IN-030B	Section 8.0	C2-324
TSS, COD, BOD, ammonia-N	(none required)		

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)			

SECTION 7
ANALYTICAL PROCEDURES

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

7.1 FIELD ANALYTICAL PROCEDURES

Operation and QC procedures for the scale, pH meter and temperature/oxygen probes are provided in Appendix A of this QAPP. The operation and QC procedure for the PID is discussed in the Site Safety and Health Plan, Appendix A of the Full-Scale Operational Plan (MK, 1998b). Operation and QC procedures for the EnSys test kits are discussed in Appendix A, Section 5.0 of this QAPP.

7.2 LABORATORY ANALYTICAL PROCEDURES

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

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

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

7.2.1 List of Project Target Compounds and Laboratory Reporting Limits

A listing of project target compounds, cleanup levels, analytical methods, and analytical reporting limits to be used for samples collected during the project are summarized in Tables 1-1, 1-2, 1-3, 1-4a, and 1-4b. Other analytical results will be reported using standard method PQLs as reporting limits. Standard method detection limits for each of these methods are provided in Appendix B.

7.2.2 List of Associated QC Samples

The analytical methods and laboratory SOPs include a QC section which address the minimum QC requirements for the analysis of specific analyte groups. Compounds to

be spiked will be representative of the target compounds of interest as specified in Section 1.4.2.1. Section 7.1 of the Sampling and Chemical Analysis Quality Assurance Requirements for the Navy Installation Restoration Program (NEESA, 1988) contains a complete listing of the associated QC samples required for this project. This document is provided as Appendix E.

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	~~8081~~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)

SECTION 8
INTERNAL QUALITY CONTROL CHECKS

8.1 FIELD QUALITY CONTROL CHECKS

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

8.2 LABORATORY QUALITY CONTROL CHECKS

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

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

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

SECTION 9
DATA REDUCTION, VALIDATION AND REPORTING

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

9.1 DATA REDUCTION

9.1.1 Field Data Reduction Procedures

Data from direct read instrumentation will be recorded on the daily log immediately after measurements are taken. If errors are made, results will be legibly crossed out, initialed and dated by the field sampler, and corrected in a space adjacent to the original (erroneous) entry.

The precision of field duplicate measurements will be determined by calculating the relative percent difference (RPD) between the original and the duplicate measurement. The calculation for RPD is provided in Section 12.0 of this QAPP.

9.1.2 Laboratory Data Reduction Procedures

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

For this project, the equations that will be employed in reducing data are presented in Section 12. Such formulas make pertinent allowances for matrix type. All calculations will be checked by the Organic Section supervisor at the conclusion of each operating day. Errors will be noted, corrections made, with the original notations crossed out legibly. Analytical results for soil samples shall be calculated and reported on a dry weight basis, and TCLP results will not be matrix spike recovery-corrected.

Quality control data (e.g. laboratory duplicates, surrogates, matrix spikes, and matrix

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

9.2 DATA VALIDATION

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

9.2.1 Procedures Used to Evaluate Field Data

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

9.2.2 Procedures to Validate Laboratory Data

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

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

9.3 DATA REPORTING

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

9.3.1 Field Data Reporting

Field measurement data will be tabulated and submitted as required. The reports will provide summary tables and graphs of the field measurement data. All field measurement data sheets and logbooks will be retained. Raw data will be identified and included in a separate appendix of the final report. The report will contain a section on QA issues documenting any corrective actions taken during the full-scale operations. Reporting requirements are described in Section 12.0 of this QAPP.

9.3.2 Laboratory Data Reporting

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

1. Case Narrative:
   a. Date of issuance;
   b. Laboratory analysis performed;
   c. Any deviations from intended analytical strategy;
   d. Laboratory batch number;
   e. Numbers of samples and respective matrices;
   f. Quality control procedures utilized and also references to the acceptance criteria;
   g. Laboratory report contents;
   h. Project name and number;
   I. Condition of samples 'as-received' including cooler temperature;
   j. Discussion of whether or not sample holding times were met;
   k. Discussion of technical problems or other observations which may have created analytical difficulties;
   l. Discussion of any laboratory quality control checks which failed to meet project criteria; and
   m. Signature of the Laboratory QA Manager.

2. Chemistry Data Package

   a. Case narrative for each analyzed batch of samples;
   b. Summary page indicating dates of analyses for samples and laboratory

quality control checks;

c. Cross referencing of laboratory sample number to project sample identification numbers;
d. Data qualifiers to be used should be adequately described;
e. Sample preparation and analyses for samples ;
f. Sample results;
g. Raw data for sample results and laboratory quality control samples;
h. Results of (dated) initial and continuing calibration checks, and GC/MS tuning results;
i. Matrix spike and matrix spike duplicate recoveries, laboratory control samples, method blank results, calibration check compounds, and system performance check compound results; and
j. Labeled (and dated) chromatograms/spectra of sample results and laboratory quality control checks.

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

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

Contaminants	Method Requirements	Deliverables
Organics	Method blank spikes with results and control charts. Run with each batch of samples processed.	Control chart
	Results to be reported on Form 1. Sample results using standard data flags. Raw data must be submitted with data deliverable package.	Form 1, Sample chromatograms and mass spectra
	Surrogate recovery from samples reported on Form 2. ~~For volatiles by GC, the names of surrogates should be changed to reflect the surrogate used.~~	Form 2
	Matrix spike/spike duplicate data; one spike and spike duplicate per QC lot of samples of similar matrix reported on Form 3.	Form 3, sample chromatograms and mass spectra
	Method blank data reported on Form 4.	Form 4, sample chromatograms and mass spectra
	GC/MS tuning results for volatiles/semi-volatiles.	Form 5
	Initial calibration data. Initial calibration data with response factors for GC/HPLC methods.	Form 6, sample chromatograms and mass spectra No form Form 9, sample chromatograms and mass spectra Raw data will be submitted as applicable.
	Continuing calibration GC/MS data For GC and HPLC methods, the response factors and their percent differences from the initial calibration must be reported. Internal Standard Area for volatiles.	Form 7 SLF Form 8
	Second column confirmation shall be done for all GC and HPLC work when compounds are detected above reporting limits.	Form 9 Chromatograms
	Internal standard area for GC/MS analyses shall be supplied.	Form 8
	Instrument Logs	SLF (std. lab form)
	Standard Preparation Logs	SLF

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

Contaminants	Method Requirements	Deliverables
Organics	Method blank spikes with results and control charts. Run with each batch of samples processed.	Control chart
	Results to be reported on Form 1. Sample results using standard data flags. Raw data must be submitted with data deliverable package.	Form 1, Sample chromatograms and mass spectra
	Surrogate recovery from samples reported on Form 2. For volatiles by GC, the names of surrogates should be changed to reflect the surrogate used.	Form 2
	Matrix spike/spike duplicate data; one spike and spike duplicate per QC lot of samples of similar matrix reported on Form 3.	Form 3, sample chromatograms and mass spectra
	Method blank data reported on Form 4.	Form 4, sample chromatograms and mass spectra
	GC/MS tuning results for volatiles/semi-volatiles.	Form 5
	Initial calibration data. Initial calibration data with response factors for GC/HPLC methods.	Form 6, sample chromatograms and mass spectra No form Form 9, sample chromatograms and mass spectra Raw data will be submitted as applicable.
	Continuing calibration GC/MS data For GC and HPLC methods, the response factors and their percent differences from the initial calibration must be reported. Internal Standard Area for volatiles.	Form 7 SLF Form 8
	Second column confirmation shall be done for all GC and HPLC work when compounds are detected above reporting limits.	Form 9 Chromatograms
	Internal standard area for GC/MS analyses shall be supplied.	Form 8
	Instrument Logs	SLF (std. lab form)
	Standard Preparation Logs	SLF

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

Contaminants	Method Requirements	Deliverables
Organics (Cont'd.)	Sample Preparation Logs	SLF
	Internal COC form	SLF
Metals	Method blank spikes with results and control charts. Run with each batch of samples processed.	Control chart
	Sample results with standard CLP flagging system. Raw data must be submitted with the data deliverable package.	Form 1/Raw Data
	Initial and continuing calibration.	Form 2, Part 1 only, Raw Data
	Blanks 10% frequency	Form 3
	Method blank taken through digestion (1 per 20 samples of same matrix).	Form 3
	ICP interference check sample.	Form 4
	Matrix spike recovery (1 per 20 samples of similar matrix).	Form 5, Part 1
	Postdigestion spike sample recovery for ICP metals. Only done if predigest spike recovery exceed laboratory standards.	Form 5, Part 2 (never used for GFAA work)
	Postdigest spike for GFAA. Duplicates (1 per 20 samples will be split and digested as separate).	Recovery noted on raw data Form 6 samples
	Method blank spike information will be plotted on control chart, one per batch of samples processed.	Control chart
	Standard addition, as required.	Form 8
	Holding times	Form 10
	Instrument Logs	Standard Lab Form (SLF)
	Standard Preparation Logs	SLF
	Sample Preparation Logs	SLF
	Internal COC form	SLF

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

Contaminants	Method Requirements	Deliverables
Wet Chemistry	Method blank spikes with results and control charts. Run with each batch of samples processed.	Report result, control chart
	Method blank 1 per batch	Report result, no format
	Sample results (raw and final data)	Report result, no format
	Matrix spike/spike duplicate or calibration information (Raw data)	Report result if applicable
	Instrument Logs	Standard Lab Form (SLF)
	Standard Preparation Logs	SLF
	Sample Preparation Logs	SLF
	Internal COC form	SLF
	Calibration check report percent RSD or percent difference from initial calibration	Report percent or percent difference, no format
General Requirements	Method blank spikes with results and control charts. Run with each batch of samples processed.	Report result, control chart
	Internal Chain-of-Custody forms	SLF
	Instrument logs (what samples have been run and under what conditions)	SLF
	Summaries of Extraction Logs	SLF
	Standards Logs	SLF
	Spiking Logs	SLF
	Quantification Reports	SLF
	Percent Moisture Logs	SLF
	Standard Chromatograms	SLF
	Standard Spectra	SLF
	QC Comments (unless already included in the case narrative)	SLF

SECTION 10
PERFORMANCE AND SYSTEM AUDITS

10.1 PERFORMANCE AND SYSTEM AUDITS AND FREQUENCY

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

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

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

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

10.2 FIELD PERFORMANCE AND SYSTEM AUDITS

10.2.1 Internal Field Audits

Internal Field Audit Responsibilities

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

requirements.

Internal Field Audit Frequency

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

Internal Field Audit Procedures

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

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

10.2.2 External Field Audits

External Field Audit Responsibilities

External field audits may be conducted by the U.S. EPA Project Coordinator.

External Field Audit Frequency

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

10.3 LABORATORY PERFORMANCE AND SYSTEMS AUDITS

10.3.1 Internal Laboratory Audits

Internal Lab Audit Responsibilities

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

Internal Lab Audit Frequency

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

Internal Lab Audit Procedures

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

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

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

10.3.2 External Laboratory Audits

External Lab Audit Responsibilities

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

External Lab Audit Frequency

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

Overview of the External Lab Audit Process

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

SECTION 11
PREVENTATIVE MAINTENANCE

11.1 FIELD INSTRUMENT PREVENTATIVE MAINTENANCE

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

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

11.2 LABORATORY INSTRUMENT PREVENTATIVE MAINTENANCE

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

TABLE 11-1
SUMMARY OF MAINTENANCE - FIELD EQUIPMENT

Instrument	Maintenance Schedule	Spare Parts
PID (MicroTIP)	1. Calibrate at least once daily, prior to use, and as necessary during use. 2. Check battery and recharge when low. 3. Clean lamp window every 24 hours of operation. 4. Replace dust filter every 240 hours of operation. 5. Replace sample pump every 5000 hours of operation.	Battery charger Spare lamps Spare filter cartridges Span gas (isobutylene) Tedlar bags
pH Meter	1. Calibrate daily, prior to use.	pH buffers Batteries paper towels disposable gloves plastic cups
Temperature/Oxygen Meters (probes)	1. Calibrate if monitor goes off. 2. Check battery and recharge when low.	Battery charger

TABLE 11-1
SUMMARY OF MAINTENANCE - FIELD EQUIPMENT

Instrument	Maintenance Schedule	Spare Parts
PID (MicroTIP)	1. Calibrate at least once daily, prior to use, and as necessary during use. 2. Check battery and recharge when low. 3. Clean lamp window every 24 hours of operation. 4. Replace dust filter every 240 hours of operation. 5. Replace sample pump every 5000 hours of operation.	Battery charger Spare lamps Spare filter cartridges Span gas (isobutylene) Tedlar bags
pH Meter	1. Calibrate daily, prior to use.	pH buffers Batteries paper towels disposable gloves plastic cups
Temperature/Oxygen Meters (probes)	1. Calibrate if monitor goes off. 2. Check battery and recharge when low.	Battery charger
Field Colorimetry	1. Verify correct wavelength setting. 2. Zero prior to use	Cuvettes Spare lamp

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

12.1 ACCURACY ASSESSMENT

In order to assure the accuracy of the analytical procedures, an environmental sample is randomly selected from each sample shipment received at the laboratory, and spiked with a known amount of the analyte or analyses to be evaluated. For volatile organics in soils and many liquid analyses, however, field personnel randomly select samples to provide additional volume for spiking purposes (stated in Section 4.6.5). In general, a sample spike should be included in every set of 20 samples tested on each instrument. The spike sample is then analyzed. The increase in concentration of the analyte observed in the spiked sample, due to the addition of a known quantity of the analyte, compared to the reported value of the same analyte in the unspiked sample determines the percent recovery. Control charts are plotted for each commonly analyzed compound and kept on instrument, matrix, and analyte specific bases. The percent recovery for a spiked sample is calculated according to the following formula:

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

Acceptable limits for the data are presented in Tables 3.1 - 3.8 of QAPP Section 3.6.

12.2 PRECISION ASSESSMENT

12.2.1 Laboratory Data

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

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

Acceptable limits for the data are presented in Tables 3.1 - 3.8 of QAPP Section 3.6.

12.2.2 Field Data

RPD for field data will be calculated on a weekly basis. The criteria in Table 6-1 will be used to determine the need for closer examination of data collection and instrument calibration procedures, however, accedence of these suggested values will not be cause for rejection of the data. Note that Oxygen criteria varies significantly due to the wide variety of material consistency and instrument readouts in 1° increments. Duplicate readings shall be monitored. The RPD between the initial reading and duplicate reading are calculated. The RPD is calculated according to the following formula:

$$RPD = \frac{|\text{Initial Reading} - \text{Duplicate Reading}|}{0.5(\text{Initial Reading} + \text{Duplicate Reading})} \times 100$$

TABLE 12-1
DATA ACCEPTANCE CRITERIA AND SUGGESTED CORRECTIVE ACTION

Measurement	Acceptable RPD	Corrective Action
Temperature	10%	• check instrument calibration • verify adherence to Field SOP QAPP-4.0
Moisture Level	20%	• verify adherence to Field SOP QAPP-4.0
pH	15%	• check instrument calibration • verify adherence to Field SOP QAPP-4.0

12.3 COMPLETENESS ASSESSMENT

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

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

SECTION 13
CORRECTIVE ACTION

13.1 CHANGE CONTROL

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

13.2 FIELD CORRECTIVE ACTION

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

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

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

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

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

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

- Cause and description of the nonconformance,

- Method(s) for correcting the nonconformance (corrective action) or description of the variance granted,

- Schedule for completing corrective action, and
- Any required approval signatures.

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

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

13.3 LABORATORY CORRECTIVE ACTION

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

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

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

- Schedule for completing corrective action, and
- Any required approval signatures.

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

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

These guidelines are presented in the laboratory SOPs for corrective action provided in Appendix C.

These corrective actions are performed prior to release of the data from the laboratory. The corrective action will be documented in both Laboratory's corrective action log (signed by analyst, section leader and quality control coordinator), and the narrative

data report sent from the laboratory to the MK Project Chemist. If corrective action does not rectify the situation, the laboratory will contact the NSWC Project Manager.

13.4 CORRECTIVE ACTION DURING DATA VALIDATION AND DATA ASSESSMENT

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

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

SECTION 14
QUALITY ASSURANCE REPORTS (POST-AUDIT)

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

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

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

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

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

SECTION 15
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APPENDIX A
FIELD STANDARD OPERATING PROCEDURES

FIELD SOP: QAPP-1.0
TITLE: SOIL SAMPLE COLLECTION

1.0 PURPOSE

This procedure describes the methods which will be used to establish sampling locations and collect soil samples for laboratory analysis. Quality assurance requirements for field sampling are described in Section 4.0 of this QAPP for Full-Scale Operations at the Biofacility.

Task 1:
-Identify areas for Full-Scale excavation.
-Measure "entry " concentrations of explosives in soil to be treated.
-Determine if soil is hazardous with respect to metals and VOCs.

Task 5:
-Determine levels of explosives and metals still remaining following Full-Scale excavation

2.0 ESTABLISH SAMPLING LOCATIONS

This section describes the procedure to be followed for establishing the locations for collecting soil samples for laboratory analyses for full-scale operations at the various excavation sites (SWMUs).

2.1 Tools and Materials

The following tools and materials are required to establish field sample collection locations for off-site laboratory analysis.

small stainless steel trowel, stainless steel hand shovel, stainless steel spoons
large shovel
flagging and pins
measuring tape
sample containers
PID
~~drive hammer~~auger
~~core sampler, split core sampler~~
~~brass sleeves and end caps~~
~~Teflon film~~Encore samplers with T-handles
plastic sheets
stainless steel bowls
disposable gloves
logbook
indelible pen

2.2 Placing the Sampling Grid

Task 1: Excavation Site Characterization

1. Layout grid as specified in the Soil Excavation Plan, Appendix E of the Full-Scale Operational Plan. Each grid block may or may not be square, due to the presence of utilities, buildings or roads.

2. Label these blocks or sampling areas, as shown in the Soil Excavation Plan, Appendix E of the Full-Scale Operational Plan.

Task 5: Post-Excavation Sampling

1. Layout a grid using nodal spacings (as shown in the Soil Excavation Plan, Appendix E of the Full-Scale Operational Plan) along the base of the excavation(s). Place pin flags at each node.

2. For the sidewalls, place pin flags every 20 feet, along each wall. Place the flags midway between ground surface and the excavation base. If a sidewall is less than one foot in height, do not place flags and do not collect samples from the wall.

3.0 SAMPLE COLLECTION

This section describes the procedure to be followed for collecting sample material from the excavation sites. All non-disposable sample extraction tools which come in contact with the sample shall be decontaminated prior to use as described in Field SOP 7.0 of the Full-Scale Operational Plan, Appendix D. VOC sampling procedure using the EnCore Sampler is provided as Attachment A to this SOP.

3.1 Pre-Excavation Sample Collection Procedure

Follow these steps when sampling each grid block or sampling area:

3.1.1 Grid Blocks (Task 1)

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

1. Place flags near each corner and at the center.

2. At the center of the grid block, clear surface vegetation and debris in a 6-inch by

6-inch or larger area. ~~Using a stainless steel trowel and spoon or shovel, remove the upper few inches of soil to remove vegetation roots.~~

3. Using an auger, collect soil from the 0 to 12-inch depth and place into the first stainless steel bowl marked grabs for metals and explosives - 0 to 1 foot.

4. Using ~~a core sampler with a brass sleeve, collect the~~an EnCore soil sampler, collect three separate EnCore samplers from the 12 to 18 inch depth. ~~Cover both ends of the brass sleeve~~Seal with ~~Teflon tap, cap, and label for volatile organic analysis~~the cap provided verifying the correctly seated cap. Label for volatile organic analysis. In each group and every twenty samples collect two additional EnCore sample vials for ~~QC~~spiking purposes and two additional EnCore sample vials for field duplicate analysis. Use remaining soil from this depth to fill a 2-ounce jar for moisture determination.

5. Remove soil from the boring down to 2 feet.

6. Using an auger or slit core sampler, collect soil from the 2-foot to 2.5-foot range. Push the contents into a disposable sealable plastic bag filling one half full and break up the soil. Wait a few minutes, open the plastic bag, and screen the headspace using a PID. Place the soil from the bag in the second stainless steel bowl marked metals/explosives grabs - 2 to 2.5 feet. If the PID readings are above background levels, collect ~~a sample~~three EnCore samplers from the 2.5 to 3 foot depth ~~with the core sampler using a brass sleeve. Cover the ends of the sleeve with Teflon tape, cap, and label for volatile organic analysis.~~ Seal with the cap provided verifying the correctly seated cap. Label for volatile organic analysis.

7. At each of the four corners, clear surface vegetation and debris in a 6-inch by 6-inch area or greater. ~~Using a stainless steel trowel and spoon or shovel, remove the upper few inches of soil to remove vegetation roots.~~

8. Using an auger or split core sampler collect a soil sample from the 0 to 12-inch depth. Push the contents into a disposable sealable plastic bag filling one half full and break up the soil. Wait a few minutes, open the plastic bag, and screen the headspace using a PID. If the PID readings are above background levels, collect ~~a sample~~three samples from the 1 to 1.5 foot depth with ~~a core~~an Encore sampler ~~using a brass sleeve. Cover~~. Seal with the cap provided verifying the ~~ends of the sleeve with Teflon tape, cap,~~correctly seated cap and label for volatile organic analysis. Place the soil in the bag into the first stainless steel bowl for all other analysis.

9. Remove soil from the boring down to 2 feet.

10. Using an auger or split core sampler, collect soil from the 2-foot to 2.5-foot range. Push the contents into the stainless steel bowl labeled "grabs".

11. At a minimum, one bowl of soil will result from the 0-12 inch depth and one bowl of soil will result from the 2-3 foot depth. A minimum of one sample, collected from the center of the grid block, will be tested for VOCs.

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

Background levels are established at every sampling event and are based on ambient air independent of the excavation location.

12. For forming a composite sample for each depth, place each grab sample into a large stainless steel bowl~~, after removing~~ and remove vegetation and large rocks. ~~Break~~Homogonize samples by breaking up the soil using ~~gloved hands, remove large rocks~~a pestle or spoon (the soil may also be sieved)~~, and allow soil to air dry~~.

13. Mix the soil thoroughly in the bowl.

14. Collect equal portions from the bowl to fill one 8-ounce jar for explosives analysis.

15. Fill the remaining jars listed in Table 4-4 of QAPP.

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

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

3.2 In-Process Excavation Sample Collection Procedure

3.2.1 Grid Blocks (Task 5)

Samples will only be analyzed for explosives using the field screening kits (RDX and TNT).

1. At the center of the grid, using an auger sampler, collect soil from the 0 to ~~12-inch~~6-inch depth.

2. Place the sample in a disposable sealable plastic bag and label. Store the bag in a cooler with bagged ice.

3. At each of the four corners, using an auger sampler, collect soil from the 0 to ~~12-inch~~6-inch depth. Place in stainless steel bowl and homogonize into a single composite. Remove vegetation large rocks. Homogonize samples by breaking up soil using a pestle or spoon(the soil may also be sieved).

4. Place the sample in a disposable sealable plastic bag and label. Store the bag in a cooler with bagged ice.

3.2.2 Sidewalls (Task 5 only)

Collect a sample from each flagged location along each sidewall from 0-6 inches for explosives analysis using the field screening kits (RDX and TNT).

3.3 Post-Excavation Sample Collection Procedure

3.3.1 Grid Blocks (Task 5)

Follow the same procedure as for Section 3.1.1 (Task 1) to identify sample locations, however ~~do not collect EnCore samplers or samples from the 2-3 foot depth. Samples will~~ only one composite sample and one grab sample (for VOCs) will be ~~analyzed for SWMU-specific contaminants listed in Section 1.4.2.1 of QAPP~~collected from each grid block. The composite sample will be analyzed for SWMU-specific parameters (Table 1-11), and will be collected from 0"-6" interval in a manner similar to pre-excavation composite samples. The grab sample will be analyzed for site-specific VOCs listed in Section 1.4.2.1 of QAPP and will be collected from the center of the grid block 0"-6" below excavated ground surgace using three EnCore samplers.

3.3.2 Sidewalls (Task 5 only)

Collect a sample from each flagged location along each sidewall using the procedure from Step 2 of Section 3.1.1. Submit ~~one sleeve~~three EnCore samplers for SWMU-specific VOCs analysis from 12-18 inches below ground surface. Submit one sample from ~~0-120~~-6 inches for SWMU-specific contaminants listed in Section 1.4.2.1 of QAPP.

3.4 QA/QC Sample Collection

One field duplicate will be collected for every 10 explosive samples and every 20 regular samples collected. Field duplicate locations will be marked in the field using different colored flags for VOC samples. Additional volume for the field duplicate will be drawn from the composite mixture for SWMU-specific contaminants. For the VOCs field duplicate, ~~a sleeve of soil~~two extra EnCore samplers will be collected from the 18-24 inch depth, at the center of the grid block. For Tasks 1 and 5, the duplicate will be analyzed for SWMU-specific contaminants.

Field equipment will be decontaminated using procedures in Section 4.7. One equipment rinsate will be collected for every 20 times equipment is rinsed . Sampling parameters are listed in Table 4-3.

One field blank will be collected of the water used in decontamination once a month during excavation. Sampling parameters are listed in Table 4-3.

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

Attachment A
EnCore Sampler -- Sampling Procedures

Disposable En Core™ Sampler

En Novative Technologies, Inc.
1241 Bellevue Street
Green Bay, WI 54302
Telephone: 920-465-3960 • Toll Free: 1-888-411-0757
Fax: 920-465-3963

SAMPLING PROCEDURES

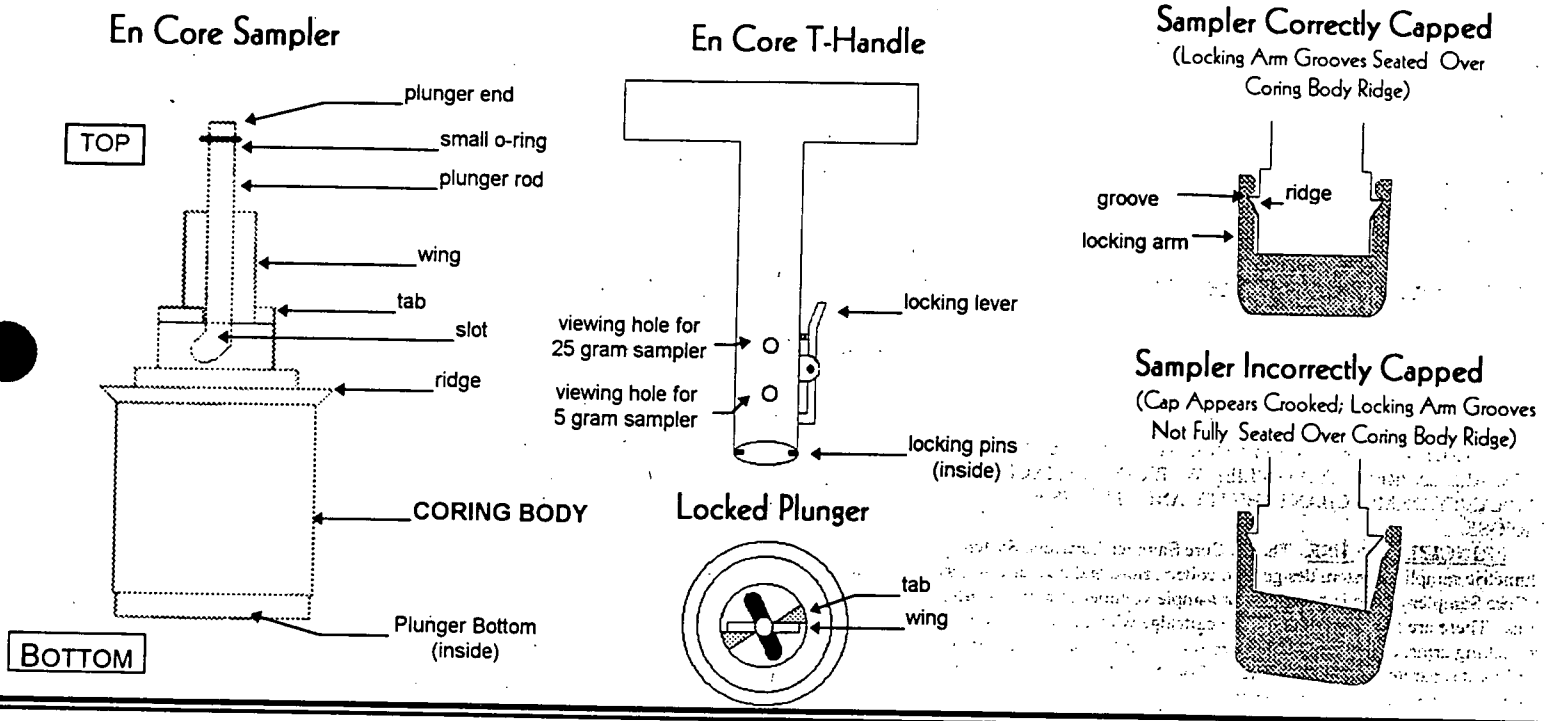
USING THE En Core™ T-HANDLE

NOTE:

1. En Core Sampler is a SINGLE USE device. It cannot be cleaned and/or reused.

2. En Core Sampler is designed to store soil. Do not use En Core Sampler to store solvent or free product!

3. En Core Sampler must be used with En Core™ T-Handle and/or En Core™ Extrusion Tool exclusively. (These items are sold separately.)



BEFORE TAKING SAMPLE:

1. Hold **coring body** and push **plunger rod** down until **small o-ring** rests against **tabs**. This will assure that plunger moves freely.

2. Depress **locking lever** on En Core T-Handle. Place coring body, **plunger end** first, into open end of T-Handle, *aligning the (2) slots on the coring body with the (2) **locking pins** in the T-Handle*. Twist coring body clockwise to lock pins in slots. Check to ensure Sampler is locked in place. Sampler is ready for use.

TAKING SAMPLE:

3. Turn T-Handle with T-up and coring body down. This positions plunger bottom flush with bottom of coring body (ensure that plunger bottom is in position). Using T-Handle, push Sampler into soil until coring body is completely full. When full, small o-ring will be centered in T-Handle **viewing hole**. Remove Sampler from soil. Wipe excess soil from coring body exterior.

4. Cap coring body while it is still on T-handle. Push and twist **cap** over bottom until **grooves** on **locking arms** seat over **ridge** on coring body. CAP MUST BE SEATED TO SEAL SAMPLER (see diagram).

PREPARING SAMPLER FOR SHIPMENT:

5. Remove the capped Sampler by depressing locking lever on T-Handle while twisting and pulling Sampler from T-Handle.

6. Lock plunger by rotating extended plunger rod fully counter-clockwise until **wings** rest firmly against tabs (see plunger diagram).

7. Attach completed circular label (from En Core Sampler bag) to cap on coring body.

8. Return full En Core Sampler to zipper bag. Seal bag and put on ice.

Disposable En Core™ Sampler

EXTRUSION PROCEDURES

USING THE En Core™ EXTRUSION TOOL

CAUTION! Always use the Extrusion Tool to extrude soil from the En Core Sampler. If the Extrusion Tool is not used, the Sampler may fragment, causing injury.

1. Use a pliers to break locking arms on cap of En Core Sampler. Do not remove cap at this time. **(CAUTION: Broken edges will be sharp.)**
2. To attach En Core Sampler to En Core Extrusion Tool: Depress locking lever on Extrusion Tool and place Sampler, plunger end first, into open end of Extrusion Tool, *aligning slots on coring body with pins in Extrusion Tool.* Turn coring body clockwise until it locks into place. Release locking lever.
3. Rotate and gently push Extrusion Tool plunger knob clockwise until plunger slides over wings of coring body. (When properly positioned plunger will not rotate further.)
4. To release soil core, remove cap from Sampler and push down on plunger knob of En Core Extrusion Tool. Remove and properly dispose of En Core Sampler.

Warranty and Disclaimers

IMPORTANT: FAILURE TO USE THE EN CORE™ SAMPLER IN COMPLIANCE WITH THE WRITTEN INSTRUCTIONS PROVIDED HEREIN VOIDS ALL EXPRESS AND IMPLIED WARRANTIES, INCLUDING WARRANTY OF MERCHANTABILITY AND FITNESS FOR A PARTICULAR PURPOSE.

PRINCIPLE OF USE. The En Core Sampler Cartridge System is a volumetric sampling system designed to collect, store and deliver a soil sample. The En Core Sampler comes in two sizes for sample volumes of approximately 25 or 5 grams. There are four components: the cartridge with a movable plunger; a cap with two locking arms; a T-handle (purchased separately); and an extrusion handle (purchased separately). NOTE: The En Core Sampler is designed to store soil. It is not designed to store solvent or free product.

The soil is stored in a sealed headspace-free state. The seals are achieved by three special Viton®.* o-rings, two located on the plunger and one on the cap of the Sampler. **At no time and under no condition should these o-rings be removed or disturbed.**

QUALITY CONTROL. The cartridge is sealed in an airtight package to prevent contamination prior to use. Due to the stringent quality control requirements associated with the use of this system, the disposable cartridge is designed to be used only once.

WARRANTY. En Novative Technologies, Inc. ("En Novative Technologies") warrants that the En Core Sampler shall perform consistent with the research conducted under En Novative Technologies' approval, within thirty (30) days from the date of delivery, provided that the Customer gives En Novative Technologies prompt notice of any defect or failure to perform and satisfactory proof thereof. THIS WARRANTY DOES NOT APPLY TO THE FOLLOWING, AS SOLELY DETERMINED BY EN NOVATIVE TECHNOLOGIES: (a) Damage caused by accident, abuse, mishandling or dropping; (b) Samplers that have been opened, taken apart or mishandled; (c) Samplers not used in accordance with the directions; and (d) Damages exceeding the cost of the sampler. Seller warrants that all En Core Samplers are free from defects in title. THE FOREGOING WARRANTIES ARE IN LIEU OF ALL OTHER WARRANTIES, WHETHER ORAL, WRITTEN, EXPRESSED, IMPLIED OR STATUTORY, INCLUDING ANY INFORMATION PROVIDED BY SALES REPRESENTATIVES OR IN MARKETING LITERATURE. IMPLIED WARRANTIES OF FITNESS AND MERCHANTABILITY SHALL NOT APPLY. En Novative Technologies' warranty obligations and Customer's remedies, except as to title, are solely and exclusively as stated herein.

LIMITATION OF LIABILITY. IN NO EVENT SHALL EN NOVATIVE TECHNOLOGIES BE LIABLE FOR ANTICIPATED PROFITS, INCIDENTAL, SPECIAL OR CONSEQUENTIAL DAMAGES, INCLUDING, BUT NOT LIMITED TO, DAMAGES FOR LOSS OF REVENUE, DOWN TIME, REMEDIATION ACTIVITIES, REMOBILIZATION OR RESAMPLING, COST OF CAPITAL, SERVICE INTERRUPTION OR FAILURE OF SUPPLY, LIABILITY OF CUSTOMER TO A THIRD PARTY, OR FOR LABOR, OVERHEAD, TRANSPORTATION, SUBSTITUTE SUPPLY SOURCES OR ANY OTHER EXPENSE, DAMAGE OR LOSS, INCLUDING PERSONAL INJURY OR PROPERTY DAMAGE. En Novative Technologies' liability on any claim of any kind shall be replacement of the En Core Sampler or refund of the purchase price. En Novative Technologies shall not be liable for penalties of any description whatsoever. In the event the En Core Sampler will be utilized by Customer on behalf of a third party, such third party shall not occupy the position of a third-party beneficiary of the obligation or warranty provided by En Novative Technologies, and no such third party shall have the right to enforce same. All claims must be brought within one (1) year of shipment, regardless of their nature.

En Novative Technologies, Inc.
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Green Bay, WI 54302
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The En Core™ Sampler is covered by One or More of the Following U.S. Patents: 5,343,771; 5,505,098; 5,517,868; 5,522,271. Other U.S. and Foreign Patents Pending.

* Vitron® is a registered trademark of DuPont Dow Elastomers.

FIELD SOP: QAPP-2.0
TITLE: WATER SAMPLE COLLECTION

1.0 PURPOSE

The purpose of this procedure is to collect representative samples and obtain data necessary to properly dispose of waste water generated during site activities.

This procedure describes the methods which will be used to establish sampling locations and collect water samples for laboratory analysis. Quality assurance requirements for field sampling are described in Section 4.0 of this QAPP for Full-Scale Operations at the Biofacility.

Decontamination water generated from the cleaning of sampling equipment and reusable personal protective equipment (PPE) will be collected and stored in portable temporary storage tanks or 55 gallon drums. Accumulated storm water and surface runoff will be collected in two retention ponds. The water will be sampled and analyzed for disposal or release. Table 4-3 of this QAPP shows the Water Sampling and Analysis Schedule for the waste water.

2.0 TOOLS AND MATERIALS

The following tools and materials are required to perform field water sample collection for off-site laboratory analysis.

stainless steel bucket or ladle
slow rate pump and disposable tubing (may be used for tank sampling if tank is too large to access with a bailer)
fishing line, cord, or reel
disposable Teflon or polyethylene bottom filling bailer ~~or~~ sampling scoop ~~or jar on a long stick~~, dipper (type used for surface water sampling)
appropriate sample containers (see Section 4.0 of this QAPP)
appropriate chain-of-custody, labels, tags, etc.
disposable gloves
logbook
indelible pen

3.0 WASTE WATER STORAGE TANK SAMPLING

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

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

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

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

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

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

5. Follow the sample labeling, tagging and identification, chain of custody, and shipping and handling procedures described in Section 5.0 of this QAPP. Document all sampling activities in the field logbook.

4.0 ROCK RINSE WATER SAMPLING

Rock rinse water is required to be sampled and analyzed to determine effectiveness of washing and rinsing procedures prior to disposition of the rocks.

4.1 Sample Collection

1. After performing the decontamination process as described in SOP 7.0 of the Full-Scale Operational Plan, Appendix D, elevate the bin containing the decontaminated rocks high enough to place a rinse sample collection container underneath.

2. Place the new or decontaminated rinse water collection container under the elevated rock bin and pour slightly more than 2 liters of ~~distilled or micro-filtered~~deionized water over the rocks as a final rinse.

3. Transfer the final rinse water into two one-liter amber glass jars for off-site laboratory analysis. Follow the sample labeling, tagging and identification, chain of custody, and shipping and handling procedures described in Section 5.0 of this QAPP. Document all sampling activities in the logbook.

4. Cover and store the decontaminated rocks in a lined and bermed area until final disposition has been obtained.

5.0 STORM WATER RETENTION POND SAMPLING

Storm water is required to be sampled and analyzed to determine proper disposal/release disposition of the water.

5.1 Sample Collection Procedure

The same procedure described above for collecting samples from waste water (Section 3.0) will be used to collect samples of storm water and run-off water in the retention ponds. If the water level in the retention ponds is too low to sample using a bottom-filling bailer, then a ~~sampling device on a stick or rod~~ decontaminated polyethylene dipper will be filled by submerging in the water. The sample shall be transferred to the appropriate sampling container. At no time will the person collecting the sample enter the retention ponds.

5.2 Alternative Storm/run-off Water Sampling

An NSWC Crane representative may perform the storm/run-off water sampling using their procedures, in lieu of the above description, based on the IDEM approved NPDES permit requirements.

6.0 QA/QC SAMPLE COLLECTION

~~one~~One field duplicate for every 10 explosive samples and every 20 ~~samples taken will be~~regular samples collected and analyzed for the same parameters as the regular samples.

If non-disposable equipment is used for sampling (i.e stainless steel bailer or polyethylene dipper), one rinsate blank will be collected every 20 rinses and analyzed for the same parameters as the regular samples.

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

Documentation procedures are provided in Section 5.0 of this QAPP. Packaging and shipping instructions are provided in SOP QAPP-6.0 of this QAPP.

FIELD SOP: QAPP-3.0
TITLE: COMPOST SAMPLE COLLECTION

1.0 PURPOSE

This procedure describes the methods which will be used to establish sampling locations and collect compost samples for laboratory analysis. Quality assurance requirements for field sampling are described in Section 4.0 of this QAPP for Full-Scale Operations at the Biofacility.

2.0 ESTABLISH SAMPLING LOCATIONS

This section describes the procedure to be followed for establishing the locations for collecting compost samples for laboratory analyses for full-scale operations at the Bioremediation Facility.

2.1 Tools and Materials

The following tools and materials are required to establish field sample collection locations for off-site laboratory analysis.

4 ft 7 in.-long measuring stick
pin flags
logbook
indelible pen

2.2 Sample Cross-sections and Locations

Cross-sections

Locate cross-sections at Column Numbers 3, 5, 7, 9, and 11 as described in Section 2.2 of SOP QAPP-4.0.

Sample locations

Three sample locations will be established at each of the cross-sections as shown in Figure 1 of this SOP. The samples will be taken after all required activities discussed in SOP QAPP-4.0 have first been performed. The following procedure will then be used to identify the sample collection locations:

1. Sample location 1 is located on the longitudinal centerline of the pile at a depth of 3 feet below the top surface.

2. Sample location 2 is located on the south side of the pile at a depth of 2.5 feet perpendicular to the surface. Lay the 4 ft 7 in. measuring stick on the side of the

pile from the ground surface up and proceed to collect the sample per Section 3.0.

3. Sample location 3 is located on the north side of the pile at a depth of 2.5 feet below the surface. Lay the 4 ft 7 in. measuring stick on the side of the pile from the ground surface up and proceed to collect the sample per Section 3.0.

3.0 SAMPLE COLLECTION

This section describes the procedure to be followed for collecting sample material from the compost windrows for laboratory analyses.

3.1 Tools and Materials

stainless steel push tubes with sleeves or split spoon and/or hand auger
4 ft 7 in.-long measuring stick
appropriate sample containers (see Section 4 of this QAPP)
disposable gloves
stainless steel bowl
logbook
indelible pen
sample tags
sample labels
coolers
preservatives
chain of custody forms
sealable, disposable plastic bags

All non-disposable sample extraction tools which come in contact with the sample shall be decontaminated prior to use as described in Field SOP 7.0 of the Full-Scale Operational Plan, Appendix D.

3.2 Sample Collection Procedure

Samples for off-site laboratory analysis will be discrete as described in Section 4 of this QAPP. Discrete samples will be collected from locations 1, 2, and 3 at each cross-section, using a hand auger. Samples for explosives analysis using field test kits will be composites from locations 1, 2, and 3. Table 4-3 of this QAPP shows the Process Sampling and Analysis Schedule for the compost material.

FIELD SOP: QAPP-3.0
TITLE: COMPOST SAMPLE COLLECTION

The following procedure shall be used to collect samples at each cross-section:

1. Locate cross-sections at column numbers 3, 5, 7, 9, and 11 and sample locations 1, 2, and 3 at each cross-section as described in Section 2.2 above.

2. Insert the push tube/auger into and perpendicular to the pile at each sample location. Rotate and advance the tube/auger to the designated depth (3 feet for sample location 1, and 2.5 feet for sample locations 2 and 3) or a little deeper to insure a complete sample at that depth. Remove the material between the surface and the designated depth at each sampling location, placing the removed material on the pile, until the auger has reached the designated depth (3 ft for sample location 1, and 2.5 feet for sample locations 2 and 3). Care shall be taken so as not to collapse the hole or have surface material fall in.

3. Fill the tube/auger with the material at the designated sampling depth. Remove the push tube/auger and extract the material from the tube/auger from the appropriate depth location. Place the extracted material directly into the appropriate sample container.

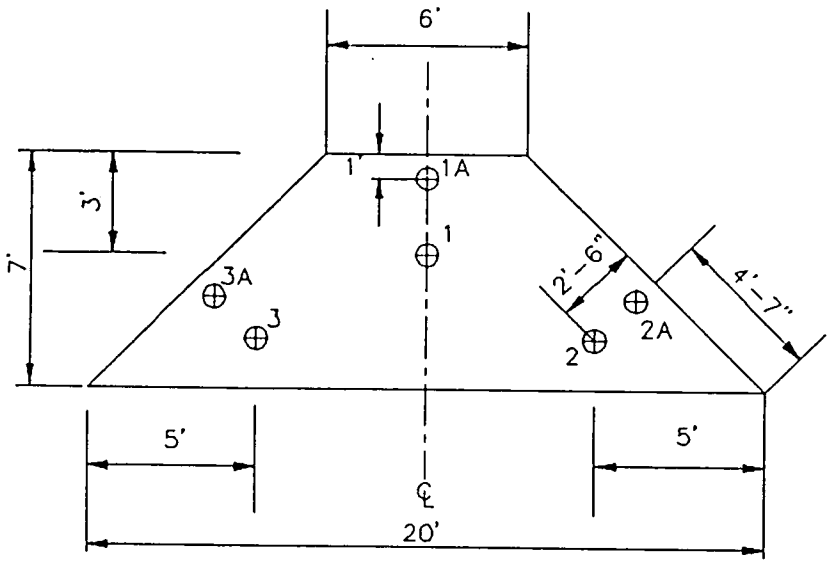
4. Place the remaining sample on the pile.

5. The samples will be taken to the on-site laboratory for preparation before shipment or field analysis. Follow the sample labeling, tagging, identification, chain of custody, and shipping and handling procedures described in Section 4.0 of the approved Full-Scale QAPP. Document all sampling activities in the logbook.

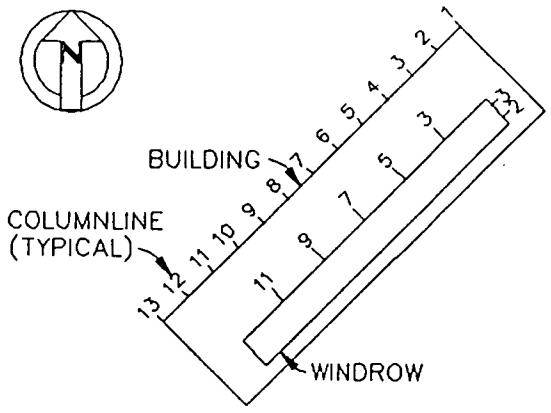
6. New sample containers shall be used for each sample. Decontamination procedures are described in Field SOP 7.0 of the Full-Scale Operations Plan, Appendix D.

7. For composite samples, steps 1 through 4 will be repeated except the samples collected from locations 1,2, and 3 will be placed in a stainless steel bowl and ~~thoroughly mixed~~homogonized. ~~The samples will be taken to the on-site laboratory for analysis.Coarse fractions and vegetation will be removed. Then using~~ Using a pestle or spoon, break up and thoroughly mix the soil. Quarter the soil in the bowl and collect equal portions from each quarter to fill an 8-ounce jar.

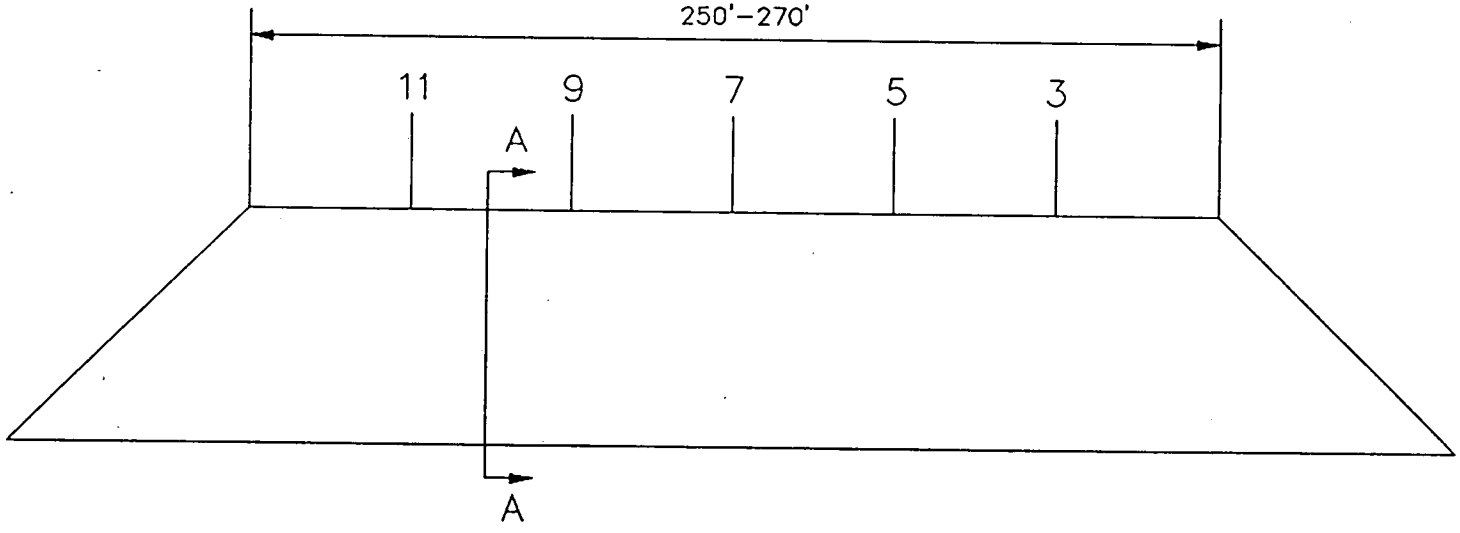
NOTE: ALL SAMPLE NUMBERS FOLLOWED BY AN "A" WILL BE TAKEN AT 1' BELOW THE SURFACE.



SECTION A–A



KEY PLAN



FULL SCALE
WINDROW SAMPLE LOCATIONS

MORRISON KNUDSEN CORP.	
Drawing FIGURE 1	Rev. 0
Date 1/27/98	Sheet 1

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1.0 PURPOSE

This procedure describes the methods which will be used to establish field measurement locations; and measure the temperature, moisture content, pH, and oxygen level of windrows. Quality assurance requirements for field measurements are described in Section 4.0 of this QAPP for Full-Scale Operations at the Biofacility.

2.0 ESTABLISH SAMPLING AND MONITORING LOCATIONS

This section describes the procedure to be followed for establishing the field measurement locations.

2.1 Tools and Materials

The following tools and materials are required to establish field measurement locations:

4 ft 7 in.-long measuring stick
Windrow Process Data - Daily Log form
indelible pen
oxygen and temperature monitoring probes

2.2 Establish Field Measurement Locations

Cross-section Locations

Each 250 feet long windrow will be divided into as many as five cross-sections, labeled 3, 5, 7, 9, and 11 as shown in Figure 1 of this SOP. Cross-section locations will be based on building column locations as described in the following procedure:

1. Locate cross-section 3 at building column line 3 as shown on Figure 1 of this SOP.

2. Locate cross-sections 5, 7, and 9 at column lines 5, 7, and 9 respectively.

3. Locate cross-section 11 at column line 11 initially, however, due to volume reduction during the composting process, cross-section 11 location may vary as the compost process progresses. Cross-section 11 will remain at column line 11 until the shoulder of the windrow (top southwest edge) reaches column line 11. Each day as the windrow continues to reduce in length, cross-section 11 will move with the shoulder from column line 11 towards column line 10. Once it appears beyond column line 10, it will be deleted and only four cross-sections will be located for the remaining compost cycle.

Field measurement locations

Six field measurement locations will be established at each of the windrow cross-sections. The correct depth for each measurement will be indicated by marked intervals on the shaft of the oxygen/temperature probe for oxygen and temperature measurements. The following procedure will be used to locate the field measurement locations:

1. Sample location 1A is located on the longitudinal centerline of the pile at a depth of 1 foot below the top surface. Insert the marked probe shaft to the 1-foot mark.

2. Sample location 1 is located on the longitudinal centerline of the pile at a depth of 3 feet below the top surface. Insert the marked probe shaft to the 3-foot mark.

3. Sample location 2A is located on the south side of the pile at a depth of 1 foot below the surface. Lay the 4 ft. 7 in. measuring stick on the side of the pile from the ground surface up, hold the marked probe shaft perpendicular to the side surface of the pile, and insert to the 1-foot mark.

4. Sample location 2 is located on the south side of the pile at a depth of 2.5 feet below the surface. Lay the 4 ft. 7 in. measuring stick on the side of the pile from the ground surface up, hold the marked probe shaft perpendicular to the side surface of the pile, and insert to the 2.5-foot mark.

5. Sample location 3A is located on the north side of the pile at a depth of 1 foot below the surface. Lay the 4 ft. 7 in. measuring stick on the side of the pile from the ground surface up, hold the marked probe shaft perpendicular to the side surface of the pile, and insert to the 1-foot mark.

6. Sample location 3 is located on the north side of the pile at a depth of 2.5 feet below the surface. Lay the 4 ft. 7 in. measuring stick on the side of the pile from the ground surface up, hold the marked probe shaft perpendicular to the side surface of the pile, and insert to the 2.5-foot mark.

3.0 OXYGEN AND TEMPERATURE MONITORING

This section describes the procedure to be followed for collecting oxygen and temperature measurements within the compost windrows. Procedures for decontamination of monitoring and sampling equipment are described in Field SOP 7.0 in the Full-Scale Operational Plan, Appendix D.

3.1 Tools and Materials

The following tools and materials are required to collect oxygen and temperature measurements:

primary instrument - Demista Instruments Model OT-21 oxygen-temperature monitor
backup instrument (temperature only) - K-type thermocouple with hand-held temperature meter
4 ft 7 in.-long measuring stick
Windrow Process Data - Daily Log form
indelible pen

3.2 Calibration

Demista Oxygen-Temperature Monitor

The Demista oxygen-temperature monitor can be calibrated only for percent oxygen. Temperature calibration shall be performed at the instrument manufacturer's facility. The recommended calibration frequency is minimum once every 12 months based on daily use, however, temperature checks of the probe reading to the ambient temperature will be performed prior to beginning monitoring of each pile.

Oxygen calibration is performed by activating the 'oxygen' mode, aspirating (squeezing and releasing the rubber bulb) several times, and noting the display reading. If the reading is other than 21%, the monitor is adjusted to '21' by turning the oxygen calibration screw with a small screwdriver. Oxygen calibration of the probe will be performed prior to beginning monitoring of each pile. Calibration shall be documented on the Windrow Process Data - Daily Process Log Form (Figure 2 of this SOP).

K-type Thermocouple

Calibration shall be performed at the instrument manufacturer's facility. The recommended calibration frequency is minimum once every 6 months based on daily use.

3.3 Measurement Procedure

The monitoring and sample collection will be performed in the following order:

1. In-situ reading of Oxygen.

2. In-situ reading of Temperature.

3. Sample withdrawn for on-site laboratory analysis of moisture, pH and RDX and TNT analysis.

4. Sample withdrawn for off-site laboratory analysis (Days 0 and 28 or at completion).

Oxygen and temperature measurements at each monitoring location shall be performed prior to any samples being removed. The following procedure shall be used to collect oxygen and temperature measurements with the Demista oxygen-temperature monitor:

1. Post-turn oxygen measurements shall be collected as soon as practical after completion of the turning event. Preferrably within 15 minutes.

2. Prior to the first reading of the day, calibrate the oxygen/temperature probe as described in Section 3.2 above. Measurements will be taken at locations 1A, 1, 2A, 2, 3A, and 3 at each cross-section. Record the information on the Daily Process Log Form (Figure 2 of this SOP) including date, monitoring start time of first measurement, and finish time of last measurement, ambient humidity and temperature, etc..

3. Insert the oxygen/temperature probe into the pile at each of the monitoring locations described in Section 2.2 above. Oxygen measurements should always be made by inserting the probe into an undisturbed location so that the measurements are representative of the settled material, and not biased by void spaces or channels in the pile.

4. Aspirate 4-6 times, or until the reading stabilizes.

5. Activate the "oxygen" mode by pressing down the rear portion 'O' of the control switch on the monitor. Record the oxygen measurement on the Daily Process Log Form (Figure 2 of this SOP).

6. Activate the temperature mode, wait for reading to stabilize, and then record the temperature measurement on the Daily Process Log Form (Figure 2 of this SOP).

7. Decontamination of the probe shall occur at the end of each windrow according to Field SOP 7.0 in the Full-Scale Operational Plan, Appendix D.

Temperature Monitoring with the Thermocouple Probe and Temperature Meter

In the event that the Demista oxygen-temperature probe is not functional, temperature measurements shall be collected using the thermocouple probe and temperature meter. There is no backup method for measuring oxygen level. The following procedure will be used to collect temperature measurements using the thermocouple probe:

1. Connect the probe to the P1 connector on the temperature meter.

2. Press the C key for °C readout. The legend on the LCD display will indicate °C.

3. Press the MODE key for the meter to display the temperature of P1.

4. Press the RESET key to clear the internal memory.

5. Insert the probe to the appropriate depth for the sample location for the windrow being measured, as described in Section 2.2 above.

6. Record the temperature value after the reading stabilizes.

4.0 MOISTURE LEVEL MONITORING

This section describes the procedure to be followed for collecting moisture level monitoring measurements.

4.1 Tools and Materials

The following tools and materials are required to collect moisture level measurements:

stainless steel push tubes with sleeves or split spoon/ hand auger
4 ft 7 in.-long measuring stick
stainless steel bowls (1 per windrow)
sealable disposable plastic bags
drying oven
desiccator
tongs/gloves
pre-labeled weigh boats (aluminum)
analytical balance (+/- 0.1g)
squeeze bottle with distilled water
paper towels/Kimwipes
sample tags
sample labels

sample and instrument transporting device from buildings to the on-site lab
Laboratory Moisture Data Input Form (Figure 3 of this SOP)
indelible pen

4.2 Calibration

The scale shall be calibrated once per week using standard weights in accordance with the manufacturer's Instruction Manual. Calibration shall be documented in the on-site laboratory logbook.

4.3 Sample Collection

Samples for moisture level monitoring will be collected a minimum of three times per week following oxygen/temperature measurement collection. If water is added to the windrow during turning, moisture level measurements will be collected no sooner than 1 hour after moisture is added. Moisture level samples shall be collected as quickly as possible to minimize moisture loss prior to analysis. Moisture level sub-samples will be collected from locations 1, 2, and 3 at each cross-section, using a push tube or hand auger. The three sub-samples will then be composited to form one sample for each cross-section. The following procedure shall be used to collect moisture level monitoring samples at each cross-section:

1. Locate cross-sections 3, 5, 7, 9, and 11, and sample locations 1, 2, and 3 at each cross-section, as described in Section 2.2 above.

2. Rotate the push tube/hand auger into and perpendicular to the pile surface at each sample location. Advance the tube/auger to the designated depth (3 feet for sample location 1, and 2.5 feet for sample locations 2 and 3) or slightly deeper to insure a complete sample at that depth. Using the auger/push tube, remove the material between the surface and the designated depth, if necessary, placing the removed material on top of the pile until the auger has reached the designated depth. Care shall be taken to not collapse the hole or allow material to fall in.

3. Remove the push tube/auger and extract the material in the tube/auger from the appropriate depth location (i.e., the bottom portion of the tube/auger). Place the material from the tube/auger into a stainless steel bowl. Combine the material from sample locations 1, 2, and 3 at a single cross-section in the stainless steel bowl.

4. Thoroughly mix the material in the bowl, then place the composited sample into a new disposable re-sealable plastic bag pre-labeled with the windrow number and

the cross-section letter. Tape or wire tie a tag to the bag identifying the sample identification number, date, time, sampler's name, windrow number and cross-section letter.

5. A new decontaminated stainless steel bowl shall be used for each windrow.

6. Sample tags shall be retained in the field office files as evidence once lab measurements are complete.

4.4 Measurement Procedure

The following procedure will be used to obtain the parameters for calculating moisture levels for each cross-section composite sample. All weights shall be recorded on the Laboratory Moisture Data Input Form (Figure 3 of this SOP). Weights shall be reported to the tenth of a gram.

1. For each cross-section sample, weigh the pre-labeled weigh boat. Record this as tare weight, T1.

2. Place a 10-15 gram sample in the weigh boat and record the gross wet weight, as G_1.

3. Remove the sample and weigh boat from the scale. Slowly wet the sample with distilled water from the squeeze bottle until the sample is saturated and water begins to run off into the weigh boat. Carefully pour off the excess water or absorb it with a paper towel/Kimwipe attempting to not remove any of the solid material.

4. Re-weigh the sample and weigh boat and record this as the gross saturated weight, GS.

5. Dry the sample in the oven at 105 - 110 degrees C for 12 hours or until a constant weight is reached.

6. Place the dried sample in a dessicator for 20 minutes, or until cool, and then record the gross dry weight of the sample and weigh boat as GD_1. Cooling periods for aluminum weigh boats may be very short with small sample sizes. Therefore, use of the dessicator may not be required.

5. Collect all used sample material from a single pile (windrow), after the moisture and pH measurements have been completed, and place in a disposable sealable plastic bag labeled for disposal back onto that same pile where the sample were

collected. Do not mix samples from different piles (windrows).

7. Dispose of contaminated soil bags as contaminated waste, according to NSWC Crane EPD procedures.

4.5 Calculations

The following calculations shall be performed as indicated on the Laboratory Moisture Data Input Form (Figure 3 of this SOP):

PM -Percent Moisture of Original Sample ($[M_{W1} / W_1] \times 100$)
PMS -Percent Moisture at Saturation ($[M_{S1} / S_1] \times 100$)
PS -Percent Saturation (water holding capacity) ([PM / PMS] x 100)
PWHC-Percent of Water Holding Capacity ($[M_{W1} / M_{S1}) \times 100$)
M_W -Mass of Water in Original Sample ($W_1 - D_1$)
M_{S1} - Mass of Water at Saturation ($S_1 - D_1$)
T_1 - Tare Weight of Weigh Boat
G_1 - Gross Weight of Sample and Weigh Boat
W_1 - Net Weight of Sample ($G_1 - T_1$)
GS_1 - Gross Weight of Saturated Sample and Weigh Boat
S_1 - Saturated Weight of Sample ($GS_1 - T_1$)
GD_1 - Gross Dry Weight
D_1 - Dry Weight of Sample ($GD_1 - T_1$)

$$PWHC = \frac{(G_1 - GD_1)}{(GS_1 - GD_1)} \qquad PS = \frac{M_{W1} / W_1}{M_{S1} / S_1} \times 100 \qquad PS = \frac{(W_1 - D_1) / (G_1 - T_1)}{(S_1 - D_1) / (GS_1 - T_1)}$$

Calculation sheets shall be maintained in the Operations office.

4.6 Decontamination

Decontaminate the sampling equipment at the end of sampling each windrow. All sampling equipment must be decontaminated or dedicated and disposable for each windrow as described in Field SOP 7.0 of the Full-Scale Operational Plan. Appendix D.

Decontaminate moisture weighing tools upon completion of the moisture weighing event as described in SOP 7.0 of the Full-Scale Operational Plan, Appendix D.

5.0 pH MONITORING

This section describes the procedure to be followed for collecting pH measurements.

5.1 Tools and Materials Required

The following tools and materials are required to collect pH level measurements:

250 ml glass beaker
100 ml graduated cylinder
stir rod
scale
Hach EC10 portable or Horiba Twin pH meter
~~distilled~~deionized water
Laboratory Moisture Data Input Form (Figure 3 of this SOP)
indelible pen

5.2 Calibration

The pH meter shall be calibrated prior to use in accordance with the manufacturer's Instruction Manual. Calibration shall be documented in the laboratory logbook.

5.3 Sample Collection

pH measurements shall be made no less than once per week on the composite sample material from each cross-section which remains after the moisture level sample portion has been removed from the disposable plastic bag. If it is necessary to collect a separate sample for pH monitoring, the sample locations shall be established as described in Section 2.2 above, and sample material shall be collected with a push tube/hand auger as described in Section 4.3. A new sample bag, tag, and label must then be used.

5.4 Measurement Procedure

The following procedure will be used to measure pH for each cross-section composite sample. All measurements shall be recorded on the pH Data form. Weights shall be reported to the tenth of a gram. pH measurements shall be recorded to the tenth of a pH unit.

1. For each cross-section sample, weigh a 250 ml pre-labeled glass beaker.

2. Place approximately 10 grams of the remainder of the cross-section sample into

5.0 pH MONITORING

This section describes the procedure to be followed for collecting pH measurements.

5.1 Tools and Materials Required

The following tools and materials are required to collect pH level measurements:

250 ml glass beaker
100 ml graduated cylinder
stir rod
scale
Hach EC10 portable or Horiba Twin pH meter
distilled water
Laboratory Moisture Data Input Form (Figure 3 of this SOP)
indelible pen

5.2 Calibration

The pH meter shall be calibrated prior to use in accordance with the manufacturer's Instruction Manual. Calibration shall be documented in the laboratory logbook.

5.3 Sample Collection

pH measurements shall be made no less than once per week on the composite sample material from each cross-section which remains after the moisture level sample portion has been removed from the disposable plastic bag. If it is necessary to collect a separate sample for pH monitoring, the sample locations shall be established as described in Section 2.2 above, and sample material shall be collected with a push tube/hand auger as described in Section 4.3. A new sample bag, tag, and label must then be used.

5.4 Measurement Procedure

The following procedure will be used to measure pH for each cross-section composite sample. All measurements shall be recorded on the pH Data form. Weights shall be reported to the tenth of a gram. pH measurements shall be recorded to the tenth of a pH unit.

1. For each cross-section sample, weigh a 250 ml pre-labeled glass beaker.

2. Place approximately 10 grams of the remainder of the cross-section sample into

the labeled beaker.

3. Remove the sample and beaker from the scale. Measure 50 ml distilled water in the graduated cylinder and add the water to the sample in the beaker.

4. Stir the sample material and distilled water in the beaker with the stir rod for 15 seconds.

5. Allow the sample to sit for one hour or until the solids settle.

6. Immerse the pH electrode in the liquid portion in the beaker. Do not allow the electrode to rest on the bottom of the beaker, but keep the electrode above the solid layer.

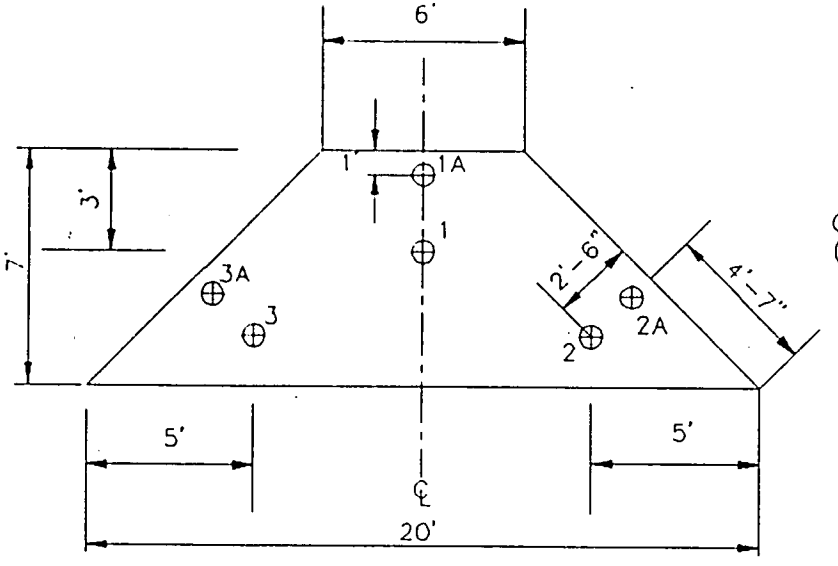
7. Allow the reading on the pH meter to stabilize, then record the pH value on the Laboratory Moisture Data Input Form (Figure 3 of this SOP).

8. Thoroughly rinse the pH probe with distilled water between sample measurements.

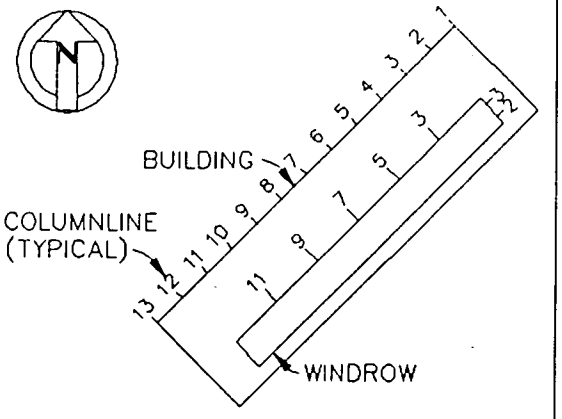
5.5 Decontamination

Decontaminate the sampling equipment at the end of sampling each windrow. All sampling equipment must be decontaminated or dedicated and disposable for each windrow as described in Field SOP 7.0 of the Full-Scale Operational Plan, Appendix D.

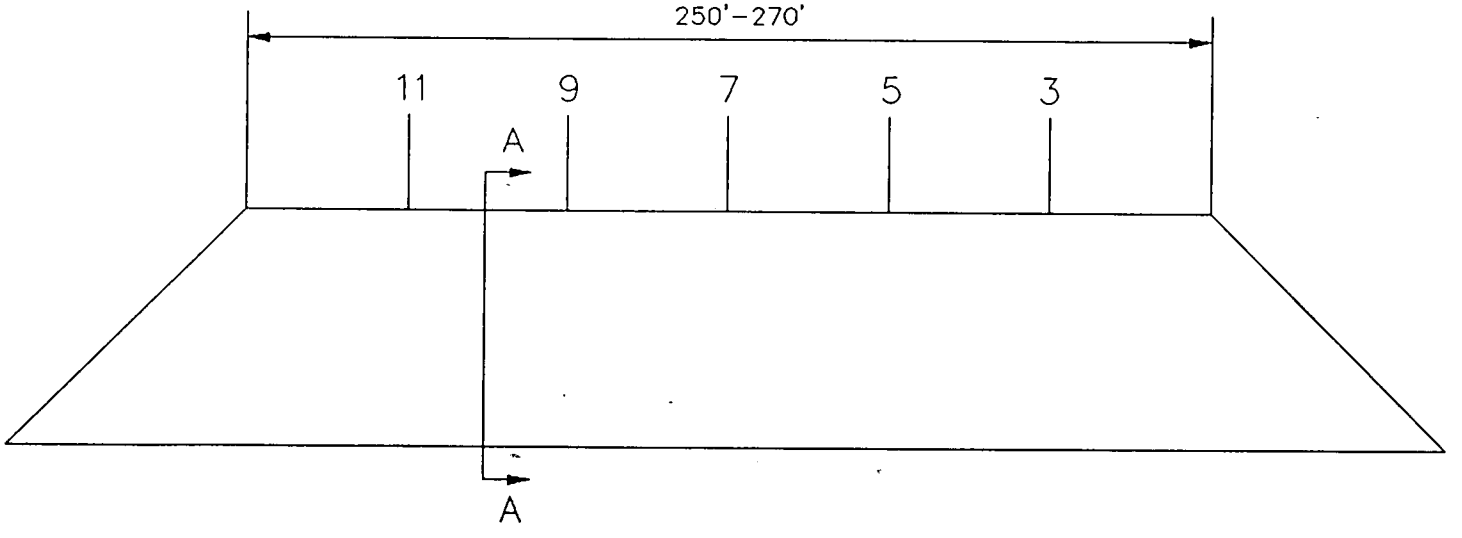
NOTE: ALL SAMPLE NUMBERS FOLLOWED BY AN "A" WILL BE TAKEN AT 1' BELOW THE SURFACE.



SECTION A–A



KEY PLAN



FULL SCALE
WINDROW SAMPLE LOCATIONS

MORRISON KNUDSEN CORP.	
Drawing FIGURE 1	Rev. 0
Date 1/27/98	Sheet 1

FILE: F:\JOKYSER\CRANE\TWOSV.DWG

windro2.xls
Revision No. 2, 03/12/98

QAPP-4.0 Figure 2

Windrow Process Data - Daily Log DATE:

Age (days):	Ambient Conditions	Inspected by:	
Size (feet) :H L W	Relative Humidity: %	Performed by:	
Texture:	Temperature:	Time:	Start Finish
Time Turned:		Pre-Turn	
Moisture added (gal);	**Water Source:**	Post-Turn	

PRE-TURN MONITORING

Cross Section	Sample Location	Temp C		Oxygen (%)		% MWHC	pH
		1' (a)	3' or 2.5' *	1' (a)	3' or 2.5' *		
3	1						
	2						
	3						
5	1						
	2						
	3						
7	1						
	2						
	3						
9	1						
	2						
	3						
11	1						
	2						
	3						
Duplicates							

POST-TURN MONITORING

Cross Section	Sample Location	Temp C		Oxygen (%)		% MWHC
		1' (a)	3' or 2.5' *	1' (a)	3' or 2.5' *	
3	1					
	2					
	3					
5	1					
	2					
	3					
7	1					
	2					
	3					
9	1					
	2					
	3					
11	1					
	2					
	3					
Duplicates						

Monitor Calibration: Pre: % C Post: %

Description of windrow

Comments:

Note: * denotes 3' depth at sample location 1 and 2.5' depth at locations 2 and 3

MOICAL2.XLS
Revision No. 1, 03/04/98

QAPP-4.0 Figure 3
Laboratory Moisture Data Form

Date: 3-Oct

Bldg/Win	Cross Section	T_1	G_1	W_1	GS_1	S_1	GD_1	D_1	M_{W1}	M_{S1}	PM	PMS	PS	PWHC	pH	Day #
N/001	3			-		-		-	-	-	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!		0 pre post
	5			-		-		-	-	-	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!		
	7			-		-		-	-	-	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!		
	9			-		-		-	-	-	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!		
	11			-		-		-	-	-	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!		
Dup																
N/002	3			-		-		-	-	-	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!		0 pre post
	5			-		-		-	-	-	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!		
	7			-		-		-	-	-	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!		
	9			-		-		-	-	-	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!		
	11			-		-		-	-	-	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!		
Dup																
M/001	3			-		-		-	-	-	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!		0 pre post
	5			-		-		-	-	-	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!		
	7			-		-		-	-	-	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!		
	9			-		-		-	-	-	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!		
	11			-		-		-	-	-	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!		
Dup																
M/002	3			-		-		-	-	-	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!		0 pre post
	5			-		-		-	-	-	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!		
	7			-		-		-	-	-	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!		
	9			-		-		-	-	-	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!		
	11			-		-		-	-	-	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!		
Dup																
S/001	3			-		-		-	-	-	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!		0 pre post
	5			-		-		-	-	-	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!		
	7			-		-		-	-	-	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!		
	9			-		-		-	-	-	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!		
	11			-		-		-	-	-	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!		
Dup																
S/002	3			-		-		-	-	-	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!		0 pre post
	5			-		-		-	-	-	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!		
	7			-		-		-	-	-	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!		
	9			-		-		-	-	-	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!		
	11			-		-		-	-	-	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!		
Dup																

MOICAL2.XLS
Revision No. 1, 03/04/98

QAPP-4.0 Figure 3
Laboratory Moisture Data and pH Input Form

Date: 3-Oct

Bldg/Win	Cross Section	T_1 (weight of weigh boat)	G_1 (gross weight of sample & weigh boat)	GS_1 (gross weight of saturated sample & weigh boat)	GD_1 (gross dry weight)	pH sample weight	pH	Day#
N/001	3							0 pre post
	5							
	7							
	9							
	11							
Dup								
N/002	3							0 pre post
	5							
	7							
	9							
	11							
Dup								
M/001	3							0 pre post
	5							
	7							
	9							
	11							
Dup								
M/002	3							0 pre post
	5							
	7							
	9							
	11							
Dup								
S/001	3							0 pre post
	5							
	7							
	9							
	11							
Dup								
S/002	3							0 pre post
	5							
	7							
	9							
	11							
Dup								

MOIDEL1.XLS
Revision No. 1, 03/04/98

QAPP-4.0 Figure 3
Full Scale Moisture Deltas

Date								Ave. PWCH
13-Oct	Water Added							-
	PWHC							
	Delta PWHC							
14-Oct	Water Added							
	PWHC							
	Delta PWHC							
15-Oct	Water Added							
	PWHC							
	Delta PWHC							
16-Oct	Water Added							
	PWHC							
	Delta PWHC							
17-Oct	Water Added							
	PWHC							
	Delta PWHC							
18-Oct	Water Added							
	PWHC							
	Delta PWHC							
19-Oct	Water Added							
	PWHC							
	Delta PWHC							
20-Oct	Water Added							
	PWHC							
	Delta PWHC							
21-Oct	Water Added							
	PWHC							
	Delta PWHC							
22-Oct	Water Added							
	PWHC							
	Delta PWHC							
23-Oct	Water Added							
	PWHC							
	Delta PWHC							

FIELD SOP: QAPP-5.0
TITLE: FIELD TESTING OF SOIL AND COMPOST FOR EXPLOSIVES

1.0 PURPOSE

This procedure describes materials and methods used to determine explosives contamination in soil and compost using field analytical methods.

2.0 RELATIONSHIP TO OTHER PROCEDURES

Separate procedures have already been approved for various aspects of this task. These include sample collection and general requirements of this Quality Assurance Project Plan (QAPP).

3.0 MATERIALS

The field screening methods are based on a commercially available test kits supplied by Strategic Diagnostics Inc. (SDI) of Delaware. There are two basic versions of the test kits. One format is suited to the analysis to TNT related nitroaromatics. The other format is suited to the analysis of RDX and HMX (nitramines). Additional reagents and materials are required to remove color introduced from compost derived samples. These materials have been identified by SDI, and are available from other suppliers. The SDI test kits will be used by the site personnel to test soil and compost. These kits contain consumable reagents and disposable hardware which are designed to be used with dedicated hardware previously purchased from SDI.

The hardware includes the Hach spectro-photometer, scale, pipettes, cuvettes and assorted glassware and plasticware. Additional equipment includes sample collection hardware and other material located within the on-site laboratory.

4.0 SAMPLE COLLECTION FREQUENCY

Samples will be collected throughout the full-scale operations based on a schedule given in Table ~~1-14~~ 2 of ~~SOPs QAPP-1.0 and QAPP-3.0QAPP-4.0~~ this QAPP.

5.0 ANALYTICAL METHOD

Each sample for field screening purposes is defined as a 20 gram fraction of soil and/or compost ~~that has been air dried to a constant weight~~. Analysis of samples and QC should be performed according to manufacturer's recommendations (See Attachment A). A general summary of the procedures is provided below.

5.1 Field Analysis of TNT in Soil

1. Clean Cuvettes and Zero the Spectrophotometer at 540 nm. Record

spectrophotometer setting.

2. Verify QC limits on field kit have been met. Perform a QA/QC check sample analysis and method blank.

3. Add 20 grams of soil and 100 mL of acetone to extraction jar. Shake for 3 minutes and let settle. Draw up 25 mL extract and filter into cuvette.

4. Read $Abs_{initial}$ and record the value. Add 1 drop of developer solution and shake. Read Abs_{sample} and record the value

5. Multiply $Abs_{initial}$ by 4, subtract from Abs_{sample}, and divide by 0.0323, i.e.,

$$TNT_{[ppm]} = \frac{Abs_{sample} - (Abs_{initial} \times 4)}{0.0323}$$

This information is taken from the Strategic Diagnostics Inc. user's guide *TNT SOIL TEST SYSTEM* which is incorporated into this SOP as Attachment A. The test is designed to provide results over the range of concentrations from 1-30 ppm. If initial concentrations are believed to be above this range, extraction solutions will require dilution. The appendix contains the guidelines for preparing standard dilutions of 1:10, 1:100, and 1:1000. These dilutions may be necessary to bring the absorbance reading into the preferred range of 0.3 to 1.3 at 540 nm.

The test will detect other nitroaromatics such as DNT, monoamino-DNT and diamino-nitrotoluene—therefore results obtained from field test kits will be utilized only to monitor the end of cycle of the compost piles. End of cycle confirmation analysis will be performed by an off-site laboratory.

5.2 Field Analysis of RDX in Soil

1. Clean cuvettes and zero the Spectrophotometer at 510 nm. Record spectrophotometer setting.

2. Verify QC limits on field kit have been met. Perform a QA/QC check sample analysis and method blank.

~~2~~3. Add 20 grams of soil and 100 mL of acetone to the extraction jar, shake for 3 minutes and let settle, or use surplus TNT extract previously generated.

~~3~~4. Draw up 5.5 mL extract and filter into a 13 mL tube (see Section 5.3 for compost samples). Open the Bulb pipet, add Acetic Acid to the 13 mL tube and mix.

~~4~~5. Add NitriVer to a 50 mL Reaction Vial.

~~5~~6. Pour the extract from the 13 mL tube into the Zinc Syringe (hydrogen evolution). Invert 2 times and filter into the 50 mL Reaction Vial. Shake for 30 seconds.

~~6~~7. Let stand for 15 minutes to develop color.

~~7~~8. Read Abs at 510 nm and calculate the RDX concentration:

$$RDX_{[ppm]} = (Abs - 0.014/ 0.0225)$$

This information is taken from the Strategic Diagnostics Inc. user's guide *RDX SOIL TEST SYSTEM* which is incorporated into this SOP as Attachment A. The test is designed to provide results over the range of concentrations from 1-30 ppm. If initial concentrations are believed to be above this range, extraction solutions will require dilution. The appendix contains the guidelines for preparing standard dilutions of 1:10, 1:100, and 1:1000. These dilutions may be necessary to bring the absorbance reading into the preferred range of 0.2 and 0.7 at 510 nm.

5.3 Analysis of Compost Samples

The acetone used to extract the explosive compounds from the compost matrix may also remove other colored substances associated with plant material. This is especially true if alfalfa is used as part of the amendment recipe. Therefore, a color removal step must be added to both of the above procedures to obtain meaningful results.

SDI has produced separate procedures for TNT and RDX testing. The procedures are highlighted below. The TNT compost-protocol incorporates a 5-fold dilution of the extract into water. This essentially makes the test five-fold less sensitive, thus the 1 ppm minimum test level becomes 5 ppm, and the maximum test level becomes 150 ppm. This does not hold true for RDX, because it does not get diluted with water prior to analysis.

The initial preparation and extraction of samples can be combined for the analysis of TNT and RDX. Test kits can be ordered to avoid buying extra extraction supplies when the RDX and TNT analysis is performed on the same sample. An assumption has been made that NSWC Crane soils do not contain ammonium picrate or picric acid. These explosives can cause interference with test procedure.

5.3.1 Procedure for TNT Analysis with Compost Interference Removal

1. Extract 20 grams of sample with 100 mL of acetone. Place soil in

extraction bottle with acetone and shake for 3 minutes.

2. Using a 30 cc syringe, carefully draw up 15 mL of water (tap or distilled) then 5 mL of extract, draw 2 to 3 mL of air into the syringe and attach a syringe filter. Invert the syringe back and forth to mix.

3. Load Column with TNT. Place a column into the vacuum apparatus (see Attachment A) and prime by drawing 15 mL of acetone and subsequently, 30 mL of water through the column. (Do not allow the column to run dry between additions of acetone, water and sample).

4. Filter the sample extract from the syringe into the column and allow to be drawn through.

5. Remove Interference from Column. Wash the column with 15 mL of 25% acetone/75% water solution (made by adding 25 mL of acetone and 75 mL of water to a 100 mL graduated cylinder and mixing). Allow to run through until column is totally devoid of liquid.

6. Desorb TNT. Remove column from the vacuum apparatus and attach the column cap (see Attachment A). Remove the plunger from a 30 cc syringe, attach the syringe to the column cap and add 25 mL of acetone to the syringe. Replace the plunger and depress to elute the TNT from the column. Collect the eluant in the cuvette.

7. Read the initial absorbance of the eluant.

8. Add 1 drop of developer solution, cap, mix, and read the final absorbance of the eluant.

9. Use the following equation to determine the TNT concentration of the samples.

$$TNT_{[ppm]} = \frac{Abs_{final} - 2(Abs_{initial})}{0.0323} \times 5$$

5.3.2 Procedure for RDX Analysis with Compost Interference Removal

Use carbon column to remove organic matrix interference, then run according to procedure in Section 5.2. The test will measure both RDX and HMX, but does not have cross-reactivity to TNT.

1. Fit Alltech carbon column into the 13 mL snap-cap tube.

2. Using a 10 cc syringe, draw up 6.5 mL of sample extract.

3. Attach syringe filter.

4. Filter extract into carbon column until entire 6.5 mL is dispensed into the 13 mL tube (approximately 5 mL remains).

5. Remove and dispose of column.

6. Proceed with test as outlined in Section 5.2

7. Final concentration correction. Multiply result obtained with standard kit equation by 1.1. This will give a corrected value for the RDX concentration of the sample. If the sample concentration is greater than 30 ppm (e.g. Absorbance reading greater than 0.7), dilute extract with acetone and rerun.

6.0 WASTE DISPOSAL

Handle all disposable test materials, sample residues, and PPE according to State, Federal and Navy guidelines. Acetone is a RCRA-Listed solvent and will require proper disposal.

7.0 QUALITY ASSURANCE

The QC requirements for the laboratory samples have been defined in Section 4.0 of this QAPP. In addition the manufacture has defined specific parameters for blank samples, standards, and for absorbance within an operating range as specified in Table 3.8 of this QAPP. These parameters must be followed to produce valid test results.

Attachment A
~~SC-~~SDI User's guides

ATTACHMENT A
SDI User's guides

ENSYS INC.
ENVIRONMENTAL PRODUCTS

RDX SOIL TEST SYSTEM

User's Guide

IMPORTANT NOTICE

The range of the test is between 1 and 30 ppm RDX/HMX. The relative standard deviation is 10%. The least detectable concentration is 0.8 ppm.

This test system should be used only under the supervision of a technically qualified individual who is capable of understanding any potential health and environmental risks of this product as identified in the product literature. The components must only be used for the analysis of soil samples for the presence of RDX/HMX. After use, the kits must be disposed of in accordance with applicable federal and local regulations.

PHASE 1

TEST PREPARATION

READ ALL INSTRUCTIONS BEFORE PROCEEDING WITH THE TEST

ITEMS INCLUDED IN TEST KIT WITH EXTRACTION SET-UPS

- ☐ 2 Cuvette Stopper Plugs
- ☐ 1 Ampule Cracker
- ☐ 40 Syringe Filters
- ☐ 20 Wooden Spatulas
- ☐ 20 10cc Syringe
- ☐ 20 50mL Reaction Vials w/ H_2O
- ☐ 20 Extraction Jars
- ☐ 1 Bulb Pipette
- ☐ 1 50mL Conical Tube
- ☐ 20 5cc Zinc syringes
- ☐ 20 12mL Tubes
- ☐ 20 Acetic Acid Bulb Pipets
- ☐ 1 RDX Control Ampule
- ☐ 20 30 cc Syringes
- ☐ 20 Weigh Boats
- ☐ 20 Filter Pillows

* Your kit will not contain wooden spatulas, extraction jars or weigh boats if it was purchased to use in conjunction with the TNT Soil Test.

ITEMS NOT INCLUDED IN TEST KIT

- ☐ 2 matched HACH cuvettes
- ☐ Paper towels
- ☐ Disposable gloves
- ☐ Acetone
- ☐ Calculator
- ☐ Scissors
- ☐ Waste container
- ☐ Hach DR/2000
- ☐ Balance

READ BEFORE PROCEEDING

- Recovery of the RDX from some soil samples is most consistent when the soil samples are air dried prior to extraction and testing.
- It is recommended that a control be run each day. See p.8 for instructions.
- Nitrates and Nitrites cause false positive results with the RDX test. Therefore, it is necessary to evaluate the soil for these compounds prior to sample analysis. See p.9 for instructions.
- The Hach DR/2000 is designed to turn off after a few minutes of inactivity. Press the "READ/ENTER" key every few minutes to prevent DR/2000 from turning off. If DR/2000 turns off, use Reference cuvette to rezero. Newer DR/2000 models have an override "constant on" feature that allows the machine to run indefinitely. See p.12 of HACH DR/2000 User's manual.
- If you are using the RDX soil test kit in conjunction with the TNT soil test kit, the sample extract generated with the TNT test may be used for the RDX test. (Skip steps 2a - 3e of the RDX test if this scenario applies.) An RDX kit without extraction set-ups can be provided specifically for this purpose.

PHASE 1

TEST PREPARATION

READ ALL INSTRUCTIONS BEFORE PROCEEDING WITH THE TEST

CLEAN CUVETTES

1a Fill 2 HACH matched cuvettes with approximately 5 mL water.

1b Cap each with cuvette stopper plug and, holding plug in place, shake.

1c Empty into waste container.

1d Fill cuvettes with approximately 5 mL acetone.

1e Cap each with cuvette stopper plug and, holding plug in place, shake.

1f Empty into waste container.

1g Repeat acetone wash (steps **1d** - **1f**).

1h Wipe outside of cuvette with paper towels. Take care to especially clean the side labeled "25 mL" and the side opposite.

Cuvette

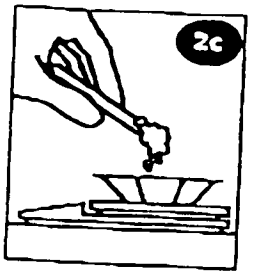
Cuvette stopper

PHASE 2

EXTRACTION & PREPARATION OF THE SAMPLE

READ ALL INSTRUCTIONS BEFORE PROCEEDING WITH THE TEST

WEIGH SAMPLE

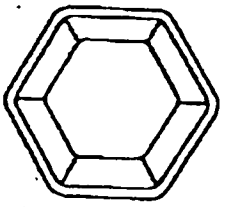


2a Place an unused weigh boat on pan balance.

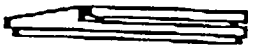
2b Press ON/MEMORY button on pan balance. Balance will beep and display 0.0.

2c Weigh out 10+/-0.1 grams of soil.

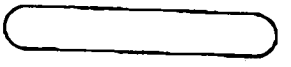
2d If balance turns off prior to completing weighing, use empty weigh boat to retare, then continue.



Weigh Boat

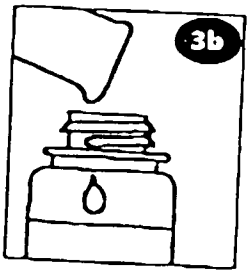


Pan balance



Wooden spatula

EXTRACT RDX



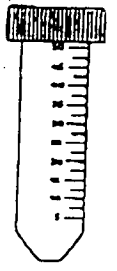
3a Measure 50mL acetone in the 50mL graduated conical tube.

3b Pour acetone into the extraction jar.

3c Using wooden spatula, transfer 10 grams of soil from weigh boat into extraction jar.

3d Recap extraction jar tightly and shake vigorously for three minutes.

3e Allow to settle for five minutes.



50mL Conical Tube

Extraction Jar

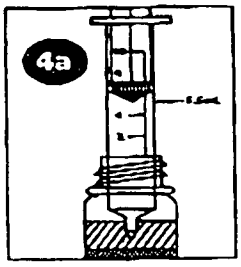
PHASE 3

SAMPLE ANALYSIS

READ ALL INSTRUCTIONS BEFORE PROCEEDING WITH THE TEST

If nitrates/nitrites are present, follow instruction in bold type, if not, ignore.

ANALYZE SAMPLE



4a Using the 10cc syringe slowly draw up exactly 5.5mL of sample extract being careful to exclude air bubbles. **(8-10mL if nitrate/nitrite interferents are present)**

4b **(If nitrate/ nitrite interferents are present, attach Alumina-A cartridge to syringe filter discarding single drops of filtrate into a waste container until 5 mL of extract remain. Dropwise, add the remaining 5 mL of filtrate to the 13 mL tube.)** Attach the syringe filter securely to the syringe and dispense into 13mL tube. Cut open tip of Acetic Acid bulb pipet and expel contents into 13mL tube. Cap & shake. Repeat steps **4a - 4b** for remaining samples.

4c Cut open one end of a NitriVer pillow and pour it into a 50mL Reaction Vial containing water. Prepare a vial for each sample. (Do not let the NitriVer powder/water solution stand longer than 10 minutes before adding sample.)

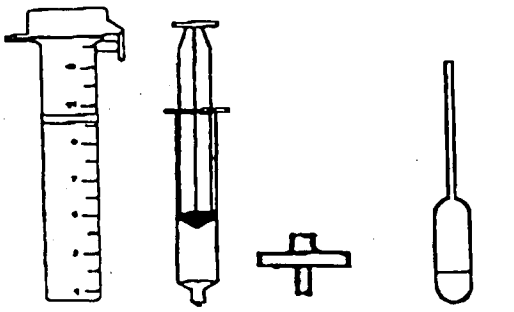
4d Remove plunger from 5cc zinc syringe and quickly pour the solution from the 13mL tube into the syringe barrel. Hold syringe over Reaction Vial as dripping will occur.

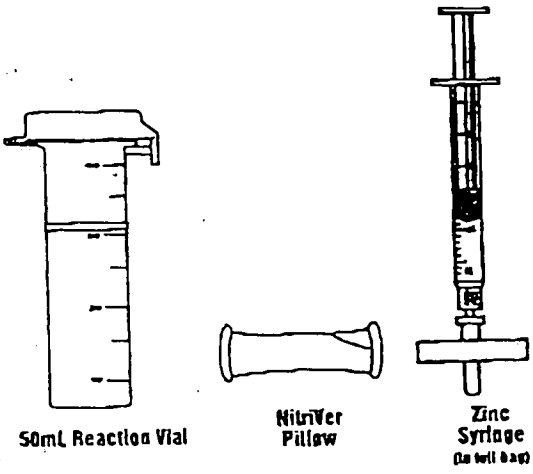
4e Replace the plunger & invert twice.

4f Rapidly filter the solution into the 50mL Reaction Vial. Cap and shake for 30 seconds. Repeat **4d - 4f** for remaining samples.

4g Allow this reaction to incubate for 15 minutes while color develops.

4h Proceed to page 6 during incubation.





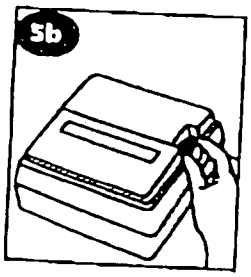
PHASE 4
INTERPRETATION

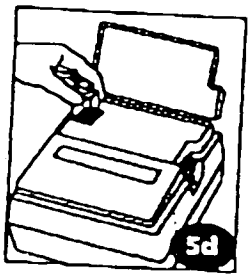
READ ALL INSTRUCTIONS BEFORE PROCEEDING WITH THE TEST

READ BEFORE PROCEEDING

- Designate a "Reference" and "Sample" cuvette.
- Prepare DR/2000 during incubation step of sample analysis.

SPECTROPHOTOMETER PREPARATION





5a Turn on HACH DR/2000. The instrument will read "SELF-TEST" followed by "Method?". Select Method "0" and press the "READ/ENTER" key.

5b Rotate the wavelength dial until the small display shows: 510 nm.

5c Fill both cuvettes with acetone to the 25 mL line.

5d Insert "Reference" cuvette into cell holder on HACH DR/2000 with side marked "25 mL" on the right.

5e Close light shield and press "CLEAR/ZERO" key to establish the reference. The display will read "WAIT" and then "0.000 Abs.".

5f Remove the "Reference" cuvette and place the "Sample" cuvette in the cell holder.

5g Press the "READ/ENTER" key and record the absorbance on the worksheet as "$Abs_{background}$".

5h If reading is greater than 0.002 in magnitude (+ or -), clean cuvettes and redo steps **5a - 5g**.

5i Empty acetone from "Sample" cuvette into waste container.

Cuvette

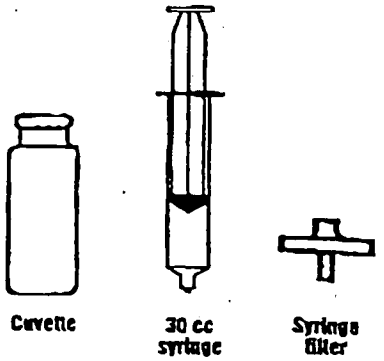
PHASE 4
INTERPRETATION

READ ALL INSTRUCTIONS BEFORE PROCEEDING WITH THE TEST

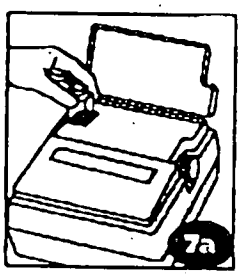
FILTER SAMPLE

6a Disassemble a 30cc syringe and attach a syringe filter.

6b After incubation, shake reacted sample vigorously and pour into barrel of 30cc syringe. Insert plunger. Press firmly and expel total contents into the HACH cuvette.



READ SAMPLE



7a Place the "Sample" cuvette in the cell holder.

7b Press the "READ/ENTER" key and record the absorbance on the worksheet.

7c Clean cuvette between samples using procedure in steps **1a - 1h**.

INTERPRETATION OF RESULTS

8a Subtract 0.014 value from the sample absorbance values

8b Divide this value by 0.0225 and record on the worksheet. This value is the RDX concentration of the sample in parts per million.

$$[RDX]\ (ppm) = \frac{Abs - 0.014}{0.0225}$$

Note: For sample concentrations greater than 30ppm the sample extract should be diluted with acetone and reanalyzed.

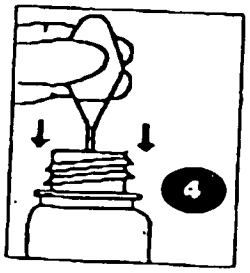
Remember to multiply the result by the dilution factor in order to determine the correct concentration.

CONTROL (QA/QC) CHECK

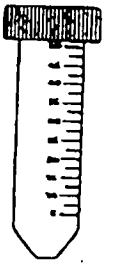
READ ALL INSTRUCTIONS BEFORE PROCEEDING WITH THE TEST

- The RDX control is optional but it is recommended that it be run daily.

PREPARE CONTROL



1. Measure 50 mL acetone in a graduated 50mL conical tube.
2. Pour into extraction jar.
3. Open RDX control ampule by slipping ampule cracker over top, and then breaking tip at scored neck.
4. Transfer entire contents of RDX control ampule into extraction jar using empty bulb pipette.
5. Cap extraction jar and shake.


50mL Conical Tube

Extraction Jar

RDX

Bulb pipette

Ampule cracker

ANALYZE THE CONTROL

Repeat steps **4a - 7c** on pages 5 - 7

Record the absorbance on the worksheet as "$Abs_{control}$".

Absorbance must be between 0.174 - 0.274 for the test to be in control.

If test is not in control, clean "Sample" cuvette, and then redo steps **4a- 7c** using the remaining liquid in the extraction jar.

If test is in control clean "Sample" cuvette before proceeding with samples.

If kept tightly capped, the control can be used again for additional QC runs.

BACKGROUND - NITRATE/NITRITES TEST

READ ALL INSTRUCTIONS BEFORE PROCEEDING WITH THE TEST

- Site representative samples must be run prior to analysis for RDX to ensure that Nitrate/Nitrite interferents (i.e., fertilizers, degraded explosives, etc.) are not present. Please call Technical Services at (919) 941-5509.
- These interferents cause a color reaction with the test identical to RDX and will lead to false positives.
- If Nitrates/Nitrites are present, a kit containing alumina–a cartridges must be purchased from EnSys. These will quickly & easily remove the interferents from the soil extract during the extract filtration steps.

READ BEFORE PROCEEDING

- Sample should be mixed to ensure a homogeneous sample.

1) Repeat steps **2a - 4c** on page 4 & 5.

2) Omit steps **4d - 4e***

   * Zinc syringe is not used when testing for Nitrates/Nitrites.

3) Proceed with steps **4f - 7c**

   Record the absorbance on the worksheet as "Abs Nitrate/Nitrite".

   If the absorbance is <0.05, the samples are free of Nitrates/Nitrites and the samples can be tested.

   If absorbance is > 0.05, then Alumina–A cartridges must be purchased from EnSys to remove nitrate/nitrite interferents.

QUALITY CONTROL

READ ALL INSTRUCTIONS BEFORE PROCEEDING WITH THE TEST

System Description

Each RDX Soil Test System contains enough material to perform 20 complete tests.

The RDX Soil Test is divided into four phases. The instructions and notes should be reviewed before proceeding with the test.

Hotline Assistance

If you need assistance or are missing necessary Test System materials, call toll free: 1-800-242-RISC (7472).

Validation and Warranty Information

Product claims are based on validation studies carried out under controlled conditions. Data has been collected in accordance with valid statistical methods and the product has undergone quality control tests of each manufactured lot.

The company does not guarantee that the results with the RDX Soil Test System will always agree with instrument-based analytical laboratory methods. All analytical methods, both field and laboratory, need to be subject to the appropriate quality control procedures.

EnSys, Inc. warrants that this product conforms to the descriptions contained herein. No other warranties, whether expressed or implied, including warranties of merchantability and of fitness for a particular purpose shall apply to this product.

EnSys, Inc. neither assumes nor authorizes any representative or other person to assume for it any obligation or liability other than such as is expressly set forth herein.

Under no circumstances shall EnSys, Inc. be liable for incidental or consequential damages resulting from the use or handling of this product.

How It Works

Controls, Samples, and color-change reagents are added to cuvettes. The concentration of RDX in an unknown Sample is determined by evaluating how much color is developed.

Quality Control

Standard precautions for maintaining quality control:

- Do not use reagents or components from one Test System with reagents or components from another Test System.
- Do not use the Test System after its expiration date.
- The sample must be analyzed within 60 minutes of the color incubation step.
- Results may not be valid if DR/2000 reading for Control is outside of the range of 0.174 - 0.274.

Storage and Handling Precautions

- Wear protective gloves and eye wear.
- Store kit at room temperature and out of direct sunlight (less than 80°F).
- If acetone comes into contact with eyes, wash thoroughly with cold water and seek immediate medical attention.
- Operate test at temperatures greater than 4° C/40° F and less than 39° C/100° F.
- After use, dispose of kit components in accordance with applicable federal and local regulations.

ON-SITE QUALITY CONTROL/QUALITY ASSURANCE RECOMMENDATIONS
EnSys RISc® TEST SYSTEM

Please read the following before proceeding with field testing.

SAMPLING

The result of your screening test is only as valid as the sample that was analyzed. Samples should be homogenized thoroughly to ensure that the 10 grams you remove for field testing is representative of the sample as a whole. All other applicable sample handling procedures should be followed as well.

PRIOR TO TESTING SAMPLES

Carefully follow the instructions in the User's Guide included with every test kit. This is the key element in obtaining accurate results. In addition, store your unused test kits at room temperature and do not use them past their expiration date (see label on each test kit).

INTERNAL TEST QC

One control is provided with each Kit to provide internal test system quality control. Test runs resulting in a number that falls outside of the specified range should be repeated to ensure valid conclusions.

QA/QC

The validity of field test results can be substantially enhanced by employing a modest, but effective QA/QC plan. EnSys recommends that you structure your QA/QC plan with the elements detailed below. These have been developed based on the data quality principles established by the U.S. Environmental Protection Agency.

A. Sample Documentation
   1. Location, depth
   2. Time and date of collection and field analysis

B. **Field analysis documentation** - provide raw data, calibration, any calculations, and final results of field analysis for all samples screened (including QC samples)

C. **Method calibration** - this is an integral part of EnSys tests; an RDX control analysis should be performed daily (see the instructions in the User's Guide)

D. **Method blank** - field analyze fresh acetone

E. **Site-specific matrix background field analysis** - collect and field analyze uncontaminated sample from site matrix to document matrix effect

F. **Duplicate sample field analysis** - field analyze duplicate sample to document method repeatability; at least one of every 20 samples should be analyzed in duplicate

G. **Confirmation of field analysis** - provide confirmation of the quantitation of the analyte via an EPA-approved method different from the field method on at least 10% of the samples; provide chain of custody and documentation such as gas chromatograms, mass spectra, etc.

H. **Performance evaluation sample field analysis (optional, but strongly recommended)** - field analyze performance evaluation sample daily to document method/operator performance

I. **Matrix spike field analysis (optional)** - field analyze matrix spike to document matrix effect on analyte measurement

J. **Nitrate/Nitrite test** - this is an integral part of the EnSys RDX Test; it should be performed at least once for each site.

FURTHER QUESTIONS?

EnSys technical support personnel are always prepared to discuss your quality needs to help you meet your data quality objectives. (919)941-5509 (OPTION 4)

RDX Soil Test - Abbreviated Procedure

1	• Clean cuvettes • Zero the spectrophotometer at 510 nm
2	• Add 10 g soil and 50mL acetone to extraction jar • Shake 3 min., let settle
3	• Draw up 5.5 mL extract, filter into 13 mL tube **(If NO3/NO2 contaminants present: 8-10 mL of extract, filtered slowly through Alumina-A cartridge)** • Open bulb pipet, add Acetic Acid to 13 mL tube, mix • Add NitriVer to 50 mL Reaction Vial • Pour from 13 mL Tube into zinc syringe • Invert 2X and filter into 50 mL Reaction Vial • Shake 30 seconds • Incubate 15 minutes
4	• Read Abs at 510 • Calculate RDX concentration • [RDX]ppm = (Abs-0.014)/0.0225

RDX SOIL TEST KIT WORKSHEET

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

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

STRATEGIC DIAGNOSTICS INC.

TNT EnSys® SOIL TEST SYSTEM

RAPID FIELD SCREEN

User's Guide

IMPORTANT NOTICE

The range of this test is between 1 and 30 ppm TNT/TNB/DNT. The relative standard deviation is 8% The least detectable concentration is 0.7 ppm (TNT).

This test system should be used only under the supervision of a technically qualified individual who is capable of understanding any potential health and environmental risks of this product as identified in the product literature. The components must only be used for the analysis of soil samples for the presence of TNT. After use, the kits must be disposed of in accordance with applicable federal and local regulations.

PHASE 1 TEST PREPARATION

READ ALL INSTRUCTIONS BEFORE PROCEEDING WITH THE TEST

ITEMS INCLUDED IN TEST KIT

- ❑ 2 Cuvette stopper plugs
- ❑ 1 Ampoule cracker
- ❑ 20 Syringe filters
- ❑ 20 Wooden spatulas
- ❑ 20 Extraction jars
- ❑ 1 Bulb pipette
- ❑ 1 Developer solution
- ❑ 1 - 50mL graduated conical tube
- ❑ 1 TNT control ampoule
- ❑ 20 - 30cc syringes
- ❑ 20 Weigh boats

ITEMS NOT INCLUDED IN TEST KIT

- ❑ 2 matched HACH cuvettes
- ❑ Paper towels
- ❑ Disposable gloves
- ❑ Acetone
- ❑ Hach DR/2000 or DR/2010
- ❑ Calculator
- ❑ Waste container
- ❑ Balance

READ BEFORE PROCEEDING

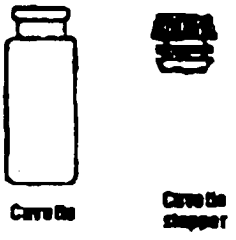
- For some matrices, air drying the soil samples may result in better TNT recovery or more reproducible data.
- A slightly modified protocol should be used if the primary analyte of concern is DNT. Please refer to the modification outlined on page 6.
- It is recommended that a control be run each day. See page 8 for instructions.
- SDI's EnSys® TNT Soil Test System is designed for use with either of Hach models DR/2000 or the newer DR/2010 spectrophotometers. Protocols for use of both instruments are provided in this User's Guide. Ensure the instrument protocol followed is appropriate for the instrument being used.
- The Hach DR/2000 is designed to turn off after a few minutes of inactivity. Press the "READ/ENTER" key every few minutes to prevent DR/2000 from turning off. If DR/2000 turns off, use Reference cuvette to rezero. Newer DR/2000 models and the DR/2010 have an overide "constant on" feature that allows the machine to run indefinitely. Refer to the Instrument Operation: Spectrophotometer Setup section of the HACH DR/2000 or DR/2010 User's manuals.

If you are using the TNT test in conjunction with the RDX test it is important to save your sample extracts. They will be used in the RDX test. Remember to cap the extracts tightly after use. An RDX kit without extraction set-ups can be purchased specifically for this purpose.

PHASE 1 TEST PREPARATION

READ ALL INSTRUCTIONS BEFORE PROCEEDING WITH THE TEST

CLEAN CUVETTES



- **1a** Fill 2 Hach matched cuvettes with approximately 5 mL water.
- **1b** Cap each with cuvette stopper plug and, holding plug in place, shake vigorously for 3 seconds.
- **1c** Empty into waste container.
- **1d** Fill cuvettes with approximately 5 mL acetone.
- **1e** Cap each with cuvette stopper plug and, holding plug in place, shake vigorously for 3 seconds.
- **1f** Empty into waste container.
- **1g** Repeat acetone wash (steps 1d - 1f).
- **1h** Wipe outside of cuvette with paper towels. Take care to especially clean the side labeled "25 mL" and the side opposite.

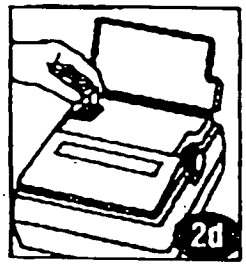
PHASE 1 TEST PREPARATION

READ ALL INSTRUCTIONS BEFORE PROCEEDING WITH THE TEST

READ BEFORE PROCEEDING

- Designate a "Reference" and "Sample" cuvette.

SPECTROPHOTOMETER PREPARATION



2a1 Turn on Hach **DR/2000**. The instrument will read "SELF-TEST" followed by "Method?". Select Method "0" and press the "READ/ENTER" key.

or

2a2 Turn on the Hach **DR/2010**. The instrument will read "Self-Test V.xx", then "Enter Program #". Press the [Shift] key (do not hold) and then the [ABS/8] key. Note: Select Program # "0" may also be used to select absorbance mode on the **DR/2010**.

2b Rotate the wavelength dial until the small display shows: 540 nm.

2c Fill both cuvettes with acetone to the 25 mL line.

2d Insert "Reference" cuvette into cell holder on Hach DR/2000 or DR/2010 with side marked "25 mL" on the right.

Cuvette

2e1 Close light shield of the **DR/2000** and press "CLEAR/ZERO" key to establish the reference. The display will read "WAIT" and then "0.000 Abs.".

or

2e2 Close the light shield of the **DR/2010** and press the [ZERO] key. The display will read "Zeroing..." then "0.000 Abs.".

2f Remove the "Reference" cuvette and place the "Sample" cuvette in the cell holder.

2g1 On the **DR/2000**, press the "READ/ENTER" key and record the absorbance on the worksheet as "$Abs_{background}$".

or

2g2 On the **DR/2010**, press the [READ] key and record the absorbance on the worksheet as "$Abs_{background}$".

2h If reading is greater than 0.002 in magnitude (+ or -), clean cuvettes and redo steps 2a - 2g.

2i Empty acetone from "Sample" cuvette into waste container.

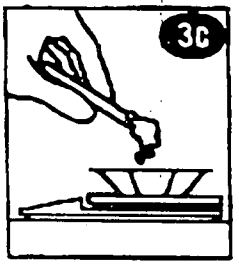
PHASE 2 SAMPLE EXTRACTION & PREPARATION

READ ALL INSTRUCTIONS BEFORE PROCEEDING WITH THE TEST

READ BEFORE PROCEEDING

- Sample should be mixed to ensure a homogeneous sample.

WEIGH SAMPLE



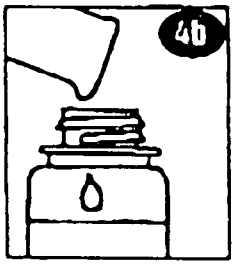
3a Place an unused weigh boat on pan balance.

3b Press ON/MEMORY button on pan balance. Balance will beep and display 0.0.

3c Weigh out 10+/- 0.1 grams of soil.

3d If balance turns off prior to completing weighing, use empty weigh boat to retare, then continue.

EXTRACT TNT



4a Measure 50 mL acetone in the 50mL graduated conical tube.

4b Pour acetone into an extraction jar.

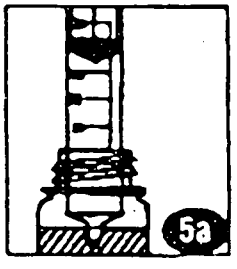
4c Using wooden spatula, transfer 10 grams of soil from weigh boat into extraction jar.

4d Recap extraction jar tightly and shake vigorously for three minutes.

4e Allow to settle for five minutes.

Repeat steps 3a - 4e for each sample to be tested.

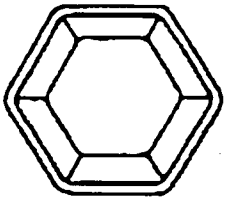
FILTER SAMPLE



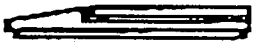
5a Place tip of 30 cc syringe into liquid above the sediment layer in the extraction jar and draw up 25 mL of the sample.

5b Screw the syringe filter onto the end of the syringe.

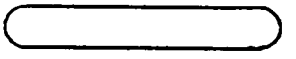
5c Press the plunger firmly and dispense the sample into the "Sample" cuvette.



Weigh Boat



Pan balance



Wooden spatula

50mL Graduated Conical Tube

Extraction Jar

30 cc syringe

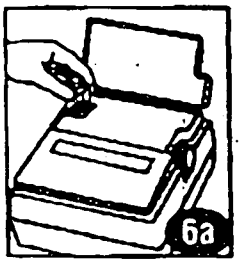
Syringe filter

Cuvette

PHASE 3 SAMPLE ANALYSIS

READ ALL INSTRUCTIONS BEFORE PROCEEDING WITH THE TEST

READ SAMPLE



6a Place the "Sample" cuvette in the cell holder.

6b Press the "READ/ENTER" key and record the absorbance on the worksheet as "$Abs_{initial}$".

6c Remove the "Sample" cuvette from the cell holder.

6d Add 1 drop of Developer Solution.

6e Cap the "Sample" cuvette and shake vigorously for 3 seconds.

DNT Analysis Note:

For analysis of samples containing DNT, and/or where DNT concentration is of concern, samples must be allowed to develop for 10 minutes before reading sample absorbance. This will not effect color development for other nitroaromatics.

6f Remove the cuvette stopper and place the "Sample" cuvette in the cell holder.

6g Press the "READ/ENTER" key and record the absorbance on the worksheet as "Abs_{sample}".

6h Clean cuvette between samples using procedure in steps 1a - 1h.

PHASE 4 INTERPRETATION

READ ALL INSTRUCTIONS BEFORE PROCEEDING WITH THE TEST

INTERPRETATION OF RESULTS

7a Multiply the "$Abs_{initial}$" value for each sample by 4. Enter these values on the worksheet.

7b Subtract this value from the "Abs_{sample}" values for each sample and record on the worksheet.

7c Divide the adjusted sample value by 0.0323 and record on the worksheet. This value is the TNT concentration of the sample in parts per million.

$$TNT_{(ppm)} = \frac{Abs_{sample} - (Abs_{initial} \times 4)}{0.0323}$$

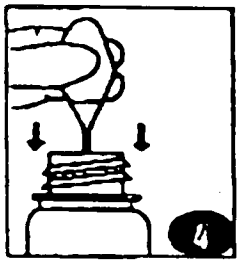
Note: For sample concentrations greater than 30ppm the sample extract should be diluted with acetone and reanalyzed. Remember to multiply the result by the dilution factor in order to determine the correct concentration.

CONTROL (QA/QC) CHECK

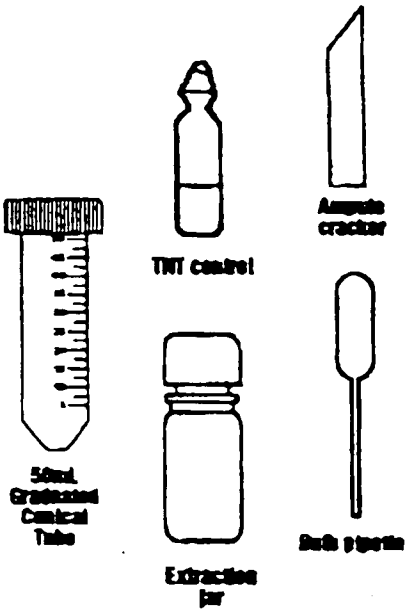
READ ALL INSTRUCTIONS BEFORE PROCEEDING WITH THE TEST

- **The TNT control is optional, but it is recommended that it be run daily.**

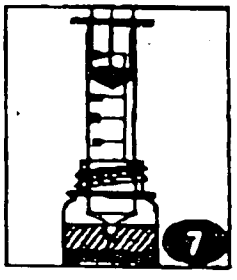
PREPARE CONTROL

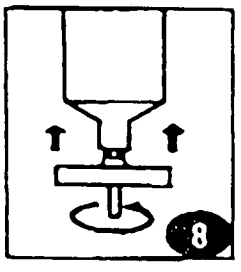


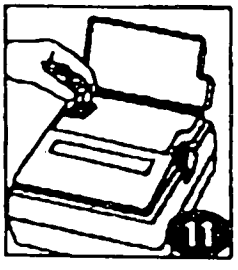
1. Measure 50 mL acetone in the 50mL graduated conical tube.
2. Pour into extraction jar.
3. Open TNT control ampule by slipping ampule cracker over top, and then breaking tip at scored neck.
4. Transfer entire contents of TNT control ampule into extraction jar using bulb pipette.
5. Cap extraction jar and shake vigorously for 3 seconds.



ANALYZE THE CONTROL



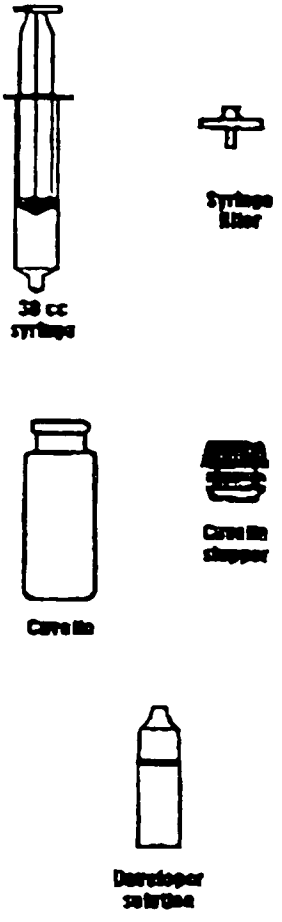




7. Place tip of 30 cc syringe in extraction jar and draw up 25 mL.
8. Attach syringe filter and dispense into "Sample" cuvette.
9. Add 1 drop of developer solution.
10. Cap the cuvette and shake vigorously for 3 seconds.
11. Remove the cuvette stopper and place in the cell holder.
12. Press "READ/ENTER" key and record the absorbance on the worksheet as "$Abs_{control}$".

    Absorbance must be between 0.307 - 0.373 for the test to be in control.

    If test is not in control, clean "Sample" cuvette, and then redo steps 7-12 using the remaining liquid from the extraction jar.
13. If test is in control clean "Sample" cuvette before proceeding with samples.



QUALITY CONTROL

READ ALL INSTRUCTIONS BEFORE PROCEEDING WITH THE TEST

System Description

Each SDI EnSys® TNT Soil Test System contains enough material to perform twenty complete tests.

The TNT Soil Test is divided into four phases. The instructions and notes should be reviewed before proceeding with the test.

Hotline Assistance

If you need assistance or are missing necessary Test System materials, call toll free: 1-800-544-8881.

Validation Information

Product claims are based on validation studies carried out under controlled conditions. Data has been collected in accordance with valid statistical methods and the product has undergone quality control tests of each manufactured lot.

Strategic Diagnostics Inc. does not guarantee that the results with the TNT Soil Test System will always agree with instrument-based analytical laboratory methods. All analytical methods, both field and laboratory, need to be subject to the appropriate quality control procedures.

How It Works

Controls, Samples, and color-change reagents are added to cuvettes. The concentration of TNT in an unknown **Sample** is determined by evaluating how much color is developed.

Quality Control

Standard precautions for maintaining quality control:

- Do not use reagents or components from one Test System with reagents or components from another Test System.
- Do not use the Test System after its expiration date.
- The sample must be analyzed immediately after adding the Developer Solution.
- Results may not be valid if DR/2000 reading for **Control** is outside of the range of 0.307 - 0.373.

Storage and Handling Precautions

- Wear protective gloves and eye wear.
- Store kit at room temperature and out of direct sunlight (less than 80°F).
- If acetone comes into contact with eyes, wash thoroughly with cold water and seek immediate medical attention.
- Operate test at temperatures greater than 4° C/40° F and less than 39° C/100° F.
- After use, dispose of kit components in accordance with applicable federal and local regulations.

ON-SITE QUALITY CONTROL/QUALITY ASSURANCE RECOMMENDATIONS SDI EnSys® TEST SYSTEM

Please read the following before proceeding with field testing.

SAMPLING

The result of your screening test is only as valid as the sample that was analyzed. Samples should be homogenized thoroughly to ensure that the 10 grams you remove for field testing is representative of the sample as a whole. All other applicable sample handling procedures should be followed as well.

PRIOR TO TESTING SAMPLES

Carefully follow the instructions in the User's Guide included with every test kit. This is the key element in obtaining accurate results. In addition, store your unused test kits at room temperature and do not use them past their expiration date (see label on each test kit).

INTERNAL TEST QC

One control is provided with each Kit to provide internal test system quality control. Test runs resulting in a number that falls outside of the specified range should be repeated to ensure valid conclusions.

QA/QC

The validity of field test results can be substantially enhanced by employing a modest, but effective QA/QC plan. SDI recommends that you structure your QA/QC plan with the elements detailed below. These have been developed based on the data quality principles established by the U.S. Environmental Protection Agency.

A. **Sample Documentation**
   1. Location, depth
   2. Time and date of collection and field analysis

B. **Field analysis documentation** - provide raw data, calibration, any calculations, and final results of field analysis for all samples screened (including QC samples)

C. **Method calibration** - this is an integral part of SDI tests; a TNT control analysis should be performed daily (see the instructions in the User's Guide)

D. **Method blank** - field analyze fresh acetone

E. **Site-specific matrix background field analysis** - collect and field analyze uncontaminated sample from site matrix to document matrix effect

F. **Duplicate sample field analysis** - field analyze duplicate sample to document method repeatability; at least one of every 20 samples should be analyzed in duplicate

G. **Confirmation of field analysis** - provide confirmation of the quantitation of the analyte via an EPA-approved method different from the field method on at least 10% of the samples; provide chain of custody and documentation such as gas chromatograms, mass spectra, etc.

H. **Performance evaluation sample field analysis (optional, but strongly recommended)** - field analyze performance evaluation sample daily to document method/operator performance

I. **Matrix spike field analysis (optional)** - field analyze matrix spike to document matrix effect on analyte measurement

FURTHER QUESTIONS?

SDI's Technical Support personnel are always prepared to discuss your quality needs to help you meet your data quality objectives. Call 1-(800) 544-8881.

TNT SOIL TEST - ABBREVIATED PROCEDURE

1	• Clean cuvettes • Zero the spectrophotometer at 540 nm
2	• Add 10 g soil and 50 ml acetone to extraction jar • Shake 3 minutes, let settle • Draw up 25 mL extract, filter into cuvette
3	• Read $Abs_{initial}$, record • Add 1 drop developer solution, shake • Read Abs_{sample}, record
4	• Multiply $Abs_{initial}$ by 4 • Subtract from Abs_{sample} • Divide by 0.0323 • $TNT_{(ppm)} = \frac{Abs_{sample} - (Abs_{initial} \times 4)}{0.0323}$

TNT SOIL TEST KIT WORKSHEET

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

1	2	3	4	5	6
SAMPLE #	$Abs_{initial}$	Abs_{sample}	$Abs_{initial}$ x4	Abs_{final} (Column 3 - Column 4)	TNT CONC ppm (Column 5/0.0323)

FIELD SOP: QAPP-6.0
TITLE: SAMPLE PACKAGING AND SHIPPING

1.0 PURPOSE

This procedure is provided to ensure that samples are properly packaged to prevent or minimize breakage and maintain sample integrity between the field and laboratory, and to maintain proper chain-of-custody protocols.

This procedure describes the methods which will be used to package and ship samples for off-site laboratory analysis. Quality assurance requirements for field sample documentation, packaging and shipment are described in Section 5.0 of this QAPP for Full-Scale Operations at the Biofacility.

2.0 TOOLS AND MATERIALS

The following tools and materials are required to perform packaging and shipping of samples for off-site laboratory analysis.

- Chain Of Custody
- custody seals
- cooler
- ice
- disposable sealable plastic bags
- bubble wrap bags or sheets
- temperature blank sample
- shipping tape
- logbook
- indelible pen

3.0 GENERAL

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

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

4.0 SAMPLE LABELS AND TAGS

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

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

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

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

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

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

5.0 CHAIN-OF-CUSTODY FORM (COC)

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

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

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

-required analyses.

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

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

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

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

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

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

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

6.0 SAMPLE PREPARATION

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

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

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

3. Place sample jars (glass) in protective bubble wrap and seal using adhesive strip on bubble wrap package or tape. NOTE: EnCore samplers will be placed in marked plastic zipper baggies.

7.0 SAMPLE PACKAGING

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

1. Line the base and sides of the cooler with bubble wrap. Coolers designated for

-required analyses.

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

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

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

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

7.0 SAMPLE PACKAGING

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

1. Line the base and sides of the cooler with bubble wrap. Coolers designated for

decon and rinse water samples will be provided by the Contractor-procured laboratory.

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

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

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

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

6. Seal the cooler with strapping tape.

8.0 SAMPLE SHIPPING

Ship all samples to the laboratory for next-day delivery. Ensure the air bills have been completely filled out and copied. Place one airbill into each envelope. Affix the envelope to the top of the cooler, if the lid is flat. If the lid is sliding, as with a "playmate" cooler, affix the envelope to the side of the cooler. Do not obstruct address labels provided by the laboratory (if present).

NOTE: If samples are collected on Friday, the block labeled "Saturday Delivery" on the airbill MUST BE MARKED. Otherwise the samples will arrive at the lab on Monday.

Fax the Chain-of-Custody Form to the Contractor Project Chemist or designee [(216) 523-5201] the same day samples are shipped. If short turn-around time is needed, such as 24 or 48-hours, notify the Project Chemist IMMEDIATELY by phone. It is

preferable to notify the Project Chemist of short turn-around times as soon as it is known that samples will require this.

9.0 QA/QC AND DOCUMENTATION

Maintain all generated sample documentation (copies of COCs, field logbooks, etc) in the field during the course of the site work. This documentation may be managed by the Contractor, or if managed by the Subcontractor will be available for review. At the conclusion of site work, provide a copy of all documentation to the Contractor.

APPENDIX B
STANDARD METHOD DETECTION LIMITS FOR ANALYTICAL METHODS

APPENDIX B
STANDARD METHOD DETECTION LIMITS FOR ANALYTICAL METHODS

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SOUTHWEST LABORATORY OF OKLAHOMA, INC.

Polynuclear Aromatic Hydrocarbons

Test Code	GC420
Method	SW846/8310
Matrix	Water-Soil
Extract Volume	1000 mL - 30g
Initial Calibration	5 point calibration, %RSD = 20
Continuing Calibration	single point calibration, %D = 15
Instrument/analysis date (soils	inst 10 -- 4/93
Instrument/analysis date (wate	inst 10 -- 2/93

COMPOUND	CAS NUMBER	PQL's		MDL's	
		WATER	SOIL	WATER	SOIL
		mg/L	mg/Kg	mg/L	mg/Kg
Naphthalene	91-20-3	0.018	0.560	0.0018	0.0558
2-Fluorobiphenyl (surrogate)	321-60-8				
Acenaphthylene	208-96-8	0.017	0.430	0.0017	0.0430
Acenaphthene	83-32-9	0.018	1.20	0.0038	0.1494
Fluorene	86-73-7	0.0021	0.121	0.0003	0.0121
Phenanthrene	85-01-8	0.004	0.165	0.0004	0.0165
Anthracene	120-12-7	0.0001	0.006	0.00001	0.0006
Fluoranthene	206-44-0	0.0019	0.084	0.0002	0.0084
Pyrene	129-00-0	0.0027	0.180	0.0007	0.0356
Paraterphenyl (surrogate)	92-94-4				
Benzo(a)anthracene	56-55-3	0.0001	0.032	0.0002	0.0080
Chrysene	218-01-9	0.0015	0.100	0.0006	0.0248
Benzo(b)fluoranthene	205-99-2	0.0002	0.012	0.00003	0.0016
Benzo(k)fluoranthene	207-08-9	0.0002	0.009	0.00002	0.0009
Benzo(a)pyrene	50-32-8	0.0002	0.015	0.0001	0.0019
Indeno(1,2,3-cd)pyrene	193-39-5	0.0007	0.034	0.0002	0.0085
Dibenz(a,h)anthracene	53-70-3	0.0009	0.132	0.0009	0.0331
Benzo(g,h,i)perylene	191-24-2	0.0007	0.142	0.0007	0.0354

Quantitation limits listed for soil/sediment are based on wet weight. The quantitation limits calculated the laboratory for soil/sediment, calculated on dry weight basis will be higher. Practical Quantitation Li will be twice for GPC cleanup.

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

PQL = (practical quantitation limit) based on the product of the MDL and a mulitplier ranging from 5 to 10.

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SOUTHWEST LABORATORY OF OKLAHOMA, INC.

Tetra-Octa Dioxin/Furans-High Res Mass Spec

Test Code MS790
Method SW846/8290, High Resolution Method
Matrix Water-Soil
Extract Volume 1000 mL - 10g
Initial Calibration (b) 1.0/2.5/5 - 200/500/1000 ng/mL
Continuing Calibration (b) 10/25/50 ng/mL

COMPOUND PCDD/PCDF (a)	CAS NUMBER	PQL's WATER pg/L	PQL's SOIL ng/Kg	MDL's WATER pg/L	MDL's SOIL ng/Kg
2378-TCDD	1746-01-6	10	1	6.79	0.42
12378-PeCDD	40321-76-4	50	5	6.64	0.27
123478-HxCDD	39227-28-6	50	5	17.63	1.15
123678-HxCDD	57653-85-7	50	5	13.56	1.43
123789-HxCDD	19408-74-3	50	5	15.35	1.66
1234678-HpCDD	35822-39-4	50	5	14.44	0.98
OCDD	3268-87-9	100	10	21.46	4.78
2378-TCDF	51207-31-9	10	1	2.96	0.97
12378-PeCDF	57117-41-6	50	5	5.58	0.94
23478-PeCDF	57117-31-4	50	5	13.26	1.28
123478-HxCDF	70648-26-9	50	5	7.96	1.03
123678-HxCDF	57117-44-9	50	5	8.68	1.69
123789-HxCDF	72918-21-9	50	5	17.87	1.32
234678-HxCDF	60851-34-5	50	5	16.00	1.16
1234678-HpCDF	67562-39-4	50	5	10.99	1.16
1234789-HpCDF	55673-89-7	50	5	17.98	0.78
OCDF	39001-02-0	100	10	10.63	2.89

Water MDL Performed <12-Feb-97>
Soil MDL Performed <09/23/97>

Internal Standards (c) 13C Labeled Compounds #(1,2,3,4,7,9,13,14,16)) Uses 9 Internal Standards
Recovery Standards (d) 13C-1,2,3,4-TCDD, 13C-1,2,3,7,8,9-HxCDD
Surrogate Standard 37C-2,3,7,8-TCDD
Second Column Confirmation Required for TCDF

(a) **Polychlorinated dioxins and Furans (PCDD/PCDF)**

TCDD = Tetrachlorodibenzo-p-dioxin — TCDF = Tetrachlorodibenzofu
PeCDD = Pentachlorodibenzo-p-dioxin — PeCDF = Pentachlorodibenzo
HxCDD = Hexachlorodibenzo-p-dioxin — HxCDF = Hexachlorodibenzof
HpCDD = Heptachlorodibenzo-p-dioxin — HpCDF = Heptachlorodibenz
OCDD = Octachlorodibenzo-p-dioxin — OCDF = Octachlorodibenzofu

(b) It is common practice to use MS780 Calibration levels for MS790, which are: In Calibration: 0.5/2.5/5.0 — 200/1000/2000 ng/mL, Continuing Calibration 01/50/

(c) The term Internal Standard is equivalent to surrogate standards in Method 827 EPA Method 1613A uses the term "labeled compounds".

(d) The term Recovery Standard is equivalent to Internal Standard in Method 8270 EPA Method 1613A uses the term "Internal Standard".

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SOUTHWEST LABORATORY OF OKLAHOMA, INC.

	Semivolatile
Test Code	MS500
Method	SW846 8270B, Rev. 2.0, Sept. 1994
Matrix	Water-Soil
Extract Volume	1000 mL - 30g
Initial Calibration	10, 20, 50, 80, 100 ng,%RSD for CCC compounds=30%, SPCC=RF > 0.05
Continuing Calibration	50 ng, %D = 20% for CCC Compounds, SPCC = RF > 0.05

PAGE 1 OF 2

COMPOUND		CAS NUMBER	PQL's		MDL's	
			WATER	SOIL	WATER	SOIL
			ug/L	ug/Kg	ug/L	ug/Kg
Phenol	*	108-95-2	10	330	3.3	100
bis(2-Chloroethyl)ether		111-44-4	10	330	3.4	100
2-Chlorophenol		95-57-8	10	330	3.3	97
1,3-Dichlorobenzene		541-73-1	10	330	2.6	100
1,4-Dichlorobenzene	*	106-46-7	10	330	2.8	120
Benzyl alcohol		100-51-6	10	330	3.6	82
1,2-Dichlorobenzene		95-50-1	10	330	3.0	100
2-Methylphenol		95-48-7	10	330	2.9	130
bis(2-Chloroisopropyl)ether		108-60-1	10	330	3.5	89
4-Methylphenol		106-44-5	10	330	3.4	94
N-Nitroso-di-n-propylamine	**	621-64-7	10	330	2.8	87
Hexachloroethane		67-72-1	10	330	2.3	94
Isophorone		78-59-1	10	330	3.0	100
Nitrobenzene		98-95-3	10	330	3.5	100
2-Nitrophenol	*	88-75-5	10	330	3.4	99
2,4-Dimethylphenol		105-67-9	10	330	3.9	160
bis(2-Chloroethoxy)methane		111-91-1	10	330	3.3	99
2,4-Dichlorophenol	*	120-83-2	10	330	2.6	110
Benzoic acid		65-85-0	50	1600	9.2	150
1,2,4-Trichlorobenzene		120-82-1	10	330	2.9	94
Naphthalene		91-20-3	10	330	2.6	110
4-Chloroaniline		106-47-8	10	330	3.3	210
Hexachlorobutadiene	*	87-68-3	10	330	3.0	90
4-Chloro-3-methylphenol	*	59-50-7	10	330	2.6	90
2-Methylnaphthalene		91-57-6	10	330	2.4	85
Hexachlorocyclopentadiene	**	77-47-4	10	330	NA	75
2,4,6-Trichlorophenol	*	88-06-2	10	330	2.6	110
2,4,5-Trichlorophenol		95-95-4	50	1600	2.7	110
Carbazole	#	86-74-8	10	330	1.1	290

WATER MDLS PERFORMED ON INST V <01/22/97>
SOIL MDL'S PERFORMED ON INST P <02/06/97>
- Cabazole MDL performed on instr. P <1/97>

Semivolatile
PQLs/MDLs

Method SW846 8270B, Rev. 2.0, Sept. 1994

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2-Chloronaphthalene		91-58-7	10	330	2.1	110
2-Nitroaniline		88-74-4	50	1600	2.8	110
Dimethylphthalate		131-11-3	10	330	0.8	120
Acenaphthylene		208-96-8	10	330	2.4	120

rev 4.0 02/06/98

2,6-Dinitrotoluene		606-20-2	10	330	4.0	110
3-Nitroaniline		99-09-2	50	1600	3.6	150
Acenaphthene	*	83-32-9	10	330	2.2	100
2,4-Dinitrophenol	**	51-28-5	50	1600	2.9	100
4-Nitrophenol	**	100-02-7	50	1600	2.6	93
Dibenzofuran		132-64-9	10	330	1.9	110
2,4-Dinitrotoluene		121-14-2	10	330	3.9	100
Diethylphthalate		84-66-2	10	330	1.2	120
Fluorene		86-73-7	10	330	1.8	100
4-Chlorophenyl-phenylther		7005-72-3	10	330	2.2	120
4-Nitroaniline		100-01-6	50	1600	2.8	150
4,6-Dinitro-2-methylphenol		534-52-1	50	1600	2.4	100
N-nitrosodiphenylamine	*	86-30-6	10	330	2.4	110
4-Bromophenyl-phenylether		101-55-3	10	330	2.3	86
Hexachlorobenzene		118-74-1	10	330	2.6	84
Pentachlorophenol	*	87-86-5	50	1600	2.3	76
Phenanthrene		85-01-8	10	330	2.6	110
Anthracene		120-12-7	10	330	2.6	100
Di-n-butylphthalate		84-74-2	10	330	2.0	110
Fluoranthene	*	206-44-0	10	330	1.9	100
Pyrene		129-00-0	10	330	1.2	120
Buytylbenzylphthalate		85-68-7	10	330	1.1	120
Benzo(a)anthracene		56-55-3	10	330	1.0	100
3,3'-Dichlorobenzidine		91-94-1	20	660	2.4	120
Chrysene		218-1-9	10	330	0.9	100
bis(2-Ethylhexyl)phthalate		117-81-7	10	330	3.5	140
Di-n-octylphthalate	*	117-84-0	10	330	2.0	110
Benzo(b)fluoranthene		205-99-2	10	330	1.8	120
Benzo(k)fluoranthene		207-08-9	10	330	2.1	100
Benzo(a)pyrene	*	50-32-8	10	330	1.6	83
Indeno(1,2,3-cd)pyrene		193-39-5	10	330	1.6	110
Dibenz(a,h)anthracene		53-70-3	10	330	1.6	120
Benzo(g,h,i)perylene		191-24-2	10	330	1.6	130

* CCC compounds **SPCC compounds

Quantitation limits listed for soil/sediment are based on wet weight. The quantitation limits calculated the laboratory for soil/sediment, calculated on dry weight basis will be higher. Practical Quantitation Li will be twice for GPC cleanup. NOTE: compounds with elevated detections limits (50 ug/l and 1600 u have a 4 point calibration.

ref col
14-Feb-97
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SOUTHWEST LABORATORY OF OKLAHOMA, INC.

Volatiles - TCLP compounds

Test Code MS340

Method SW846 8240, 3rd Edition, Nov. 1986/Sept. 1994

Matrix Water

Sample Volume 5 mL

Initial Calibration 5-20-50-100-200 ppb ,%RSD<30% for CCC compounds, SPCC RRF >0.300 except for Bromoform RRF>0.100

Continuing Calibration 50 ppb, %D < 20% for CCC Compounds, SPCC RRF > 0.300, except for Bromoform RRF>0.100

COMPOUND		CAS Number	PQL's WATER ug/l	MDL's WATER ug/l
Vinyl Chloride	*	75-01-4	10	1.4
1,1-Dichloroethene	*	75-35-4	5	1.4
2-Butanone		78-93-3	10	0.88
Benzene		71-43-2	5	15
1,2-Dichloroethane		107-06-2	5	0.44
Trichloroethene		79-01-6	5	1.0
Tetrachloroethene		127-18-4	5	1.2
Chlorobenzene	**	108-90-7	5	0.83

*CCC **SPCC

MDL'S FOR WATER PERFORMED ON INSTR. N <01/17/97>

ref col
28-Feb-97
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SOUTHWEST LABORATORY OF OKLAHOMA, INC.

	Volatiles
Test Code	MS326
Method	SW846 8260 Third Edition, Nov. 1986/Sept. 1994
Matrix	Water-Soil
Sample Volume	5 mL - 5 g
Initial Calibration(5ml/5g)	5-20-50-100-200 ppb,%RSD<30% for CCC compounds,SPCC RRF>0.300 for C and 1,1,2,2-Tetrachloroethane; SPCC RRF > 0.100 for Chloromethane, 1,1,-Dic and Bromoform
Continuing Calibration(5ml/5g)	50 ppb, %D < 20% for CCC compounds, SPCC RRF > 0.300 for Chlorobenzene Tetrachloroethane; SPCC RRF > 0.100 for Chloromethane, 1,1-Dichloroethane, Bromoform
Internal Standards	Chlorobenzene-D5, 1,4-Dichlorobenzene-D4, 1,4-Difluorobenzene, Pentafluorob
Surrogates	Toluene-D8, 4-Bromofluorobenzene, Dibromofluoromethane

	COMPOUND		CAS NUMBER	PQL WATER, 5mL ug/L	PQL SOIL ug/Kg	MDL WATER, 5 ug/L	MDL SOIL ug/Kg
	1,2-Dichloroethene (total)		540-59-0	5	5	1.4	0.60
	DICHLORODIFLUOROMETHAN		75-71-8	5	5	3.0	2.7
	CHLOROMETHANE	**	74-87-3	5	5	3.7	1.0
	VINYL CHLORIDE	*	75-01-4	5	5	4.3	0.98
	BROMOMETHANE		74-83-9	5	5	3.5	0.63
	CHLOROETHANE		75-00-3	5	5	2.7	1.1
	TRICHLOROFLUOROMETHAN		75-69-4	5	5	1.1	0.57
NR	ACROLEIN		107-02-8	5	5	27	29
	1 1-DICHLOROETHENE	*	75-35-4	5	5	1.5	0.61
NR	ACETONE		67-64-1	5	5	3.9	1.6
NR	CARBON DISULFIDE		75-15-0	5	5	2.7	0.59
	METHYLENE CHLORIDE		75-09-2	5	5	4	1.9
	trans-1 2-DICHLOROETHENE		156-60-5	5	5	1.4	0.60
NR	ACRYLONITRILE		107-13-1	5	5	26	18
NR	VINYL ACETATE		108-05-4	5	5	2.2	0.75
	1 1-DICHLOROETHANE	**	75-34-3	5	5	1	0.52
	cis-1 2-DICHLOROETHENE		156-59-2	5	5	1.2	0.54
	2 2-DICHLOROPROPANE		594-20-7	5	5	4.8	1.7
NR	2-BUTANONE		78-93-3	5	5	4.4	1.4
	BROMOCHLOROMETHANE		74-97-5	5	5	0.69	0.71
	CHLOROFORM		67-66-3	5	5	0.95	0.47
	1 1 1-TRICHLOROETHANE		71-55-6	5	5	1.1	0.46
	1 1-DICHLOROPROPENE		563-58-6	5	5	1.3	0.72
	CARBON TETRACHLORIDE		56-23-5	5	5	2.1	0.32
	BENZENE		71-43-2	5	5	0.98	0.43
	1 2-DICHLOROETHANE		107-06-2	5	5	0.83	0.23
	TRICHLOROETHENE		79-01-6	5	5	2.9	0.48
	1 2-DICHLOROPROPANE	*	78-87-5	5	5	0.88	0.48
NR	1-CHLOROHEXANE		544-10-5	5	5	1.6	1.0
	DIBROMOMETHANE		74-95-3	5	5	0.88	0.64
	BROMODICHLOROMETHANE		75-27-4	5	5	1	0.43
NR	2-CHLOROETHYL VINYL ETHE		110-75-8	5	5	3.7	0.47
NR	cis-1,3-Dichloropropene		10061-01-5	5	5	0.96	0.36
NR	4-METHYL-2-PENTANONE		108-10-1	5	5	1.0	0.54
	TOLUENE	*	108-88-3	5	5	1.4	0.25
NR	trans-1,3-Dichloropropene		10061-02-6	5	5	1.5	0.43
	1 1 2-TRICHLOROETHANE		79-00-5	5	5	0.66	0.69

	TETRACHLOROETHENE		127-18-4	5	5	1.1	0.59
	1 3-DICHLOROPROPANE		142-28-9	5	5	0.84	0.82
NR	2-HEXANONE		591-78-6	5	5	1.9	1.4
	DIBROMOCHLOROMETHANE		124-48-1	5	5	0.88	0.87
	1 2-DIBROMOETHANE		106-93-4	5	5	1.1	0.29
	CHLOROBENZENE	**	108-90-7	5	5	1.1	0.70
	1 1 1 2-TETRACHLOROETHAN		630-20-6	5	5	1	0.71
	ETHYL BENZENE	*	100-41-4	5	5	0.98	0.65
	m,p-XYLENES		13-302-07	5	5	2	1.8
	Xylene (Total)		1330-20-7	5	5	3.2	2.3
	o-XYLENE		95-47-6	5	5	1	2.3
	STYRENE		100-42-5	5	5	0.8	2.4
	BROMOFORM	**	75-25-2	5	5	1.3	1.5
	ISOPROPYLBENZENE		98-82-8	5	5	1.4	1.0
	1 2 3-TRICHLOROPROPANE		96-18-4	5	5	2.7	1.0
	BROMOBENZENE		108-86-1	5	5	1.1	0.85
	1 1 2 2-TETRACHLOROETHAN	**	79-34-5	5	5	2.3	1.2
	n-PROPYLBENZENE		103-65-1	5	5	1.2	1.0
	2-CHLOROTOLUENE		95-49-8	5	5	1.3	0.91
	4-CHLOROTOLUENE		106-43-4	5	5	0.97	0.74
	1 3 5-TRIMETHYLBENZENE		108-67-8	5	5	1.2	0.76
	tert-BUTYLBENZENE		98-06-6	5	5	1.2	2.5
	1 2 4-TRIMETHYLBENZENE		95-63-6	5	5	1.1	0.71
	sec-BUTYLBENZENE		135-98-8	5	5	1.1	0.6
	1 3-DICHLOROBENZENE		541-73-1	5	5	0.9	1.2
	p-ISOPROPYLTOLUENE		99-87-6	5	5	1.1	0.69
	1 4-DICHLOROBENZENE		106-46-7	5	5	1.1	0.79
	1 2-DICHLOROBENZENE		95-50-1	5	5	0.96	1.1
	n-BUTYLBENZENE		104-51-8	5	5	1.1	0.73
	1 2-DIBROMO-3-CHLOROPROP		96-12-8	5	5	3.5	1.3
	1 2 4-TRICHLOROBENZENE		120-82-1	5	5	1.8	2.9
	HEXACHLOROBUTADIENE		87-68-3	5	5	1.4	0.91
	NAPHTHALENE		91-20-3	5	5	3.5	2.7
	1 2 3-TRICHLOROBENZENE		87-61-6	5	5	2.2	1.9

NR = NonRoutine Compounds. Analyzed only upon request.

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SOUTHWEST LABORATORY OF OKLAHOMA, INC.

	Explosives
Test Code	GC200
Method	SW846 8330, 3rd Edition, Nov. 1992
Matrix	Water-Soil
Extract Volume	1 ml - 2 g
Initial Calibration	3 replicates of a 3 point calibration, %RSD = 20
Continuing Calibration	single point calibration, %D = 15
Instrument/analysis date (soils	inst 13 -- 6/3/97
Instrument/analysis date (wate	inst 13 -- 3/97

COMPOUND	CAS NUMBER	PQL's		MDL's	
		WATER	SOIL	WATER	SOIL
		ug/L	ug/Kg	ug/L	ug/Kg
HMX	2691-41-0	13.0	1600	1.79	85.8
RDX	121-82-4	10.4	625	2.06	79.4
TNB	99-35-4	7.2	575	1.78	38.5
TETRYL	479-45-8	9.0	383	3.29	119.8
DNB	99-65-0	3.8	488	1.52	28.4
TNT	118-96-7	6.4	500	3.22	27.7
NB	98-95-3	6.8	350	3.08	126.2
26DNT	606-20-2	9.2	388	4.08	149.1
24DNT	121-14-2	5.6	240	3.62	41.9
2NT	88-72-2	11.6	725	5.81	51.0
4NT	99-99-0	8.0	475	4.07	35.3
3NT	99-08-1	7.6	500	3.01	57.7
2ADNT	355-72-78-2	5.6	400	2.93	37.07
4ADNT	1946-51-0	10	450	5.36	76.41

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

PQL = (practical quantitation limit) based on the product of the MDL and a mulitplier ranging from 5 to 10.

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13-Feb-97
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SOUTHWEST LABORATORY OF OKLAHOMA, INC.

Metals reporting limits by ICP

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

COMPOUND	CAS NUMBER	Test Services List	MDLS* WATER ug/L	MDLS* SOIL mg/kg	PQLS WATE ug/L	PQLS SOIL mg/kg
Aluminum	7429-90-5	MT005	14	2.8	100	15.0
Antimony	7440-36-0	MT025	12	1.7	60	6.0
Arsenic	7440-38-2	MT055	32	3.1	160	20.0
Barium	7440-39-3	MT065	1.0	0.19	10	1.0
Beryllium	7440-41-7	MT085	1.0	0.10	5	0.5
Boron	7440-42-8	MT115	17	2.5	100	15.0
Cadmium	7440-43-9	MT125	1.3	0.11	5	0.5
Calcium	7440-70-2	MT145	39	23.0	500	150.0
Chromium	7440-47-3	MT165	1.9	0.38	10	1.0
Cobalt	7440-48-4	MT185	2.8	0.46	20	2.5
Copper	7440-50-8	MT205	8.3	0.73	25	2.5
Iron	7439-89-6	MT225	18	1.7	100	10.0
Lead	7439-92-1	MT245	12	1.5	100	10.0
Magnesium	7439-95-4	MT265	25	6.1	250	50.0
Manganese	7439-96-5	MT285	1.2	0.10	10	1.0
Molybdenum	7439-98-7	MT325	5.7	0.25	30	2.0
Nickel	7440-02-0	MT345	6.5	0.59	35	4.0
Potassium	7440-09-7	MT365	560	57.0	5000	500.0
Selenium	7782-49-2	MT385	28	3.1	250	25.0
Silicon	7440-21-3	MT445	70	23.0	450	170.0
Silver	7440-22-4	MT405	1.4	0.25	10	1.0
Sodium	7440-23-5	MT425	27	50.0	500	250.0
Thallium	7440-28-0	MT455	48	4.6	250	25.0
Tin	7440-31-5	MT465	17	2.1	100	15.0
Titanium	7440-32-6	MT485	1.0	0.14	10	1.0
Vanadium	7440-62-2	MT505	2.2	0.27	20	2.0
Zinc	7440-66-6	MT525	11	1.1	20	2.0

*DATA BASED ON WATER MDL'S <12/02/96> & <01/29/97>
DATA BASED ON SOIL MDL'S <12/02/96> & <01/29/97>

ref col
14-Feb-97
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SOUTHWEST LABORATORY OF OKLAHOMA, INC.

Mercury by Cold Vapors

Test Code	MT310
Method	SW846 Third Edition, Nov. 1986
Matrix	Water-Soil
Extract Volume	100mL - 0.6g
Initial Calibration	0 - 10.0 ug/L
Continuing Calibration	1/2 HIGH STD

COMPOUND	CAS NUMBER	MDLS*		PQLS		Test	
		WATER	SOIL	WATER	SOIL	Services	Method
		ug/L	mg/kg	ug/L	mg/kg	List	
Mercury	7439-97-6	0.12		0.20		MT310	7470
Mercury	7439-97-6		0.030		0.033	MT310	7471

* DATA BASED ON MDL'S ON <12/18/96>

ref col
07-Jul-95
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SOUTHWEST LABORATORY OF OKLAHOMA, INC.

Metals Toxicity by TCLP/EP Toxicity

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

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

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

ref col
07-Jul-95
r2
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SOUTHWEST LABORATORY OF OKLAHOMA, INC.

Micellaneous Inorganic Analyses

Test Code Methods various
Method
Matrix Water-Soil
Extract Volume
Initial Calibration
Continuing Calibration

COMPOUND	METHOD	Test Services List	PRACT. QUANT. LIMIT WATER mg/L	SOIL mg/Kg
Acidity	SM402	IN005	1.0	-
Alkalinity	EPA310.1	IN010	20.0	-
Ammonia (ISE)	EPA350.3	IN015	0.1	1.0
Ammonia (Dist)	EPA350.2	IN020	1.0	10.0
Bromide (IC)	EPA300.0	IN030	0.2	1.0
Chemical Oxygen Demand	EPA410.4	IN090	10.0	100.0
Chloride (IC)	EPA300.0	IN055	0.2	1.0
Chloride (by titration)	EPA325.3	IN050	0.1	1.0
Chlorine (Residual)	EPA330.4	IN070	0.2	-
Chlorine (free)	SM408D	IN075	0.1	-
Color	SM204A	IN100	-	-
Corrosivity pH (@25 C)	SW846-9045	HW200	-	-
Cyanide (Total)	SW846-9010	IN120	10.0	0.5
Cyanide (Amenable)	SW846-9010	IN122	0.0	0.5
Density	SM210B	IN128	-	-
Fluoride (IC)	EPA300.0	IN135	0.2	1.0
Fluoride (ISE)	EPA340.2	IN137	0.1	-
Hardness (As CaCO3)	EPA130.2	IN140	1.0	-
Hexavalent Chromium	SW846-7196	MT169	0.05	0.25
Iodine	EPA300.0	IN150	0.2	1.0
Nitrate (Color)	EPA353.2	IN180	0.1	1.0
Nitrate (IC)	EPA300.0	IN185	0.2	0.2
Nitrite (Color)	EPA354.1	IN190	0.1	0.4
Nitrite (IC)	EPA300.0	IN195	0.2	0.4
Oil & Grease	EPA413.1	IN200	0.5	10.0
Oil & Grease by IR	EPA413.2	IN205	0.5	10.0
Ortho Phosphorus (Color)	EPA365.2	IN250	0.1	10.0
Ortho Phosphorus (IC)	EPA300.0	IN255	0.2	1.0
Paint FIlter (Filtrate)		HW500	-	-
Percent Moisture	GRAY 105c	IN650	-	
pH Value (@ 25 C)	EPA150.1	IN220	-	-
Phenols (Total)	EPA420.2	IN230	0.1	0.5
Phosphorus (Total)	EPA365.2	IN240	0.1	10.0
Reactivity Cyanide	SW 7.3.4.2	IN125	0.1	0.1
Reactivity Sulfide	SW 7.3.4.2	IN305	0.0	1.0
Silica	EPA370.1	IN260	0.2	1.0
Sodium Absorption Ratio	CALCULATION	IN620	-	-
Specific Conductance (@ --	EPA120.1	IN080	-	-
Specific Gravity	SM213E	IN139	-	-
Sulfate (Gravimetric)	SM426B	IN290	0.2	1.0
Sulfate (IC)	EPA300.0	IN295	0.2	1.0
Sulfate (Turbidimetric)	EPA375.4	IN297	1.0	1.0
Sulfide	EPA376.1	IN300	1.0	1.0
Sulfite	EPA377.1	IN307	2.0	2.0
Sulfur	ASTM D-1552	OS630	-	-
Surfactants (MBAS)	SM512B	IN160	0.1	-
Total Dissolved Solids	EPA160.1	IN270	10.0	-
Total Kjeldahl Nitrogen	EPA351.3	IN170	1.0	10.0
Total Organic Carbon	EPA415.1	IN045	0.4	100.0
Total Organic Halogens	SW9020	IN060	0.0	10.0
Total Petroleum Hydrocarbo	SM503E	IN212	0.5	10.0
Total Residue	EPA160.3	IN280	10.0	-
Total Settleable Solids	EPA160.5	IN287	-	-
Total Suspended Solids	EPA160.2	IN275	10.0	-
Total Volatile Solids	EPA160.4	IN285	10.0	-
Turbidity	EPA180.1	IN310	0.1	-
Volatile Suspended Solids	SM209D	IN286	1.0	-
Water Content (Dean Startk)	ASTM E-123	IN655	-	-

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SOUTHWEST LABORATORY OF OKLAHOMA, INC.

Micellaneous Inorganic Analyses

Test Code Methods various
Method
Matrix Water-Soil
Extract Volume
Initial Calibration
Continuing Calibration

COMPOUND		METHOD	Test Services List	PRACT. QUANT. LIMIT WATER mg/L	SOIL mg/Kg
Bromide (IC)		EPA300.0	IN030	0.2	1.0
Chloride (IC)		EPA300.0	IN055	0.2	1.0
Fluoride (IC)		EPA300.0	IN135	0.2	1.0
Nitrate (IC)		EPA300.0	IN185	0.2	0.2
Nitrite (IC)		EPA300.0	IN195	0.2	0.4
Ortho Phosphorus (IC)		EPA300.0	IN255	0.2	1.0
Phosphorus (Total)		EPA365.2	IN240	0.1	10.0
Sulfate (IC)		EPA300.0	IN295	0.2	1.0

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
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APPENDIX C
LABORATORY STANDARD OPERATING PROCEDURES

APPENDIX C
LABORATORY STANDARD OPERATING PROCEDURES

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SECTION 1
PREVENTIVE MAINTENANCE

PROCEDURAL DOCUMENTS - SYSTEM TRANSITION

Please note that the following document is in transition from a previous system. The document identification number (listed below) has been assigned in compliance with the SWL SOP titled:

"Preparation, Review, Revision, and Control of Procedural Documents", SWL-GA-101, Rev. 1.0 — 05/14/97

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Upon revision or review, the following document will be re-issued in full compliance with the guidelines set forth in GA-101, therefore consistent with newly converted or written procedural documents.

Existing ID: SWL-GENTKMR

NEW DOCUMENT IDENTIFICATION NUMBER:

SWL-GA-103

Standard Operating Procedure

"Tekmar Maintenance"
Rev. No. 1.2
Issued: 05/14/97

SOUTHWEST LABORATORY OF OKLAHOMA, INC.

Standard Operating Procedure

Tekmar Maintenance

Document No.: SWL-GA-GENTKMR

Rev No. / Date: Rev. 1.2 — 05/14/97

OFFICIAL COPY RED STAMP INDICATES ORIGINAL

APPROVALS	DATES
Janet Williamson Procedure Prepared By	5/30/97 Date
Gena Jackson Program Manager or Section Supervisor	5/30/97 Date
Mike Betts Laboratory Safety Officer	5/30/97 Date
Chuck Hoover Laboratory QA/QC Officer	5/30/97 Date

(Effective Date is 5 calendar days after the last signature above - QA/QC Officer)

Document Status

[ ] Controlled DC No. _____ Issued to: _____ Date: _____

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1.0 DAILY MAINTENANCE (ALS 2032/20116)

1.1 Remove test tubes, and using a syringe, flush clean water through purge needles. Note any discrepancies in the previous nights run sequence.

1.2 Wipe off needles lines if soils were analyzed.

1.3 Begin purging each chamber.

1.4 If a sample contained target compounds or tentatively identified compounds at high levels, they may have contaminated the chamber and/or lines. Place a test tube in that position and "Step to Bake" with the Bake Gas Bypass "off". Bake for several minutes. If sample(s) were highly contaminated, it may be necessary to run blanks in those chambers until no trace of carryover is evident.

1.5 Soil samples should be analyzed with the Bake Gas Bypass "on" which means no gas flows through the chamber during the bake cycle. This will decrease the chances of soil getting into the return lines.

1.6 Soil samples' purge needles (if not true soil needles) should be bent to angle across the test tube. This prevents possible blockage that may occur during routine soil analysis.

2.0 DAILY MAINTENANCE (PRECEPT II)

2.1 Remove VOA vials from tray, noting any discrepancies in the previous night's run sequence.

2.2 Check level of standard(s) on precept and make new standards if necessary. Prime any new standards added to the Precept before analysis.

2.3 Check water level in independent water storage tanks and refill if necessary.

2.4 Check nitrogen level in tank (used to pressurize water tanks) and replace if necessary.

2.5 Check water drain storage container and empty if necessary.

3.0 PREVENTIVE MAINTENANCE

3.1 Little operator maintenance is required beyond daily clean-up.

3.2 The trap is changed as required by protocol or as it degrades and compounds are no longer trapped efficiently.

C1-3

PROCEDURAL DOCUMENTS - SYSTEM TRANSITION

Please note that the following document is in transition from a previous system. The document identification number (listed below) has been assigned in compliance with the SWL SOP titled:

"Preparation, Review, Revision, and Control of Procedural Documents", SWL-GA-101, Rev. 1.0 — 05/14/97

The ID given below integrates the following SOP into the new system set forth in GA-101 prior to its annual review (or conversion of format and numbering). Each ID is unique and a "key" field in the new document control database system. The assignment of a new number is therefore necessary for its tracking in the new system.

Upon revision or review, the following document will be re-issued in full compliance with the guidelines set forth in GA-101, therefore consistent with newly converted or written procedural documents.

Existing ID: SWL-GENPREV

NEW DOCUMENT IDENTIFICATION NUMBER:

SWL-GA-127

Standard Operating Procedure

"Preventive Maintenance"
Rev. No. 2.1
Issued: 05/14/97

C1-4

SOUTHWEST LABORATORY OF OKLAHOMA, INC.

TM

Standard Operating Procedure

General Preventive Maintenance

Document No.: SWL-GENPREV

Rev No. / Date: Rev. 2.1 — 05/14/97

APPROVALS	DATES
Procedure Prepared By	Date
Program Manager or Section Supervisor	5-15-97 Date
Laboratory Safety Officer	5/16/97 Date
Laboratory QA/QC Officer	5-15-97 Date

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I. SUMMARY

Preventive maintenance is defined as an orderly program of positive actions for preventing failure of equipment and ensuring that the equipment is operating with the reliability required for quality results. The actions include specification checks, calibrating, cleaning, lubricating, reconditioning, adjusting and checking.

II. FIELD EQUIPMENT

A. Summary

Preventive maintenance is carried out on all field equipment prior to being shipped to the sampling site. This preventive maintenance includes regular battery checks and maintaining a sufficient stock of spare parts and supplies. Field personnel are strongly cautioned that these instructions are for general purpose only. Should equipment break down in the field and field crews are unable to repair the equipment within a reasonable amount of time, the operations manager in the laboratory is notified. A replacement is shipped immediately via overnight courier. Whenever possible, duplicates of all equipment are initially sent to the field. For specific preventive maintenance procedures, the appropriate instrument manual should be consulted.

B. pH Meter

The following is a description of the preventive maintenance procedures for the field pH meters:

- Charging Batteries

After the initial charge when first placing the instrument in operation, the batteries are recharged after each 30 hours of operation. Allow the batteries to charge 16 hours to restore them fully. Exceeding the 16-hour period will not damage the batteries. Overnight charging is recommended, and periods of operation between charges should not exceed 30 hours. With proper charging practices, a set of batteries should last for more than 300 charge cycles.

- Battery Replacement

C1-7

When batteries no longer hold a charge for a reasonable length of time, they should be replaced. This unit requires six AA size nickel-cadmium batteries. Replace them as follows:

- Remove the accessories from the foam insert above the instrument panel
- Remove the four screws securing the panel in the case
- Lift the panel from the case and place it face down on a padded surface
- Pry the batteries from their clips with a screwdriver and replace all six batteries
- Replace the panel in the case, and replace the accessories in the foam insert
- Connect the charge unit to the instrument and allow the batteries to charge for 15 hours.

- pH Electrode Care Storage

When the electrode is not in use, the wetting cap with filling solution-soaked cotton should be reinstalled over the tip, and the fill hole cover should be placed over the hole. This will prevent loss of filling solution through evaporation. Always maintain the filling solution level just below the fill hole.

- pH Electrode Cleaning

Normal cleaning of the electrode can be performed in the following manner:

- Immerse the electrode tip in 0.1N HCl followed by immersion in 0.1N NaOH and again in 0.1N HCl, each for a 2 minute period. Rinse with ASTM Type II reagent water and soak in pH 7 buffer solution for 30 minutes.

- If the electrode is slow to respond or readings are unstable and the connection cannot be remedied with

normal cleaning, the reference junction may be clogged. Clean the junction for 10 minutes in dilute potassium chloride solution. First dilute a saturated potassium chloride solution about 1:10 with water. Place the electrode tip in the boiling solution for about 10 minutes.

• Remove heat and allow the electrode to cool while immersed in the solution. Then rinse with ASTM Type II reagent water and soak in pH 7 solution before testing again.

If these steps fail to improve electrode response, replace the electrode. If the pH bulb becomes contaminated or left dry, it may be reconditioned by following the cleaning procedure above.

C. Specific Conductance Meter

The following is a description of the preventive maintenance procedures for the field specific conductivity meters:

• Battery Replacement

Low battery condition is indicated by an arrow on the display. When the arrow appears, the battery should be replaced.

D. Thermometer

After each use, the thermometer probe should be rinsed with ASTM Type II reagent water. Should the sample contain oils or other heavy hydrocarbon mixture, the probe should be washed with laboratory-grade detergent and rinsed with ASTM Type II reagent water.

III. LABORATORY INSTRUMENTATION

A. Summary

A preventive maintenance program for the instrumentation ensures fewer interruptions of analyses, personnel efficiency, and lower repair

costs. It eliminates premature replacement of parts, and reduces discrepancy among test results. It increases reliability of results.

The laboratory has established the following preventive maintenance program:

1. Each type of equipment/instrument has a written Standard Operating Procedure (SOP) which describes the methods for routine inspection, cleaning, maintenance, testing, calibration, and/or standardization of the equipment. Instrument operating manuals are kept near the instrument or where analysts have easy access.

2. Analysts using the instruments are properly trained and develop trouble-shooting skills in equipment failure to reduce dependence upon outside servicing agencies. In complicated cases, the servicing agency or supplier is called to solve the problem.

3. Written equipment records are kept to document all inspection, maintenance, trouble-shooting, calibration, or modifications. Whenever maintenance is performed on an instrument, it is properly documented in a preventive maintenance logbook, which is kept near the equipment to monitor the adequacy of maintenance schedules. The records contain the date (month, day, year), description of the maintenance done, the actual findings, the name of the person doing the maintenance and a statement of whether the maintenance operations were routine, and if those operations followed the written SOP.

4. Performance criteria is established for judging when data from instrument performance checks indicate the need to make adjustments in the instrument operating conditions. (See Section 4.0, Calibration Procedures and Frequency for details)

B. Chromatographic Instruments

Preventive maintenance is done through a daily performance check and calibration of standards. Parameters such as retention time and response factors are observed and back-checked with prior operational performance.

In addition, the following are done:

C-1-10

- GC detectors are cleaned whenever performance degradation (i.e., calibration criteria are not met, retention time shifts, noisy baseline, etc.) is observed by the analyst.

- Septa are replaced as needed.

- Incoming gas drying cartridges are changed whenever the color of the adsorbent is noticed.

- Effluent adsorbent traps are changed as needed.

- Columns (GC and HPLC) are checked by performance and operating conditions when in use or prior to use.

- Oven performance checked daily prior to use.

C. Gas Chromatography/Mass Spectroscopy (GC/MS)

The preventive maintenance includes:

Maintenance Requirements	Frequency
Clean filters on cooling fans	Monthly
Check cooling fan for proper operation	Monthly
Check cool-flow level	Monthly
Change mechanical pump oil	Every 6 months
Clean source and rods	As needed
Check power supplies in QEM Box	As needed
Clean printer inside and outside	As needed
General cleaning of instrument	As needed
All items from monthly maintenance schedule	As needed
Sensitivity analysis through BFB and DFTPP tune criteria	Every 12-hour clock

D. Atomic Absorption Spectrophotometers/ICP

Preventive maintenance is done for atomic absorption through the following checks:

- Minimum warm-up period of 30 minutes

- Alignment of hollow cathode tube to produce the maximum emitted light to the detector

- For flameless AA, the inert gas flow inside the furnace is optimized to ensure maximum sensitivity

- Digital readout values obtained for the standard curve of each element are checked to ensure linearity

- If readings are low, the operator checks the gas flows, burner or cell alignment, wavelength, slit width, photomultiplier voltage and lamp intensity prior to analysis

E. General Laboratory Equipment

Analytical balances of various capacities and operational modes are calibrated annually by a licensed specialist and officially recorded as verification of performance.

Balances are checked with standard Class S calibration weights before use. The pH/specific-ion meters are calibrated before use with a minimum of two standard solutions. All combination pH electrodes will be stored in pH 4 buffer solutions.

TCLP Extraction Equipment shall be checked as follows:
-Rotory Extractor-RPM checked monthly (28-32 rpm)
-ZHE's-Viton O-rings checked prior to use; replaced if worn and or ZHE has pressure problems.
-2L Extraction vessels-check lip for cracks (upon use).
-Teflon Liner on lids-check for (cracks/tears) upon use.

SOUTHWEST LABORATORY OF OKLAHOMA, INC.

Standard Operating Procedure

Preventative Maintenance of Gas Chromatographs

Document No.: SWL-OP- 201

Rev No. / Date: Rev.1.3 — 05/30/97

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APPROVALS	DATES
Procedure Prepared By	5/30/97 Date
Organic Program Manager	5/30/97 Date
Laboratory Safety Officer	5/30/97 Date
Laboratory QA/QC Officer	5/30/97 Date

(Effective Date is 5 calendar days after the last signature above - QA/QC Officer)

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1.0 Scope and Application

This document provides guidance for the scheduling, performance, and documentation of routine maintenance for Hewlett-Packard and Fisons gas chromatographs.

2.0 Method Summary

2.1 Preventative maintenance is performed as scheduled in this document or on an as needed basis if evaluation reveals that preventative maintenance is not necessary.
2.2 Subsequent to every maintenance, all work performed will be documented in the "instrument maintenance log" specific to that instrument.

2.2.1 Document: Problems noted, maintenance performed, H_2 instruments were thoroughly leak tested, date of maintenance, date of return to analytical control, and initials of the one performing maintenance.

3.0 Health and Safety

3.1 See the Standard Operating Procedure for Safety.
3.2 Special precautions must be taken when using H_2 as the carrier or auxiliary gas. Care should be taken to discharge any static electricity buildup by grounding before working on or adjusting an instrument where H_2 is employed. Thorough leak checking will be performed after every maintenance and documented in the "instrument maintenance log."

4.0 Sample Preservation, Containers, Handling & Storage

N/A

5.0 Interferences and Potential Problems

N/A

6.0 Equipment/Apparatus

6.1 Replacement parts including but not limited to, syringes, septum, inlet liners, inlet seals (gold seals), column nuts, weldmet assemblies, split/purge valves, needle valves, various ferrules, columns, pencil traps, ECDs, heating elements, cooling and oven fans, and integrated circuit boards.

6.2 Tools and apparatus may include but are not limited to, metric and standard crescent wrenches, column cutters, dental picks, Philips and flat head screw-drivers, flow meters, leak detectors, bubble meters (not to be used for H_2), and other instrument-specific tools.

7.0 Reagents

N/A

8.0 Procedure

8.1 Tables 1 and 2 present a schedule for preventative maintenance/part replacement for all Hewlett-Packard Gas Chromatographs. Tables 3 and 4 present a schedule for preventative maintenance/part replacement for all Fisons Gas Chromatographs. The schedules will be followed unless instrument evaluation reveals that a particular maintenance is unnecessary.

8.1.1 HP Maintenance.

Table 1.Gas Chromatograph Preventative Maintenance/Replacement Schedule (HP)

ITEM	REPLACEMENT TIME	TROUBLESHOOTING/COMMENTS
Activated Charcoal Trap	Every month or sooner.	Traps potentially dangerous compounds after they have passed through the GC detectors.
Column	As needed	A new column is required if: resolution falls below 60%, if breakdown (not caused by other problems) cannot be removed by cutting off the front portion of the guard column or any other problem traced to the column.
Detector	As needed	Refurbishing is recommended when the detector signal rises 100% above the original signal or the baseline will not

		stabilize. Baking the detectors with hydrogen and/or cleaning the detector anode may make refurbishing unnecessary.
Detector Nut Ferrule	3 months or as needed	Retention time shifts may be indicative of a faulty or old detector nut ferrule.
Injection Liner (& O-ring)	2 weeks or as needed	To ensure contaminant-free chromatography and to prevent analyte breakdown.
Injection Port	As needed	To ensure contamination-free chromatography.
Injector Seal (gold)	1 month or as needed	To ensure contamination-free chromatography and reduce analyte breakdown. Discoloration of the gold seal indicates contamination (Usually needed sooner than 1 month).
Injector Seal Washer	6 months or as needed	To ensure contamination-free chromatography.
Needle Valve	As needed	Auxiliary flow fluctuations are indicative of a faulty needle valve.
Pencil Trap	1 year or as needed	To ensure contamination-free chromatography.
Purge Valve	1 year or as needed	Poor linearity and random continuing standard linearity is indicative of a clogged purge valve; especially if the early and late eluting compounds of the standard are exhibiting exceedingly contrasting percent differences.
Rinse Vials	6 months or as needed	Rinse vial solvent should be filled and waste vials should be emptied daily. If contamination is suspected, rinse vials well with solvent and make a test injection. If the contamination persists, replace the vials.
Septum	Before every sequence	To ensure contamination-free chromatography.
Septum Nut	1 year or as needed	To ensure contamination-free chromatography.
Split Line	6 months or as needed	To ensure contamination-free chromatography; also to prevent clogging of the purge valve with heavier organics back-flushed into the lines.
Syringe	1 month or as needed	To ensure contamination-free chromatography. Needed when the syringe plunger becomes difficult to move or when contamination traced to the syringe is noted.

Table 2. Miscellaneous Gas Chromatograph Preventative Maintenance Schedule (HP)

MAINTENANCE	TIME	COMMENT(S)
Auxiliary Flow Check	2 weeks or when a flow problem	To ensure gas flow stability and to help foresee other problems.

	is suspected.	
Column Cutting	As needed	When analyte breakdown exceeds method acceptance criteria.
Column Flow Check	1 month or as needed	To ensure gas flow stability and to help foresee other problems.
Injection Port Rinse	As needed	To ensure gas flow stability and to help foresee other problems.
Split Flow Check	As needed	To ensure gas flow stability, to prevent wasting gas, and to help foresee other problems.
Purge Flow Check	As needed	To ensure gas flow stability, to prevent wasting gas, and to help foresee other problems.

8.1.1.1 Subsequent to every maintenance, the following are required:

8.1.1.1.1 Leak-check the instrument around every area that was serviced. Thorough leak testing, including all possible points of leaking in the oven and surrounding anywhere near where the instrument was manipulated.

8.1.1.1.2 Complete the "instrument maintenance log". All work performed will be documented in the "instrument maintenance log" specific to that instrument.

8.1.1.1.2.1 Document: Problems noted, maintenance performed, H_2 instruments were thoroughly leak tested, date of maintenance, date of return to analytical control, and initials of the one performing maintenance.

8.1.1.1.2.2 Hewlett-Packard service persons are also required to complete the instrument maintenance log (the person assigned that particular instrument is responsible for ensuring all appropriate entries).

8.1.1.1.3 Allow for equilibration time before beginning an analysis sequence. Injection of a column conditioner and several solvents before beginning an analytical sequence promotes stability and is highly recommended.

8.1.1.1.4 Check detector signals for stability.

8.1.2 Fisons Maintenance.

Table 3. Gas Chromatograph Preventative Maintenance/Replacement Schedule (Fisons)

ITEM	REPLACEMENT TIME	TROUBLESHOOTING/COMMENTS
Activated Charcoal Trap	Every month or sooner	Traps potentially dangerous compounds after they have passed through the GC detectors.

Column	As needed	A new column is required if: resolution falls below 60%, if breakdown (not caused by other problems) cannot be removed by cutting off the front portion of the guard column or any other problem traced to the column.
Detector	As needed	Refurbishing is recommended when the detector signal rises 100% above the original signal, when the baseline will not stabilize, and/or when linearity can no longer be established. Baking the detectors with hydrogen and/or cleaning the detector anode may make refurbishing unnecessary.
Injection Liner (& Tenuda)	2 weeks or as needed	To ensure contaminant-free chromatography and to prevent analyte breakdown.
Injection Port	As needed	To ensure contamination-free chromatography.
Split Valve	As needed	Non-reproducible injections, including poor linearity and random continuing standard linearity may indicate flow fluctuations due to a faulty needle valve; especially if the early and late eluting compounds of the standard are exhibiting exceedingly contrasting percent differences.
Gas Valves	1 year or as needed	Loss or random fluctuation of carrier or auxiliary flow is indicative of a clogged valve.
Rinse Vials	1 month or as needed	Rinse vial solvent should be filled and waste vials should be emptied daily. If contamination is suspected, rinse vials well with solvent and make a test injection. If the contamination persists, replace the vials. Note: Fisons instruments are highly susceptible to contamination from the rinse vials.
Septum	Before every sequence	To ensure contamination-free chromatography.
Syringe	1 month or as needed	To ensure contamination-free chromatography. Needed when the syringe plunger becomes difficult to move or when contamination traced to the syringe is noted.

Table 4. Miscellaneous Gas Chromatograph Preventative Maintenance Schedule (Fisons)

MAINTENANCE	TIME	COMMENT(S)
Auxiliary Flow Check	2 weeks or when a problem is suspected.	To ensure gas flow stability and to help foresee other problems.
Column Cutting	As needed	When analyte breakdown exceeds method acceptance criteria.
Column Flow Check	1 month or as needed	To ensure gas flow stability and to help foresee other problems.

Injection Port Rinse	As needed	To ensure gas flow stability and to help foresee other problems.
Split Flow Check	1 month or as needed	To ensure gas flow stability, to prevent wasting gas, and to help foresee other problems.
Purge Flow Check	1 month or as needed	To ensure gas flow stability, to prevent wasting gas, and to help foresee other problems.

8.1.2.1 Subsequent to every maintenance, the following are required:

8.1.2.1.1 Leak-check the instrument around every area that was serviced. Thorough leak testing, including all possible points of leaking in the oven and surrounding anywhere near where the instrument was manipulated.

8.1.2.1.2 Complete the "instrument maintenance log". All work performed will be documented in the "instrument maintenance log" specific to that instrument.

8.1.2.1.2.1 Document: Problems noted, maintenance performed, H_2 instruments were thoroughly leak tested, date of maintenance, date of return to analytical control, and initials of the one performing maintenance.

8.1.2.1.2.2 Fisons service persons are also required to complete the instrument maintenance log (the person assigned that particular instrument is responsible for ensuring all appropriate entries).

8.1.2.1.3 Allow for equilibration time before beginning an analysis sequence. Injection of a column conditioner and several solvents before beginning an analytical sequence promotes stability and is highly recommended. Note: If the detectors were opened or the detector end of the column was removed, then equilibration times are greatly increased (up to 1 day).

8.1.2.1.4 Check detector signals for stability.

9.0 Calculations

N/A

10.0 Quality Assurance/Quality Control

N/A

11.0 Waste Disposal

See Waste Disposal SOP

12.0 Method Performance

N/A

13.0 References

N/A

14.0 Definitions

Instrument Maintenance Logbook – A permanently bound, sequentially numbered logbook used for detailing instrument problems, maintenance performed, and return to analytical control. One logbook must be kept for every instrument.

Leak testing -- A systematic search for carrier or auxiliary gas leaks in a pressurized gas chromatographic system. The Gow Mac, 21-250 gas detection system is used in the Gas Chromatography Laboratory. Follow all manufacturer's instructions.

N/A = not applicable.

Request for New or Revised Procedure, SWL-GA-101

SWL SOP Rev. # 1.0 - 05/14/97; Form ID: GA101-DOCREQ-F: Location/File ID: F:\SOP\GA\101-ATT5

REVISED (includes Forms/Logs) Submitted By: Brett [illegible] Date: 6/18/97

Document Title: PREVENTATIVE MAINTENANCE OF GAS CHROMATOGRAPHS

Document No.: SWL-0P-201 Rev. No. 1.3 — 05/30/97

Reference: Page 4/6 of 7/7 Section: 15.0/8.0; Subpart: 15.1/8.1.1.1 / 8.1.2.1

Attachment No. 1 — Title: GC LAB INSTRUMENT MAINTENANCE LOG

Brief Description (need for revision): 1) MAKE MORE SPECIFIC REFERENCE TO MAINTENANCE LOG IN SECTIONS 8.1.1.1 & 8.1.3.1 ; 2) ATTACH COPY OF GC LAB INST. MAINTENANCE LOG.

Approved: [signature] Dated: 6/18/97 Denied: ☐ PM/SS Initials:_____
Program Manager/Section Supervisor (see attached explanation)

NEW (Procedural Document) Submitted By:_____ Date:_____

Justification for New Procedure: _____

Approved:_____ Dated:_____ Denied: ☐ PM/SS Initials:_____
Program Manager/Section Supervisor (see attached explanation)

Author: _____ Assigned By: _____ Date: _____

Review/Approval by Manager/Supervisor

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Date Rec'd by Document Control:_____ Initials:_____

GC Lab Instrument Maintenance Log

SWLO-OP-201 OP-201-MNTLOG-1 SWL Rev. #: _____

Maintenance performed and reason.	Date Maint. Performed	Analyst Initials	Date of Return to Analytical Control

C1-23

SOUTHWEST LABORATORY OF OKLAHOMA, INC. / AMERICAN ANALYTICAL & TECHNICAL SERVICES, INC.

STANDARD OPERATING PROCEDURE FOR THE INORGANIC PREVENTIVE MAINTENANCE SCHEDULE

INPREMA

REV 1.0 — 8/10/96

Document Control #AATSWL-INPREMA- 2033 2/6/98 Alan Fosdick MM.

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Signature: Chuck Hoover

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I. PURPOSE

A. The Inorganic Preventive Maintenance Schedule Logbook, which is the planned periodic and systematic care and servicing of the laboratory equipment. This element prevents, detects, and corrects possible problems leading to equipment failure or erroneous measurements.

B. Provide information on all observations, maintenance, problems, or changes in the equipment operation that may affect the quality of data that is produced.

II. PROCEDURE

A. The following instruments use the listed Preventive Maintenance Schedule in Figures 1-7.

1. TJA ET1, TJA ET2, and TJA 61E - Figure 1.
2. PS200A and PS200B - Figure 2.
3. Lachet - Figure 3.
4. VAR400A, VAR400B, and PE5100B - Figure 4.
5. DX300 and DX2010i - Figure 5.
6. Shimadzu TOC 5050 - Figure 6.
7. Leco CR-12 TOC - Figure 7.

B. Instructions for the Preventive Maintenance Schedule

1. In the "ANALYST" field enter your initials.
2. In the "DATE" field enter the current date.
3. Perform and check off all "DAILY" maintenance items each day the instrument is used.
4. Perform and check off all "WEEKLY" maintenance items on one of the following days: Tuesday, Wednesday, or Thursday.
5. Perform and check off all "MONTHLY" maintenance items in the first 10 days of each month.
6. Perform and check off all "SEMI(BI)-ANNUALLY" maintenance items in the last 10 days of April and November.

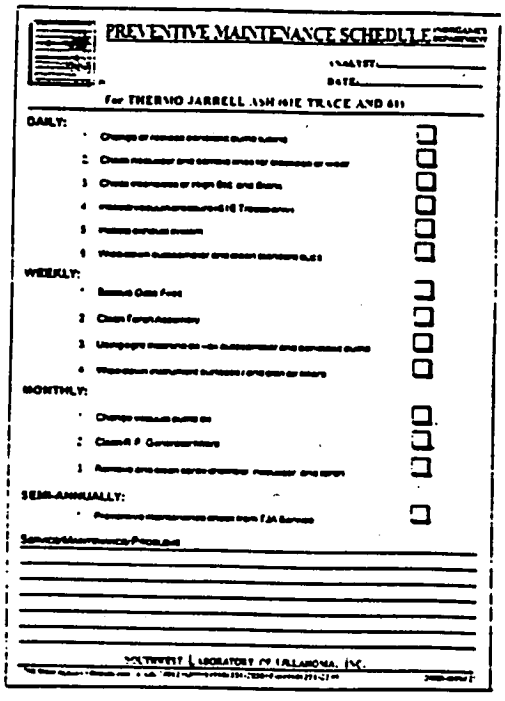
C. The following information should be written under the heading of "SERVICE \ MAINTENANCE \ PROBLEMS":

1. Corrective maintenance, which is any immediate maintenance that is performed because of sudden changes in equipment performances.
2. Any major replacements, repairs, adjustments, or alignments that have been completed by a service engineer or the analyst.

3. Minor alignments, adjustments, and item replacements that bring the equipment to a ready state.
4. Subtle changes or trends in equipment operation that may lead to possible equipment failure or erroneous measurements.
5. Equipment failures and malfunctions.
6. Significant events, such as electrical shorts, drops, etc., which could adversely affect performance.

III. FIGURES

Figure 1 - ICP


PREVENTIVE MAINTENANCE SCHEDULE

DAILY:

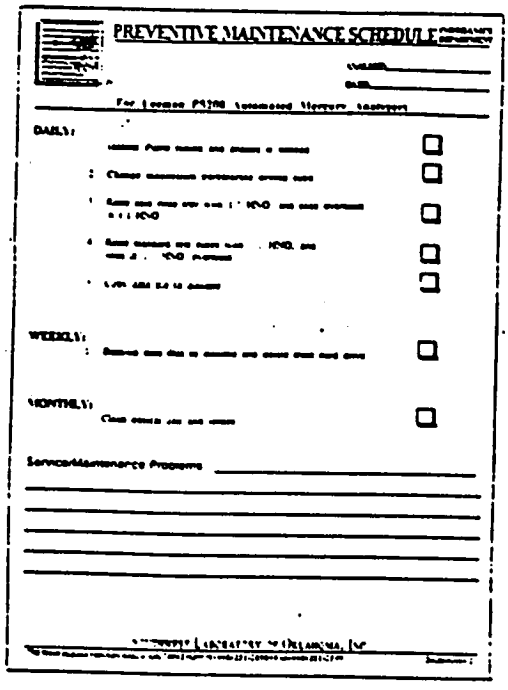
WEEKLY:

MONTHLY:

SEMI-ANNUALLY:

Service/Maintenance Problems

Figure 2 - Leeman PS200


PREVENTIVE MAINTENANCE SCHEDULE

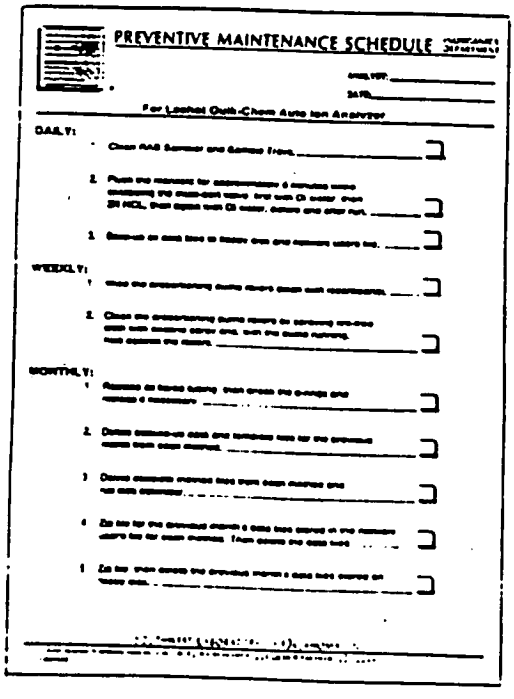
DAILY:

WEEKLY:

MONTHLY:

Service/Maintenance Problems

Figure 3 - Lachet

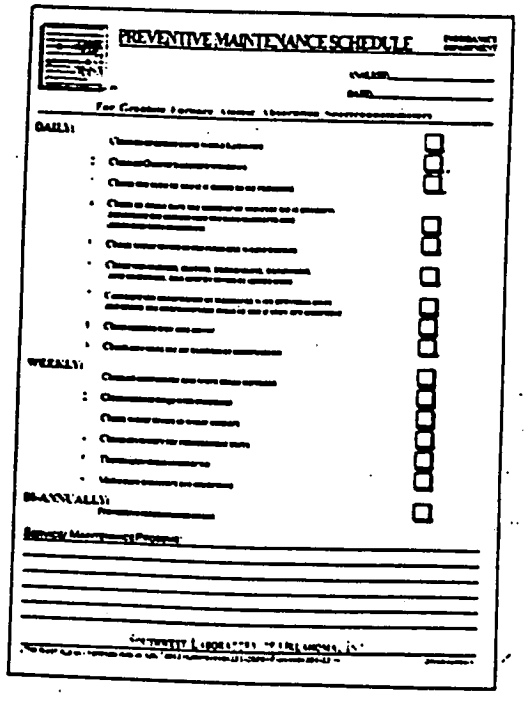

PREVENTIVE MAINTENANCE SCHEDULE

DAILY:

WEEKLY:

MONTHLY:

Figure 4 - GFAA


PREVENTIVE MAINTENANCE SCHEDULE

DAILY:

WEEKLY:

SEMI-ANNUALLY:

Service/Maintenance Problems

Figure 5 - DX300 & DX2010i

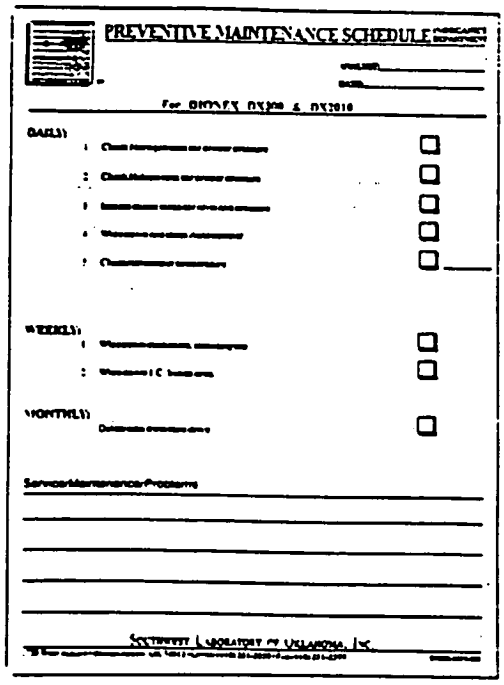


Figure 6 - Shimadzu TOC 5050

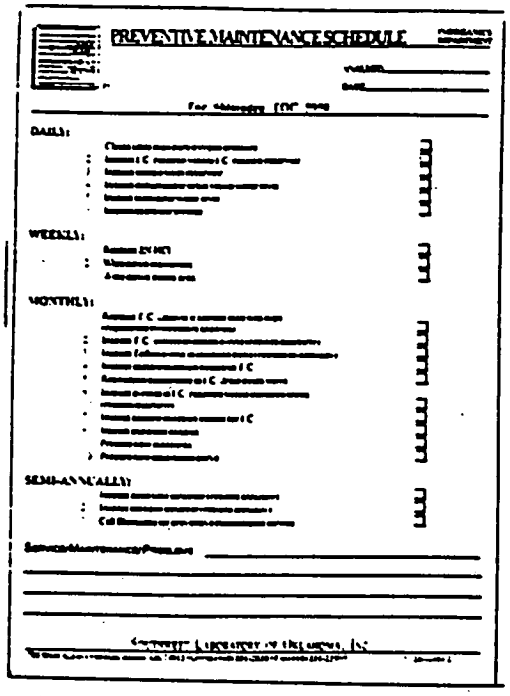
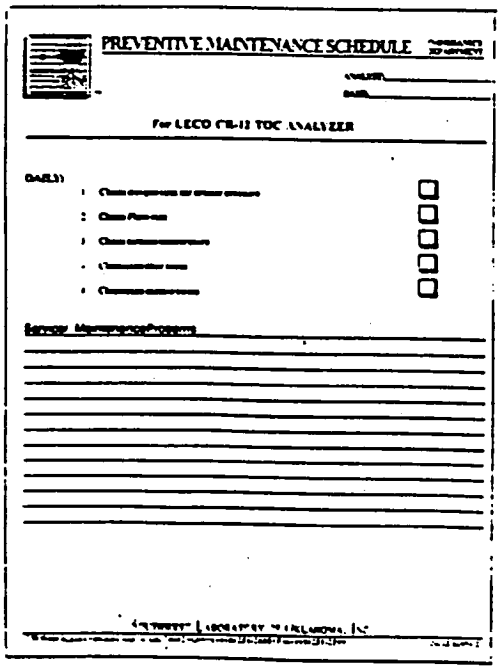


Figure 7 - Leco CR-12



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SECTION 2
STANDARD OPERATING PROCEDURES FOR ANALYSIS

SOUTHWEST LABORATORY OF OKLAHOMA, INC.

Standard Operating Procedure

Analysis of Volatile Organics by GC/MS SW846 Method 8260A

Document No.: SWL-OV-303

Rev No. / Date: Rev. 3.1 — 07/23/97

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GC/MS ANALYSIS OF PURGEABLE ORGANICS

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GC/MS ANALYSIS OF PURGEABLE ORGANICS

1.0. SCOPE AND APPLICATION

1.1 Method 8260A, test code MS326, is a purge-and-trap, gas chromatograph/mass spectrometer procedure used to determine volatile organic compounds in many different types of matrices regardless of water content. It can be used to quantitate most volatile organic contaminants with boiling points less than 200°C that are insoluble or only slightly soluble in water. It can also be used for the quantitation and analysis of the more soluble organic compounds such as ethers, acetates, acrylates, and ketones, but with quantitation limits increased accordingly due to poor purging efficiency. In general, for most contaminants, the reporting limit for this method for any one compound is 5 µg/kg for soil/sediment samples 5 µg/L for ground water samples, and 0.5 mg/kg for wastes. Clearly, reporting limits will be higher for sample extracts or samples requiring dilution due to high concentrations of contaminants.

1.2 The reporting limits and compounds listed in Table 7 are normal limits and compounds analyzed by this method.

2.0 METHOD SUMMARY

2.1 An inert gas, helium, is bubbled through either an aqueous sample or heated solid sample contained in a specifically designed purging chamber at ambient temperature. The purgeables are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column where the purgeables are trapped. After purging is completed, the sorbent column is heated and backflushed with the helium to desorb the purgeables onto a gas chromatographic column. The gas chromatograph is temperature programmed to separate the purgeables that are then detected with a mass spectrometer.

2.2 The amount of sample analyzed will vary depending upon the nature of the sample (aqueous or nonaqueous) and the desired quantitation limits of the analysis. For normal aqueous samples, a 5mL aliquot is used. For normal nonaqueous solid samples, 5.0 grams plus 5 mL filtered tap water will be analyzed.

2.3 For aqueous samples, an aliquot of the sample is diluted with filtered tap water when dilution is necessary. A 5 mL aliquot of the dilution is taken for purging. For nonaqueous samples, as little as 1.0 gram may be used for low-level diluted analyses. For medium-level analyses, 4.0 grams of sample is extracted in 10 mL methanol, and up to 100 uL of the extract may be used.

2.4 If the amount of a compound detected is outside the linear range of the instrument, then a smaller sample size is analyzed.

2.5 All samples are analyzed within 14 days of the sampling date. (See Section 4.1 for details concerning sample preservation)

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3.0 HEALTH & SAFETY

Some of the reagents solvents, and other chemicals used in this method have been clearly defined as toxic and/or carcinogenic; most, however, remain in question or are not precisely defined with respect to the hazards of handling. All chemicals and samples, therefore, are treated as potentially hazardous, and exposure to them is minimized. Also, the MSDS for all stock solutions, reagents, and solvents used are kept on file in an easily accessible place in the lab. Any spills that may occur are cleaned up immediately.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING & STORAGE

4.1 All water samples are to be preserved in the field using HCl. Verification of this preservation is performed by checking the pH of all water samples and recording their pH on the Volatile GC/MS pH Form. (Figure 1)

4.2 Samples are stored at 4° +/- 2° C. Standards are stored at –10° to –20°C. All samples and standard solutions must be allowed to warm to ambient temperature before analysis.

5.0 INTERFERENCES AND POTENTIAL PROBLEMS

5.1 Many major sources of contamination are avoided by using stainless steel, nickel, polytetrafluroethylene (PFTE), glass, and other inert materials for all equipment and materials that in any way affect the detection and quantitation of volatile contaminants in samples. However, other areas of contamination do exist, and are avoided as follows:

5.1.1 Samples can sometimes be contaminated by diffusion of volatile organics through the septum seal during shipment and storage; this can be handled by the use of a trip blank prepared from organic-free filtered tap water which is carried through the entire sampling and shipping process with all samples, in addition to holding blanks prepared in the same fashion and stored in all areas where samples will be stored in the laboratory. Our laboratory encourages the analysis of a trip blank and sends trip blanks to clients when providing a bottle order for sampling.

5.1.2 Interfering contamination can occur when a sample is analyzed immediately following a sample containing high concentrations of volatile organic compounds. All such samples are reviewed carefully for any indication of carryover and reanalyzed when there is any question of whether contaminants found in the sample are the result of true contamination or originate from the previous analysis.

5.1.3 Common laboratory solvents present in the air of most analytical laboratories, notably methylene chloride, acetone, and chloroform, can cause contamination of the analytical instruments themselves; this can be checked by analyzing an instrument blank, where the presence of volatile contaminants can alert the analyst of the need to perform any cleaning measures necessary to achieve an analytical run which is as contaminant-free as possible.

6.0. APPARATUS AND MATERIALS

6.1 Micro syringes — 10 µL and larger, 0.006 inch ID needle.

6.2 Syringe valve — two-way, with Luer ends (three each), if applicable to the purging device.

6.3 Syringe — 5 mL, gas tight.

6.4 pH Paper — Range of 1-6

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

6.6 Glassware

6.6.1 Bottle — 40 mL, screw cap, with Teflon cap liner.

6.6.2 Volumetric flasks — class A with ground-glass stoppers.

6.6.3 Vials — 20mL for medium level extraction

6.7 Purge and trap device — The purge and trap device consists of three separate pieces of equipment; the sample purger, trap and the desorber. Heating blankets are added to the Tekmar 2032 and 2016 Autosamplers when low level soils are analyzed. Sample purgers include Tekmar ALS2032, ALS2016, Precept, or equivalent. Trap and desorber include Tekmar 2000 and 3000 or equivalent.

6.8 Gas chromatograph — An analytical system complete with a temperature programmable gas chromatograph suitable for on-column injection and all required accessories including syringes, analytical column, and gases. GC is Hewlett-Packard 5890 Series II or equivalent.

6.9 Column — DB624, 75m, 0.53mm ID, 3.0 um (J&W Scientific Cat. No. 125-1334).

6.10 Jet Separator — Glass (SIS Cat. No. 113505).

6.11 Mass spectrometer — Capable of scanning from 35 to 300 amu every one second, utilizing seventy volts (nominal) electron energy in the electron impact ionization mode and producing a mass spectrum which meets all the criteria when 50 ng of 4-Bromofluorobenzene (BFB) is injected through the gas chromatograph inlet (see Table 3). MSD is Hewlett-Packard 5970, 5971, 5972, or equivalent.

6.12 Calibration requirements can be found in Table 6.

7.0 REAGENTS AND STANDARDS

7.1 Reagent water — Produced by filtering tap water through an organic filtration system containing activated charcoal prior to use.

7.2 Methanol — Pesticide quality or equivalent.

7.3 Stock standard solutions — Stock standard solutions are purchased and are traceable to EPA supplied standards. These solutions are stored in Teflon sealed screwcap bottles at -10°C to -20°C.

7.4 Secondary dilution standards — Using commercially prepared standard solutions, secondary dilution standards that contain the compounds of interest, either singly or mixed together, are prepared in methanol. Secondary dilution standards should be stored with minimal headspace and should be checked frequently for signs of degradation or evaporation especially just prior to preparing calibration standards from them. Secondary dilution standards are used until degradation is verified through daily calibration checks.

7.5 Internal Standard and Surrogate Spiking Solution — The internal standards are Pentafluorobenzene, 1,4-Difluorobenzene, Chlorobenzene-d_5, and 1,4-Dichlorobenzene-d_4. The surrogates are Dibromofluoromethane, Toluene-d_8, and 4-Bromofluorobenzene. An additional surrogate, 1,2-Dichloroethane-d4 may also be added. Separate internal standard and surrogate standard solutions are prepared containing the above at a concentration of 25 µg/mL in methanol.

8.0 PROCEDURE

8.1 This method requires the following instrumental parameters:

8.1.1 Electron Energy: 70 Volts (nominal)

8.1.2 Mass Range: 35-300

8.1.3 Scan Time: to give at least 5 scans per peak, not to exceed one second per scan

8.2 Recommended Operating conditions for the gas chromatograph and column conditions: DB-624, 75m, 0.53mm ID, 3 µm film thickness at a flow rate of 30 mL/min. Column temperature is isothermal at 45°C for five minutes, then programmed at 5°C per minute to 105°C. further programmed at 10°C/min. to 180°C.

8.3 Adjust the purge gas (helium) flow rate to 35 ± 3 mL/min on the purge and trap. Variations from this flow rate may be necessary to achieve better purging and collection efficiencies for some compounds, particularly chloromethane and bromoform.

8.4 Tune

8.4.1 Before analysis of samples can begin, the instrument must meet all calibration criteria. The first step in the calibration of a Volatiles GC/MS system is to demonstrate the ionization and fragmentation of standard mass spectral tuning compounds. This, as well as a sensitivity check, is accomplished with the use of 4-Bromofluorobenzene, an EPA specified compound. This standard is run daily to validate the GC/MS system and to begin the 12-hour clock. BFB is analyzed by direct injection of a 50-ng aliquot into the instrument.

8.4.2 When the analysis is complete, the tune is checked against standard criteria (See Table 3 for Tune Criteria). SW8260 tune criteria for BFB must be met before any other calibration standards or sample analysis is performed.

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8.4.3 If the tune fails to meet the criteria, BFB is rerun. If it fails again, the instrument must be retuned through "Auto Tune" or "Manual Tune," then BFB should be run again. If all criteria are met, the calibration sequence is run, but if it fails again, a supervisor is contacted because instrument maintenance such as source cleaning may be required.

8.5. Initial Calibration

8.5.1 Once BFB has met its tune criteria, calibration may begin. Initial calibration of the GC/MS is established and validated by the analysis of EPA traceable standards at five concentration levels which are 5, 20, 50, 100,and 200 ppb for the 5 mL or 5 gram purge method. These standards are made up in 5 mL of filtered tap water and purged on the Tekmar before analysis by GC/MS. The internal standard calibration procedure is used. In addition to surrogate recovery compounds, standards and sample extracts are spiked with internal standards that span the retention time range of the analytes of interest.

8.5.2 The Response Factors (RF) for the analytes in the standard are calculated and stored in a Target Method File. Using the RFs from the initial calibration, the percent relative standard deviation (%RSD) for all compounds is calculated utilizing the formula shown in Table 9. The criterion the initial calibration must meet to be acceptable is found in Table 6.

8.5.3 If the five-point does not meet criteria, each standard is checked for the need of a possible rerun, in case the standards have been added at the wrong amount, or the purge may have been bad. If reanalyzing a standard will not solve the problem, a supervisor is contacted. Some problems could be found to be related to purge chambers, multiplier, flows, etc.

8.5.4 If the initial calibration does meet the criteria stated in Table 6, and there is time remaining in the 12-hour time period, samples may be run.

8.5.4.1 If the % RSD of any compound is 15% or less, then the relative response factor is assumed to be constant over the calibration range, and the average relative response factor may be used for quantitation.

8.5.4.2 If the % RSD of any compound is greater than 15%, a calibration curve must be constructed of area ratios (A/Ais) versus concentration using first or higher order regression fit of the five calibration points (ie: linear regression or quadratic).

The use of calibration curves is a recommended alternative to average response factor calibration and a useful diagnostic of standard preparation accuracy and absorption activity in the chromatographic system.

8.5.5 The calibration standards are prepared by filling a syringe with filtered tap water and spiking with the standards as follows.

Soil - 5 mL Purge/Tekmar ALS2032/ALS2016

Internal Standard (µL)	Surrogate (µL)	25ppm Stock Std. (µL)	8260 Extra Cmpd.Stock Standard (µL)	Gases Stock Std. (µL)	Int. Std. Final Conc. (ng/mL)	Standard Final Conc. (ng/mL)	Acrolein/ Acrylonitrile Final Conc. (ng/mL)
10	1	1	1	1	50	5	50
10	4	4	4	4	50	20	200
10	10	10	10	10	50	50	500
10	20	20	20	20	50	100	1000
10	40	40	40	40	50	200	2000

Soil-5mL Purge/Tekmar Precept

Surrogate (µL)	25ppm Stock Std. (µL)	Gases Stock Std. (µL)	8260 Extra Cmpd. Stocks Standard (µL)	Int. Std. Final Conc. (ng/mL)	Standard Final Conc. (ng/mL)	Acrolein/ Acrylonitrile Final Conc. (ng/mL)
8	8	8	8	50	5	50
32	32	32	32	50	20	200
80	80	80	80	50	50	500
160	160	160	160	50	100	1000
320	320	320	320	50	200	2000

8.5.6 If analyzing on the Tekmar 2032/2016, the syringe is attached to the syringe valve assembly on the Tekmar. The syringe valve is opened, the sample is injected into the purging chamber, and the purging device valve is closed. Water samples are purged at ambient temperature. Low-level soils are purged at 40° +/- 1°C. If analyzing on the Precept, the standard's 40mL volatile vial is loaded into the tray. The Precept will pick up the vial, and do one of the two things: 1) If the vial is a soil standard, it will transfer the vial from the tray to a heated vial holder, spike it with internal standard, and purge the standard. 2) If the vial is a water standard, it will withdraw 5mL from the vial, spike it with internal standard, and transfer the 5ml to the Tekmar 3000 for purging. The sample is purged for 8.0 min at ambient temperature. At the conclusion of the purge time, the trapped materials are introduced to the GC column by the Desorb Mode where the trap is rapidly heated to 180°C for four minutes.

8.5.7 After the sample desorbs onto the GC, the trap is baked for four minutes at 225°C, then it is allowed to cool to less than 30°C before being ready to purge the next standard or sample. During this time, the Precept flushes its lines and needle.

8.5.8 The standard will be quantitated into a report in which concentrations, areas, response factors, standard deviations, percent differences, and regression coefficients are calculated.

8.6 Calibration Verification

8.6.1 A calibration verification of the GC/MS uses the same EPA traceable standards, but at only one level: 50 ppb. The analysis is performed in the same manner as stated before in the initial calibration. The calibration verification checks the same compounds as the initial calibration, and the criteria the calibration verification must meet to be acceptable are also found in Table 6.

All other compounds should have a %Drift less than 30%, except for the following which should have %Drift less than 60%: Freons, Ketones, 1,2,3-Trichloropropane, 1,2-Dibromo-3-Chloropropane, Naphthalene, Hexachlorobutadiene, and 2-Chloroethylvinylether.

8.6.2 The calibration verification is performed after the BFB and must meet all criteria before any other analysis continues. If the CCC/SPCC criteria are not met, the standard must be rerun, as it is possible, the standard was not prepared correctly or that there was a bad purge. However, if the second standard still doesn't meet CCC/SPCC criteria, an initial calibration must be run or corrective action performed.

8.6.3 If the calibration verification does meet the criteria stated in Table 6, samples may be run. Samples are quantitated based on the criteria stated above in Section 8.5.4.

8.7 Water Samples

8.7.1 Any headspace in the bottle is noted before opening. If analyzing on the Tekmar 2032 or 2016, the sample bottle (which has been allowed to come to ambient temperature) is opened and is carefully poured into the syringe barrel to just short of overflowing, then adjusted to a volume of 5 mL. This process of taking an aliquot destroys the validity of the sample for future analysis. Care must also be taken to prevent air from leaking into the syringe. The entire sample vial is loaded onto the Tekmar Precept.

8.7.2 When spiking samples on the Tekmar 2032 or 2016, 10 µL of the Surrogate Spiking Solution and 10 µL of the Internal Standard Spiking Solution are added through the valve bore of the syringe, which is equivalent to a concentration of 50 µg/L of each standard. If samples are analyzed on the Tekmar Precept, the instrument automatically spikes Internal Standard and Surrogate during analysis.

8.7.3 Water samples are loaded in the following manner to ensure the proper sample is placed into its designated purge chamber on the Tekmar 2032/2016 Autosampler:

- A test tube rack is filled with the samples' 40 mL VOA vials. The syringes to be used are placed in front of the samples.
- All sample purge tubes are labeled with the SWLO ID Number and Autosampler position. Each analyst verifies as he/she loads each sample into the purge chambers the syringe matches the ID on the purge tube.
- The Tekmar Precept allows the analyst to load the sample vials directly on the instrument, reducing the risk of a loading error. Purge in the same manner as stated during calibration.

8.7.4 For water samples analyzed on the Tekmar 2032 or 2016 requiring matrix spike and matrix spike duplicates, 10 µL of the matrix spike solution is added to the sample to be purged. Disregarding any dilutions, this is equivalent to a concentration of 50 µg/L of each matrix spike standard. Water samples analyzed on the Precept requiring MS/MSD, 80µL of the matrix spike solution is added to the sample to be purged, yielding a concentration of 50ug/L.

8.7.5 With all matrices, all dilutions should keep the response of the major constituents (previously saturated peaks) in the upper half of the linear range of the curve.

8.7.6 An estimated concentration for Tentatively Identified Compounds (TICs) shall be quantified by the internal standard method. For quantification, the nearest internal standard shall be used.

8.7.7 The formula for calculating concentrations for target compounds is found in Table 9. For TICs, area counts from the total ion chromatograms are to be used for both the compound to be measured and the internal standard. A response factor (RF) of one (1) is to be assumed. The value from this quantitation shall be qualified as "estimated." This estimated concentration should be calculated for all TICs as well as those identified as unknowns.

8.7.8 After analysis is complete, the pH is recorded for all water samples on a pH Log (See Figure 1). If a sample has a pH reading >2, the project officer associated with that particular client's samples is notified.

8.8 Soil Sample Analysis - Low Level

8.8.1 The low-level method is based on purging a heated sediment/soil mixed with filtered tap water containing the surrogate and internal standards.

8.8.2 The sample (for volatile organics) consists of an equal representation of the entire contents of the sample container (i.e., no supernatant liquids are discarded.) The contents of the sample container are mixed with a narrow spatula, then the sample is weighed into a disposable test tube if analyzing on the Tekmar 2032 or 2016, or the sample is weighed into a disposable 40mL volatile vial if analyzing on the Tekmar Precept. 5 grams of sample are used, unless a dilution is required, in

which case 1 - 4 grams of sample may be used. A top loading balance is used, and the actual weight is recorded to the nearest 0.1 gram.

8.8.3 Immediately after weighing the sample into the labeled test tube or vial it is loaded onto the Tekmar ALS. If analyzing on the Tekmar 2032/2016, the plunger from a 5 mL Luerlock syringe is removed and the syringe is filled to overflowing with filtered tap water. The plunger is replaced to vent trapped air, then the volume is adjusted to 5.0 mL. 10 µL each of the Surrogate Spiking Solution and the Internal Standard Solution are added to the syringe. This addition to the soil is equivalent of 50 µg/Kg of each standard. This spiked filtered tap water is added to the test tube, the sample is heated to 40°C, and purging is begun. If analyzing on the Tekmar Precept, the vial is transferred for heating and it automatically spikes the soil/water mixture with Internal Standard and Surrogate to yield a concentration for both of 50ug/Kg. The samples follow the same purge method as stated in the calibration.

8.8.4 For soil samples requiring matrix spike and matrix spike duplicates, 10 µL of the matrix spike solution is introduced into the 5 mL of the filtered tap water being added to the soil if analyzing on the Tekmar 2032/2016, or 80µL of matrix spike solution is introduced into 40mL of water if analyzing on the Tekmar Precept. Disregarding any dilutions, this is equivalent to 50 µg/Kg of each matrix spike standard.

8.8.5 With all matrices, all dilutions should keep the response of the major constituents (previously saturated peaks) in the upper half of the linear range of the curve.

8.8.6 An estimated concentration for Tentatively Identified Compounds (TICs) shall be quantified by the internal standard method. For quantification, the nearest internal standard shall be used.

8.8.7 The formula for calculating concentrations for target compounds is found in Table 9. For non-TIC compounds, area counts from the total ion chromatograms are to be used for both the compound to be measured and the internal standard. A response factor (RF) of one (1) is to be assumed. The value from this quantitation shall be qualified as "estimated." This estimated concentration should be calculated for all TICs as well as those identified as unknowns.

8.8.8 All soil samples must have their percent moisture determined so that compound contamination can be reported for "dry weight." This is performed by the Extractions Department and submitted to the Volatiles Data Clerk for reporting.

8.9 Soil Sample Analysis — Medium Level

8.9.1 Medium level extraction/analysis is required if analysis of a sample at a 1 gram level produces peak saturation, or compounds out of linear range. The medium level method is based on extracting the sediment soil sample with methanol. An aliquot of the methanol extract is added to filtered tap water containing the internal standards. The surrogate is spiked directly into the extract, thus making

the addition directly into the filtered tap water unnecessary. The extract is purged at ambient temperature.

8.9.2 The medium level extract is prepared by weighing 4 grams of the sample into the scintillation vial. Using a top-loading balance, the actual weight is noted to the nearest 0.1 g, then 9900 µL of methanol and 100 µL of the medium concentration Surrogate Standard are added. The mixture is then capped and shaken by hand for 2 minutes. The extract should be made up quickly in order to avoid the loss of volatile organics.

8.9.3 The sample is loaded in the following manner: if analyzing on the Tekmar 2032/2016, a syringe is filled just short of overflowing using filtered tap water, then the volume is adjusted to 4.9 mL. The plunger is then adjusted to 5 mL to allow for the addition of the extract and standards, and 10 µL of Internal Standard Solution is added. Finally, the volume of the methanol extract and the volume of methanol, if needed, is added to total 100 µL. If analyzing on the Tekmar Precept, a volatile vial is filled to 40mL and adjusted to 39.2mL. The volume of methanol extract and volume of methanol, if needed, is added to total 800µL.

8.9.4 The sample is loaded into the purging device (Tekmar) and the same purging method as stated during calibration is followed.

8.9.5 For samples requiring matrix spike and matrix spike duplicates, an extract is prepared in the same manner as stated above in Section 8.9.2, using 9800 µL of methanol, 100 µL of medium concentration surrogate, and 100 µL of medium concentration matrix spike solution. These samples are loaded in the same manner as other samples.

8.9.6 The method blank must be analyzed in the same manner as with low-level soils and waters, but it is prepared differently using 4 gram of a purified solid matrix. Surrogate Spiking Solution and methanol are then added to make a total volume of 10 mL, and the blank is purged at ambient temperature. An acceptable blank must contain less than or equal to the reporting limit of any volatile target compound, or else corrective action will be taken immediately.

8.9.7 With all matrices, all dilutions should keep the response of the major constituents (previously saturated peaks) in the upper half of the linear range of the curve.

8.9.8 An estimated concentration for Tentatively Identified Compounds (TICs) shall be quantified by the internal standard method. For quantification, the nearest internal standard shall be used.

8.9.9 The formula for calculating concentrations for target compounds is found in Table 9. For TIC compounds, area counts from the total ion chromatograms are to be used for both the compound to be measured and the internal standard. A response factor (RF) of one (1) is to be assumed. The value from this quantitation shall be qualified as "estimated." This estimated concentration should be calculated for all TICs as well as those identified as unknowns.

8.9.10 All soil samples must have their percent moisture determined so that compound contamination can be reported for "dry weight." This is performed by the Extractions Department and submitted to the Volatiles Data Clerk for reporting. The values may also be obtained from the LIMs database by episode.

8.10 Analysis Report Criteria--A sample is acceptable when it meets the following criteria:

8.10.1 The internal standards' EICP areas are within a factor of 2(-50%/+100%) of the calibration' internal standards.

8.10.2 The internal standards' retention times are within ±30 seconds of the calibration's internal standards.

8.10.3 Surrogate recoveries are within the limits stated in Table 4.

8.10.4 Any compound found in the sample is within the linearity range, with any dilutions showing compounds in an acceptable range of linearity. If a sample analysis contains any target compounds above the linear range, all subsequent samples will be reviewed for those target compounds. The target compounds must be below the reporting limits to be acceptable; otherwise they must be reanalyzed.

8.10.5 Chromatogram shows no signs of carryover or instrument deficiencies.

8.11 Reanalysis Criteria: If sample does not meet internal standards' or surrogates' criteria, it must be reanalyzed. If the reanalysis meets all criteria, it is submitted. If the reanalysis duplicates the results from the first analysis, both analyses are submitted. This information is recorded in the analyst's log book (See Figure 2).

8.12 A sample report is complete when it contains all of the following in order:

8.12.1 Chromatogram

8.12.2 Quant Report

8.12.3 Manual Integrations

8.12.4 Spectra of any compound, excluding internal standards and surrogates

8.12.5 Tentatively Identified Compounds (TICs)

8.12.6 Internal Standards/Surrogates

9.0 CALCULATIONS

9.1 All calculations are shown in Table 9 on page 22 of this SOP.

10.0. QUALITY ASSURANCE/QUALITY CONTROL

10.1 Method Blank (Laboratory Reagent Blank)

10.1.1 The method blank is analyzed once the calibration has been met, whether it be an initial calibration or calibration verification. The method blank, called the "VBLK", consists of loading a syringe with 5 mL of filtered tap water spiked to yield a concentration of 50 ppb internal standard and surrogate, or loading a 40mL volatile vial of filtered tap water into the Precept tray to be spiked automatically by the instrument. The VBLK is used to establish the analytical system is free of contaminants. It is also used to check for carryover contamination after a standard run.

10.1.2 An acceptable blank must meet the internal standard criteria stated in Section 8.10.1 and the surrogate recoveries found in Table 4. The concentration of the target compounds in the blank must be less than the method reporting limit for each target compound (see Table 7).

10.1.3 If a blank exceeds these criteria, corrective measures must be taken immediately, beginning with immediately reanalyzing the blank. If all criteria are still not met, the supervisor is then consulted.

10.2 Laboratory Control Spike (LCS and LCSD)

10.2.1 The Laboratory Control Spike (LCS and LCSD) are internal laboratory QC samples designed to show the capability of our laboratory to be in compliance with the protocol. They consist of filtered tap water spiked with an LCS solution which contains all compounds found in the daily calibration standards at a 50ppb level.

10.2.2 An acceptable LCS and LCSD must meet the internal standard criteria stated in Section 8.10.1 and the surrogate recoveries in Table 4. The percent recovery for each of the compounds in the LCS and LCSD should be within recovery limits shown in the QC Method Reference "Blue Book". The LCS limits are set forth by our laboratory showing our internal control limits, therefore, operators should put forth every effort to meet these limits. Limits are derived using a control chart program as per the Control Chart SOP (SWL-GA-112).

10.2.3 If the compounds are outside QC limits, corrective action is needed immediately, beginning with rerunning the LCS to verify that it was prepared correctly and that it was not a bad purge. If reanalysis does not solve the problem, a new standard may need to be made.

10.3 Matrix Spikes - MS/MSDs will be analyzed at a rate of 1 MS/MSD per batch of 20 or fewer samples. A minimum of the five compounds (1,1-Dichloroethene, Benzene, Trichloroethene, Toluene, and Chlorobenzene) will be used; however all compounds can be spiked. The recovery limits will be the same as the LCS/LCSD and will be advisory since they are dependent upon the matrix.

11.0 WASTE DISPOSAL

11.1 Residual sample material and standards are discarded as directed by the Hazardous Waste Management SOP (SWL-GENHAZW, Rev. 3.0).

12.0 METHOD PERFORMANCE

12.1 The recovery limits for each of the compounds in the LCS and LCSD are shown in the QC Method Reference "Blue Book".

12.2 The recovery limits for each of the compounds in the MS and MSD are shown in the QC Method Reference "Blue Book".

12.3 MDL'S for this method are reported in the Laboratory's *"Red Book"*.

13.0 REFERENCES

13.1 Method SW846-8260A (Revision 1 – September, 1994)

13.2 Control Chart SOP (SWL-GA-112, Rev. 2.0, 5/15/97)

13.3 Definition and Determination of Detection Limits SOP (SWL-GA-113, Rev. 3.0, 6/17/97)

13.4 Hazardous Waste Management SOP (SWL-GENHAZW, Rev. 3.0)

14.0 DEFINITIONS

14.1 BFB: Bromofluorobenzene

14.2 Calibration: Establishment of an analytical curve based on response factors of known standards.

14.3 Calibration Standards: Series of known standard solutions used by the analyst for calibration of the instrument (i.e.: preparation of the analytical curve).

14.4 GC/MS: Gas Chromatography/Mass Spectrometry

14.5 Laboratory Control Spike (LCS): A known matrix spiked with compounds representative of the target analytes. Used to document laboratory performance.

15.0 DATA ARCHIVAL

15.1 Instrument data is stored in two separate locations before being archived to ensure data does not become erased or corrupt. Data is stored on both the instrument's Chemstation and the Unix-based processing system. Data is archived from the Unix-based system using the "TAR" format onto 60 m DAT tapes. No more than two months of data is archived onto the same tape. Once a tape contains two months of data, a tape directory is printed and bound for permanent storage. Each tape is labeled with the instrument ID and the date range of data contained on the tape.

16.0 METHOD DETECTION LIMITS (MDL'S)

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16.1 MDL's are analyzed annually for both 5mL water and soil methods. Seven aliquots are analyzed at a concentration of 5ppb and their data is recorded in a spreadsheet format for easy review.

16.2 MDL's are verified to be below our reporting limit.

16.3 MDL'S are reported in the Laboratory's *"Red Book"*.

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Table 1

Characteristic Ions for Surrogates and Internal Standards for Volatile Organic Compounds

Surrogate or Internal Standard	Primary Characteristic Ion	Secondary Characteristic Ion(s)	Surrogate or Internal Standard	Primary Characteristic Ion	Secondary Characteristic Ion(s)
4-Bromofluorobenzene	95	174.176	1,4-Difluorobenzene	114	
Dibromofluoromethane	113		Chlorobenzene-d_5	117	
Toluene-d_8	98		1,4-Dichlorobenzene-d_4	152	
Pentafluorobenzene	168				

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Table 2

Characteristic Masses (m/z) for Purgeable Organic Compounds

Analyte	Primary Characteristic Ion	Secondary Characteristic Ion(s)	Analyte	Primary Characteristic Ion	Secondary Characteristic Ion(s)
Acetone	43	58	Trans-1,2-Dichloroethene	96	61, 98
Acrolein	56	55	1,2-Dichloropropane	63	112
Acrylonitrile	53	52, 51	1,3-Dichloropropane	76	78
Benzene	78	—	2,2-Dichloropropane	77	97
Bromobenzene	156	77, 158	1,1-Dichloropropene	75	110, 77
Bromochloromethane	128	49, 130	Ethylbenzene	91	106
Bromodichloromethane	83	85, 127	2-Hexanone	43	58,57
Bromoform	173	175, 254	Hexachlorbutadiene	225	223, 227
Bromomethane	94	96	Isopropylbenzene	105	120
2-Butanone	43	72, 57	p-Isopropyltoluene	119	134, 91
n-Butylbenzene	91	92, 134	Methylene Chloride	84	86, 49
sec-Butylbenzene	105	134	4-Methyl-2-Pentanone	43	58
'ert-Butylbenzene	119	91, 134	Naphthalene	129	—
Carbon Disulfide	76	78	n-Propylbenzne	91	120
Carbon Tetrachloride	117	119	Styrene	104	78
Chlorobenzene	112	77, 114	1,1,1,2-Tetrachloroethane	131	133, 119
Chloroethane	64	66	1,1,2,2-Tetrachloroethane	83	131, 85
2-Chloroethylvinyl Ether	63	43, 43	Tetrachloroethene	166	168, 129
Chloroform	83	85	Toluene	92	91
Chloromethane	50	52	Trans-1,3-Dichlorpropene	75	77
2-Chlorotoluene	91	126	1,2,3-Trichlorobenzene	180	182, 145
4-Chlorotoluene	91	126	1,2,4-Trichlorobenzene	180	182, 145
cis-1,3-Dichloropropene	75	77	1,1,1-Trichloroethane	97	99, 61
1,2-Dibromo-3-Chloropropane	75	155, 157	1,1,2-Trichlorethane	83	97, 85
Dibromochloromethane	129	127	Trichloethene	95	130, 132
1,2-Dibromoethane	107	109, 188	Trichlorofluoromethane	101	103
Dibromomethane	93	95, 174	1,2,3-Trichloropropane	75	77
1,2-Dichlorobenzene	146	111, 148	1,2,4-Trimethylbenzene	105	120
1,3-Dichlorobenzene	146	111, 148	1,3,5-Trimethylbenzene	105	120
1,4-Dichlorobenzene	146	111, 148	Vinyl Chloride	62	64
Dichlorodifluoromethane	85	87	o-Xylene	106	91
1,1-Dichloroethane	63	65, 83	m,p-Xylene	106	91
1,2-Dichloroethane	62	98	Vinyl Acetate	43	86
1,1-Dichloroethene	96	61, 63			
cis-1,2-Dichloroethene	96	61, 98			

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Table 3

BFB Key Ions and Abundance Criteria

Mass	Ion Abundance Criteria
50	15.0 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	<2% of mass 174
174	>50% of mass 95
175	5 to 9% of mass 174
176	>95% but <101% of mass 174
177	5 to 9% of mass 176

Table 4

Surrogate Recovery Limits*

Surrogate Compound	Water	Low/Med Soil/Sediment
4-Bromofluorobenzene	86-115	74-121
Dibromofluoromethane	86-118	80-120
Toluene-d_8	88-110	81-117

*Based upon method limits

Table 5

Volatile Internal Standards with Corresponding Analytes Assigned for Quantitation

Pentafluorobenzene	Chlorobenzene-d5	1,4-Difluorobenzene	1,4-Dichlorobenzene-D4
Acetone	Bromoform	Benzene	Bromobenzene
Acrolein	Chlorobenzene	Bromodichloromethane	n-Butylbenzene
Acrylonitrile	1,3-Dichloropropane	Bromofluorobenzene (Surrogate)	sec-Butylbenzene
Bromochloromethane	Ethylbenzene	Carbon Tetrachloride	tert-Butylbenzene
Bromomethane	2-Hexanone	2-Chloroethylvinylether	2-Chlorotoluene
2-Butanone	Styrene	1,2-Dibromoethane	4-Chlorotoluene
Carbon Disulfide	1,1,1,2-Tetrachloroethane	1,2-Dichloroethane	1,2-Dibromo-3-Chloropropane
Chloroethane	Tetrachloroethene	Dibromofluoromethane (Surrogate)	1,2-Dichlorobenzene
Chloroform	Dibromochloromethane	1,2-Dichloropropane	1,3-Dichlorobenzene
Chloromethane	m,p-Xylene	1,1-Dichloropropene	1,4-Dichlorobenzene
Dichlorodifluoromethane	o-Xylene	cis-1,3-Dichloropropene	Hexachlorobutadiene
1,1-Dichloroethane		trans-1,3-Dichloropropene	Isopropyl benzene
1,1-dichloroethene		4-Methyl-2-Pentanone	p-Isoproyltoluene
cis-1,2-Dichloroethene		Toluene	Naphthalene
trans-1,2-Dichloroethene		Toluene-d_8 (Surrogate)	n-Propylbenzene
2,2-Dichloropropane		1,1,2-Trichloroethane	1,1,2,2-Tetrachloroethane
Iodomethane		Trichloroethene	1,2,3-Trichlorobenzene
Methylene Chloride			1,2,4-Trichlorobenzene
1,1,1-Trichloroethane			1,2,3-Trichloropropane
Trichlorofluoromethane			1,2,4-Trimethylbenzene
Vinyl acetate			1,3,5-Trimethylbenzene
Vinyl chloride			

Table 6

Volatile Calibration Check Compounds (CCC)

- 1,1-Dichloroethene
- 1,2-Dichloropropane
- Ethylbenzene
- Chloroform
- Toluene
- Vinyl Chloride

Initial Calibration: 5,20,50,100,200ppb (soil/5mL purge)
Max%RSD: 30%
Max%Drift: 20%
Calibration Verification: 50ppb (soil/5mL purge)

Volatile System Performance Check Compounds (SPCC)

- Chloromethane
- 1,1-Dichloroethane
- Bromoform
- 1,1,2,2-Tetrachloroethane
- Chlorobenzene

Minimum Relative Response Factor (RRF)
Soil /5mL Purge RRF:
0.100 for Bromoform
1,1-Dichloroethane
Chloromethane
0.300 for Chlorobenzene
1,1,2,2-Tetrachloroethane

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Table 7

Compound List and Method Reporting Limits

Volatile Compound	CAS#	5mL Water or 5g Soil*	Volatile Compound	CAS#	5mL Water or 5g Soil*
Acetone	67-64-1	5	Trans-1,2-Dichloroethene	156-60-5	5
Acrolein	107-02-8	50	1,2-Dichloropropane	78-87-5	5
Acrylonitrile	107-13-1	50	cis-1,3-Dichloropropene	10061-01-5	5
Benzene	71-43-2	5	trans-1,3-Dichloropropene	10061-02-6	5
Bromobenzene	108-86-1	5	1,3-Dichloropropane	142-28-9	5
Bromochloromethane	74-97-5	5	2,2-Dichloropropane	594-20-7	5
Bromodichloromethane	75-27-4	5	1,1-Dichloropropene	63-58-6	5
Bromoform	75-25-2	5	Ethylbenzene	100-41-4	5
Bromomethane	71-83-9	5	2-Hexanone	591-78-6	5
2-Butanone	78-93-3	5	Hexachlorobutadiene	87-68-3	5
Carbon Disulfide	75-15-0	5	Isopropylbenzene	92-82-8	5
n-Butylbenzene	104-51-8	5	p-Isopropyltoluene	99-87-6	5
sec-Butylbenzene	135-98-8	5	Methylene chloride	75-09-2	5
tert-Butylbenzene	98-06-6	5	4-Methyl-2-Pentanone	108-10-1	5
Carbon Tetrachloride	56-23-5	5	Naphthalene	91-20-3	5
Chlorobenzene	108-90-7	5	n-Propylbenzene	103-65-1	5
Chloroethane	75-00-3	5	Sytrene	100-42-5	5
Chloroform	67-66-3	5	1,1,1,2-Tetrachloroethane	630-20-6	5
2-Chloroethylvinyl Ether	110-75-8	5	1,1,2,2-Tetrachloroethane	79-34-5	5
Chloromethane	74-87-3	5	Tetrachloroethene	127-18-1	5
2-Chlorotoluene	95-49-8	5	Toluene	108-88-3	5
4-Chlorotoluene	106-43-4	5	1,2,3-trichlorobenzene	87-61-6	5
Dibromochloromethane	124-48-1	5	1,2,4-Trichlorobenzene	120-82-1	5
1,2-Dibromo-3-Chloropropane	92-12-8	5	1,1,1-Trichloroethane	71-55-6	5
1,2-Dibromoethane	106-93-4	5	1,1,2-Trichloroethane	79-00-5	5
Dibromomethane	74-94-3	5	Trichloroethene	79-01-6	5
1,2-Dichlorobenzene	95-50-1	5	Trichlorofluoromethane	75-69-4	5
1,3-Dichlorobenzene	541-73-1	5	1,2,3-Trichloropropane	96-18-4	5
1,4-Dichlorobenzene	106-46-7	5	1,2,4-Trimethylbenzene	95-63-6	5
Dichlorodifluoromethane	75-71-8	5	1,3,5-Trimethylbenzene	108-67-8	5
1,1-Dichloroethane	75-34-3	5	Vinyl Chloride	75-01-4	5
1,2-Dichloroethane	107-06-2	5	o-Xylene	95-47-6	5
1,1-Dichloroethene	75-35-4	5	m,p-Xylene	13-302-07	5
cis-1,2-Dichloroethene	156-59-2	5	Vinyl Acetate	108-05-4	5

NOTE: Concentration Units: Water=µg/L Soil=µg/Kg
*Reporting Limits derived from method.

Table 8

Standards Preparation—Soil/5mL Purge

Compound or Standard Mix	Begin Conc. (µg/mL)	Aliquot (µL)	Total Vol. (mL)	Final Conc. (µg/mL)
Internal Standard				
NSI:				
Internal Standard Mix	2500	200	20	25
Surrogate:				
NSI:				
Surrogate Mix	2500	200	20	25
VSTD025				
NSI:				
Mix A	2000	125	10	25
Mix C	2000	125	10	25
Mix L	2000	125	10	25
Mix M	2000	125	10	25
Carbon Disulfide	5000	50	10	25
Vinyl Acetate	5000	50	10	25
Ketones Mix	1000	125	10	25
Extra Compound Mix				
Chem-Service:				
2-Chloroethylvinyl Ether	10,000	25	10	25
Acrolein	10,000	250	10	250
Acrylonitrile	10,000	250	10	250
1-Chlorohexane	10,000	25	10	25
MTBE	10,000	25	10	25
Methyl Iodide	10,000	25	10	25
Trans-1,4-Dichloro-2-Butene	10,000	25	10	25
n-Hexane	10,000	25	10	25
Gases Mix				
NSI:				
Gases Mix	1000	125	5	25

Table 9

Response Factor and Concentration Calculation

$$RF = \frac{(A_x)\ (C_{is})}{(A_{is})\ (C_x)} \quad \text{and} \quad conc_{RF} = \frac{(A_x)\ (C_{is})}{(A_{is})\ (\overline{RF})} \quad \text{or} \quad conc_{LR} = \frac{(A_x) - b}{M} \quad \text{or} \quad conc_{quad} = a + bx + cx^2$$

Where:

A_x = Area of characteristic ion for the measured compound.
A_{is} = Area of the characteristic ion for the specified internal standard
C_{is} = Concentration of the internal standard (ng/µL)
C_{RF} = Concentration of the measured compound (ng/µL) using the average response factor
$\overline{RF}$ = Average of Five Initial RF's for a compound
C_{LR} = Concentration of the measured compound (ng/µL) using Linear Regression
C_{quad} = Concentration of the measured compound (ng/µL) using the Quadratic Equation

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Surrogate Recovery: % Surrogate Recovery = $\frac{Qd}{Qa} \times 100\%$

Where:

Qd = Quantity determined by analysis
Qa = Quantity added to sample

Matrix Spike recovery and RPD:

Matrix Spike Recovery = $\frac{SSR - SR}{SA} \times 100$ Matrix Spike RPD = $\frac{|MSR - MSDR|}{(½)(MSR + MSDR)} \times 100$

Where:

SSR = Spike Sample Result
SR = Sample Result
SA = Spike Added

Laboratory Control Spike (LCS) Recovery: LCS Spike Recovery = $\frac{SSR - SR}{SA} \times 100$

Where:

SSR = Spiked Sample Result
SR = Sample Result
SA = Spike Added

Calibration Calculations:

$$\%RSD = \frac{SD}{\overline{RF}} \times 100\% \qquad \%Drift = \frac{(C_1 - C_c)}{C_1} \times 100$$

Where:

SD = Standard Deviation
$\overline{RF}$ = Average of Five Initial RF's for a compound
RSD = Relative Standard Deviation

Where:

C_1 = Calibration Check Compound Standard Concentration
C_c = Measured concentration using selected quantitation method

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VOLATILES GC/MS pH LOG

CASE _____ SDG# _____

LABORATORY ID	CLIENT SAMPLE ID	pH	ANALYST	DATE TESTED	DATE P.O. NOTIFIED*

COMMENTS: _____

- Date Project Officer Notified: Field is filled out when samples are not acidified and Project Officer is notified of situation

Figure 1. pH Log

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VOLATILES GC/MS RUN LOG

VOLATILES GC/MS DEPARTMENT

SWOK / AATS

™

MS Std. # _____
IS Std. # _____
Surr Std. # _____

TUNE FILE ID: _____ METHOD FILES _____
INSTRUMENT: _____ DATE _____
CB/ID FILE ID: _____ ARCHIVE TAPE #: _____

Time	File	SWOK/AATS Sample ID	Client ID	Vol.	IS Area	Surr. Rec.	Lin. Chk.	Comments	ALS POS	OK/ RR

149

Analyst Signature

SWOK/AATS • 1700 West Albany • Broken Arrow, Ok 74012

[VGM008-0296-03]

Figure 2. GC/MS Run Log

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SOUTHWEST LABORATORY OF OKLAHOMA, INC.

STANDARD OPERATING PROCEDURE FOR THE ANALYSIS OF NITROAROMATICS & NITRAMINES BY HPLC

DOC. # SWL-OL-200
REV 4.0 — 7/11/97

DOCUMENT CONTROL #SWL-OL-200-_____

APPROVALS	DATES
Procedure Prepared By	07/11/97 Date
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Dil Abeta for Mike Bulter Laboratory Safety Officer	7/11/97 Date
Chuck Hrach Laboratory QA/QC Officer	7/11/97 Date

(Effective Date is 5 calendar days after the last signature above - QA/QC Officer)

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Document Status

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Section 1.0 - Scope and Application

1.1 SW-846, method-8330, Rev 0, September, 1994 is intended for the analysis of explosives residues. This method is used to determine the concentration of the following compounds in a water, soil or sediment matrix:

* Chemical Abstracts Service Registry number

Compound	Abbr.	CAS No.*
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine	HMX	2691-41-0
Hexahydro-1,3,5-trinitro-1,3,5-triazine	RDX	121-82-4
1,3,5-Trinitrobenzene	TNB	99-35-4
1,3-Dinitrobenzene	DNB	99-65-0
Methyl-2,4,6-trinitrophenylnitramine	Tetryl	479-45-8
Nitrobenzene	NB	98-95-3
2,4,6-Trinitrotoluene	TNT	118-96-7
4-amino-2,6-dinitrotoluene	4ADNT	1946-51-0
2-amino-4,6,dinitrotoluene	2ADNT	35572-78-2
2,6-Dinitrotoluene	26DNT	606-20-2
2,4-Dinitrotoluene	24DNT	121-14-2
o-Nitrotoluene	2NT	88-72-2
p-Nitrotoluene	4NT	99-99-0
m-Nitrotoluene	3NT	99-08-1
SURROGATE		
3,4-Dinitrotoluene	34DNT	610-39-9

1.2 All of these compounds are either used in the manufacturing of explosives or are the degradation products of compounds used for that purpose. When making stock solutions for calibration, treat each compound as if it were extremely explosive.

1.3 The practical quantitation limits (PQLs) of target analytes are presented in Table 5.

1.4 This method is restricted to use by or under the supervision of analysts experienced in the use of HPLC, skilled in the interpretation of chromatograms, and experienced in handling explosive materials. Each analyst must demonstrate the ability to generate acceptable results with this method.

2.0 - Summary of Method

2.1 Aqueous samples are diluted 2X (v/v) with the appropriate solvent, filtered, separated on a Carbosorb reverse phase column (or equivalent), determined at 254 nm, and confirmed on an LC-CN column.

2.2 Aqueous Samples:

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2.2.1 High level water (Direct Injection Method)

2.2.1.1 The samples are diluted 2X (v/v) with filtered Methanol, separated on a Carbosorb reverse phase column (or equivalent), determined at 254nm, and confirmed on a LC-CN reverse phase column.

2.2.2 Low level water (Salting out):

2.2.2.1 The samples are extracted using NaCl and acetonitrile. The extracts are diluted 2x (v/v) with filtered water, separated on a Carbosorb reverse phase column (or equivalent), determined at 254 nm, and confirmed on a LC-CN reverse phase column.

2.2.2.2 Refer to appendix A for the extraction procedure.

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

2.3.1 Refer to section 7.0 for the extraction procedure.

Section 3.0 - Interferences

3.1 Since some of the analytes are not separated completely on the confirmation column (LC-CN), these analytes are prepared in a different calibration standard. Therefore, different stocks of standards are prepared for the confirmation column according to the analytes elution.

3.2 Tetryl decomposes rapidly in methanol/water solutions, as well as with heat and light. All aqueous samples expected to contain Tetryl should be diluted with acetonitrile prior to filtration. All samples expected to contain Tetryl should not be exposed to temperatures above room temperature.

3.3 Degradation products of Tetryl appear as a shoulder to the TNT peak.

3.4 Large amounts of salt present in acetonitrile extracted from the last step of the low water (salting out) explosive produces interfering peaks. This peak interferes with the eluted HMX at the beginning of the analysis.

Section 4.0 - Apparatus and Materials

4.1 Primary HPLC System

4.1.1 Pump — HP Model 1050 quaternary system (or equivalent.)

4.1.2 Column — Carbosorb, 5um, 250mm x 4.6mm ID (or equivalent).

4.1.3 Detector — HP Model 1050 Variable UV (or equivalent.)

4.1.4 Autosampler — HP Model 1050 autosampler (or equivalent.)

4.1.5 Data System — VG Minichrom data system or equivalent.

4.2 Confirmation HPLC System

4.2.1 Pump – Waters model 510 high pressure pump capable of 6000 psi at 10 ml/min. or equivalent.

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4.2.2 Column – Supelco LC-CN, 5 µm, 25cm x 4.6mm ID (or equivalent).

4.2.3 Detector – Waters model 486 variable wavelength Detector set at 254 nm or equivalent.

4.2.4 Autosampler – Waters model 715 WISP or equivalent.

4.2.5 Data System – VG Minichrom or equivalent.

4.3 Other Equipment

4.3.1 NEY-300 Ultrasonic Bath equipped with drainage, that can hold water at room temperature to 35°C (or equivalent). The sonicator is kept covered during sonication to keep the samples in the dark.

4.3.2 BLUE M Convection Heating Oven with the temperature range of 0-300°C or equivalent.

4.3.3 Balance ± 0.0001 g.

4.4 Materials

4.4.1 Filters – Gelman Accuspec CR 0.45 µm filters for filtering extracts, or equivalent

4.4.2 Filters – Supelco Nylon 66 0.2 µm x 47 mm membranes for filtering chromatographic solvents or equivalent.

4.4.3 Glass Pipettes – Class A, glass, 50 mL, 10 mL, 5 mL, 4 mL, 2 mL, 1 mL

4.4.4 Vials – 15 mL, 20 mL, 40 ml, glass, Teflon lined screw cap or crimp top.

4.4.5 Syringes – 5 mL, 10 mL, glass or polypropylene.

4.4.6 Volumetric flasks – Class A - 10 mL, 20 mL, 50 mL, 100 mL, 200 mL, 250 mL.

4.4.7 Mortar and pestle.

4.4.8 SMI pipetter – 50-250 µL, and 10-60 µL.

Section 5.0 - Reagents

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

5.1.1 Acetonitrile, CH_3CN – HPLC grade.

5.1.2 Methanol, CH_3OH – HPLC grade.

5.1.3 Water, H_2O – HPLC grade.

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

5.2.1 HMX – Standard Analytical Reference Material.

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5.2.2 RDX – Standard Analytical Reference Material.

5.2.3 DNB – Standard Analytical Reference Material.

5.2.4 Tetryl – Standard Analytical Reference Material.

5.2.5 TNT – Standard Analytical Reference Material.

5.2.6 2,4-DNT – Standard Analytical Reference Material.

5.2.7 2,6-DNT – Standard Analytical Reference Material.

5.2.8 1,3,5-TNB – Standard Analytical Reference Material.

5.2.9 NB – Standard Analytical Reference Material.

5.2.10 2-NT – Standard Analytical Reference Material.

5.2.11 3-NT – Standard Analytical Reference Material.

5.2.12 4-NT – Standard Analytical Reference Material.

5.2.13 2ADNT – Standard Analytical Reference Material.

5.2.14 4ADNT – Standard Analytical Reference Material.

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

5.4 Intermediate stock standard solution:

5.4.1 The intermediate stock standard solution is prepared from the immediate stock standard solution in methanol according to Table 1 (an example of a soil intermediate stock solution).

Table 1

Compound	AccuStandard#	Orig. Conc. (ug/mL)	Aliquot (uL)	Final Conc. (ug/mL)
HMX	M-8330-04	1000	600	60.0
RDX	M-8330-05	1000	250	25.0
TNB	M-8330-12	1000	179	17.9
DNB	M-8330-01	1000	96	9.60
TETRYL	M-8330-10	1000	220	22.0
NB	M-8330-06	1000	195	19.5
3,4-DNT	M-8330-IS	1000	300	30.0
TNT	M-8330-11	1000	160	16.0
4ADNT	M-8330-14	1000	140	14.0
2ADNT	M-8330-13	1000	140	14.0
26DNT	8330-03	1000	230	23.0
24DNT	8330-02	1000	140	14.0
2NT	M-8330-07	1000	200	20.0
4NT	M-8330-09	1000	200	20.0
3NT	M-8330-08	1000	190	19.0

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5.4.2 The two concentrated intermediate stock solutions are diluted with methanol, refrigerated at 4°C and kept in the dark upon preparation for 6 months .

5.5 Working Calibration Standards for soil matrices.

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

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

5.5.3 The Laboratory currently is in search for a second source calibration standard verification (ICV).

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

5.6.1 LCS and LCSD are prepared from stock standard solutions that differ from the intermediate stock standard solution which is used to prepare the standards (see Table 2). LCS contains the analytes of interest at midpoint level or higher.

Table 2

Compound	AccuStandard#	Orig. Conc. (ug/mL)	Aliquot (uL)	Final Conc. (ug/mL)
HMX	M-8330-04	1000	1920	64.0
RDX	M-8330-05	1000	750	25.0
TNB	M-8330-12	1000	690	23.0
DNB	M-8330-01	1000	585	19.5
TETRYL	8330-10	1000	459	15.30
NB	M-8330-06	1000	420	14.0
TNT	M-8330-11	1000	600	20.0
4ADNT	M-8330-14	1000	480	18.0
2ADNT	M-8330-13	1000	540	16.0
26DNT	8330-03	1000	465	15.5
24DNT	8330-02	1000	288	9.60
2NT	M-8330-07	1000	870	29.0
4NT	M-8330-09	1000	570	19.0
3NT	M-8330-08	1000	600	20.0

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

5.7.1 MS and MSD are prepared as V.F. Spike the sample at midpoint or higher calibration range. (See Table 2.)

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5.8 Surrogate

5.8.1 To monitor the extraction and analytical performance, all of the extracts are spiked with an analyte of non-interest. 34DNT is the analyte of non-interest used for the surrogate. The surrogate is spiked at mid-level or higher.

5.8.2 Due to insufficient number of data points QC limits of 60-140 is reported for the surrogate until enough data points have been collected.

5.9 Eluent

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

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

5.9.2.1 Both eluents can be used to analyze high level water. However, if HMX is an important target, 50% methanol/H2O is preferred.

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

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

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

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

Section 6.0 - Sample Collection, Preservation, and Handling

6.1 Grab samples must be collected and stored in glass containers. Follow conventional sampling procedures.

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

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

Section 7.0 - Procedure

7.1 Sample Preparation

7.1.1 Aqueous Samples

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7.1.1.1 High water aqueous samples.

7.1.1.1.1 Filter 20 mL of sample prior to analysis and keep in the dark until analysis. Prior to analysis, place 0.5 mL of the filtered sample in the autosampler vial, add 0.5 mL of methanol to the sample, cap and shake thoroughly. The sample should be ready for analysis.

7.1.1.2 Low water (salting out) aqueous samples

7.1.1.2.1 Prior to analysis, dilute each extract with HPLC water, then shake thoroughly. The samples are ready for analysis.

7.1.1.2.2 Add 251.3g of Sodium Chloride to a 1 liter volumetric flask. Measure out 770mL of a water sample in a 1000 mL graduated cylinder. Transfer part of the sample to a volumetric flask. Gently swivel the flask until part of the salt has been dissolved. Add the rest of the sample and stir bar to the flask. Mix at maximum speed on a magnetic stirrer until the salt is completely dissolved.

7.1.1.2.3 Add 164 mL of acetonitrile (measured with a 250mL graduated cylinder) while the solution is being stirred. Stir for an additional 15 minutes. Turn off the stirrer and allow the phase to separate for 10 minutes.

7.1.1.2.4 Remove the acetonitrile (upper) layer (about 8mL) with a pasteur pipette and transfer it to a 100 mL volumetric flask. Add 10 mL of fresh acetonitrile to the water sample in the 1.0 liter flask. Stir the content of the flask for 15 minutes followed by 10 minutes for phase separation. Combine the second acetonitrile portion with the initial extract. The inclusion of a few drops of salt water at this point is not important.

7.1.1.2.5 Prepare 32.5% of sodium chloride according to the number of samples (325g of NaCl dissolved in to 1000 mL of Millipore water).

7.1.1.2.6 Add 84 mL of step 4 to the acetonitrile extract from the previous step. Add a stir bar and stir the contents on a magnetic stirrer for 15 minutes followed by 10 minutes for phase separation. Carefully transfer the acetonitrile phase to a 12.0mL vial with Teflon lined screw cap. At this stage the amount of water transferred with the acetonitrile must be minimized. The water contains a high concentration of NaCl which produces an interfering peak.

7.1.1.2.7 Add an additional 1.mL of acetonitrile to the 100mL volumetric flask. Again stir the contents of the flask for 15 minutes followed by 10 minutes for phase separation. Combine the second acetonitrile portion with the initial extract. The final volume should be 5.0mL.

7.1.1.2.8 If the extract volume exceeds the requirement, very gently blow down the extract with a minute stream of nitrogen until the desired volume is achieved.

7.1.1.2.9 If the diluted extract is turbid, filter it through a 0.45um Teflon filter using a disposable syringe. Discard the first 0.5mL of filtrate, and retain the remainder in a glass vial with a Teflon lined screw cap.

7.1.1.2.10 Prior to analysis dilute the extracts 2X (v/v) with HPLC water or equivalent. If the extracts are not analyzed immediately, store them in the refrigerator at 4°C until analysis.

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7.1.2 Soil and Sediment Samples

7.1.2.1 The sample should be homogenized thoroughly prior to weighing. Using disposable weigh dishes, weigh out 20 g of wet weight sample (the weight should yield 2.0 g after drying).

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

7.1.2.3 Soil Sample extraction

7.1.2.3.1 Place a 2.0g sub-sample of each soil sample in a 40 mL glass VOA vial. Add 10.0 mL of acetonitrile volumetrically, cap the Vial with a teflon lined cap, and place in a cool ultrasonic bath for 18 hours. Water at a constant flow rate should enter the sonicator and dispose out of the ultrasonic bath. During sonication, the samples should be kept at room temperature or below.

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

7.1.2.3.3 If the samples are to be analyzed remove 5.0 mL of supernatant, using a 5.0 mL disposable glass pipette or Rainin pipetter with polypropylene disposable tips. Transfer the supernatant to 12 .0 mL glass vial, volumetrically transfer 5.0 mL of calcium chloride solution (see section 5.9.5). Shake or Vortex the vial for 15-30 seconds or until the solution is thoroughly mixed. Let the solution stand for 15 minutes. Transfer the supernatant to 10 mL disposable syringe containing Gelman Accuspec CR 0.45. Filter the solution by discarding the first 1-2 mL and retain the remainder in another 12 mL glass vial with teflon-lined screwcap. Keep the extracts in 4°C and in a dark place until analysis.

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

7.2 Chromatographic Conditions

Mobile Phase:	50/50 (v/v) methanol/HPLC water
Flow rate:	0.8 mL/min
Injection volume:	200 µL
UV Detector:	254 nm

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

7.4 Calibration of HPLC

7.4.1 Following SW-846, method 8330, the initial calibration standard at the minimal five-concentration is injected only one time. The %RSD of the calibration factors should be ≤20%. If the %RSD for the target analyte is greater than 20%, the correlation coefficient for the calibration line can be used if it is ≥0.995. The curve should be linear and intercept

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at zero. Use the mean calibration factors for all calculations as long as the continuing calibrations described below meet specifications.

7.4.2 Calibration Factor (CF): 5-Level

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

$$\overline{CF} = \sum_{i=5}^{n} \frac{CF_1}{n}$$

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

Where:

$\overline{CF}$ = Mean of the calibration factor
n = (5) Total number of values
STD = Standard deviation of the calibration factors
CF_1 = Calibration factors

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

7.4.4 Percent Deviation (%D)

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

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

7.4.5 Carbosorb reverse phase column (or equivalent) is utilized for the quantitation analysis. Since all of the analytes are not separated on this column, the laboratory is in search of an alternative column.

7.4.6 LC-CN column or equivalent is used to confirm the analytes. Since not all of the analytes are separated on this column, 4 different calibration standards are utilized to confirm all of the analytes. Only the compounds detected above the PQL are confirmed.

7.4.7 Retention times window should be established for each new calibration. Average the retention time for each injection of each component for all initial calibration analysis and continuing calibration analysis. Determine the standard deviation. The window for any compound is ± 3 times the standard deviation. If chromatographic retention drift should occur, make certain that the mobile phase is fresh and has not evaporated and that the

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pumps are functioning properly. If the column itself has deteriorated and caused loss of efficiency and retention, it may have to be replaced.

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

7.5 Sample Analysis

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

7.5.2 Table 6 presents the retention times for the analytes on both the Carbosorb and CN columns. Appendix C presents typical chromatograms.

7.5.3 Calculation for positive hits.

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

Where:

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

Section 8.0 - Quality Control

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

8.2 Method Blanks

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

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

8.3 Laboratory Control Spikes

(See Table 3 for limits)-LCS are done with each extraction batch at a rate of one LCS per 20 samples or less.

8.4 Matrix Spikes

(See Table 4 for limits)-Matrix spike/ Matrix spike duplicates are analyzed at a rate of 1 set per batch of 20 samples or fewer.

8.5 Surrogate — To monitor extraction and analytical performance, all of the extracts are spiked with a surrogate.

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8.6 Since not enough data points have been collected for 2ADNT and 4ADNT, QC limits of 60-140 are being used for the compounds.

Compound	Low Level Water QC Recovery Limits	High Level Water QC Recovery Limits	Soil QC Recovery Limits
HMX	28 - 117	46 - 151	57 - 137
RDX	25 - 114	72 - 129	77 - 113
TNB	24 - 129	74 - 118	64 - 125
TETRYL	25 - 119	58 - 120	23 - 124
DNB	22 - 117	79 - 132	82 - 118
TNT	45 - 118	61 - 145	75 - 118
NB	18 - 119	68 - 135	69 - 143
26DNT	34 - 126	77 - 125	78 - 117
24DNT	35 - 119	70 - 134	74 - 126
2NT	27 - 119	70 - 131	78 - 123
4NT	28 - 118	73 - 116	79 - 125
3NT	27 - 115	71 - 127	78 - 121
2ADNT	60 - 140	60 - 140	60 - 140
4ADNT	60 - 140	60 - 140	60 - 140

Table 3
Laboratory Control Spike Recovery QC Limits

Compound	Recovery Limits	RPD Limits	ecovery Limits	RPD Limits
HMX	44 - 145	15	54 - 128	30
RDX	65 - 139	12	66 - 109	30
TNB	56 - 129	10	57 - 130	30
TETRYL	76 - 120	14	32 - 119	45
DNB	76 - 122	15	74 - 122	30
TNT	76 - 137	15	44 - 124	35
NB	82 - 111	13	70 - 135	30
26DNT	80 - 118	13	58 - 125	35
24DNT	72 - 123	14	59 - 120	35
2NT	74 - 130	15	79 - 124	35
4NT	78 - 116	14	74 - 128	35
3NT	72 - 126	15	79 - 121	35
2ADNT	60 - 140	40	60 - 140	40
4ADNT	60 - 140	40	60 - 140	40

Table 4
Matrix Spike/Matrix Spike Duplicate QC Recovery Limits

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Section 9.0 - Method Performance — Method 8330 was tested by six laboratories. The results of this testing indicate that the results presented in Tables 7 through 9 are to be expected.

Section 10.0 - Data Verification

10.1 If the compound is found above the PQL in the quantitation column and it is within the established retention time window, the confirmational analysis of the compound will be performed. If the compound is found within the retention time window of the confirmational analysis, the compound will be reported. If all the compounds in the QA/QC samples exceeded the established laboratory QC limits, the batch of samples will be reextracted and reanalyzed. If some of the compounds in the QA/QC samples are out of the established limits, the compounds will be flagged and the data will be reported as is.

Section 11.0 - References

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

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

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

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

Section 12.0 - Safety

12.1 Standard precautionary measures used for handling other organic compounds should be sufficient for safe handling of the analytes targeted by Method 8330.

Compound	Low Level Water (µg/L)	High Level Water (µg/L)	Soil (µg/kg)
HMX	1.00	13.0	2200
RDX	0.84	14.0	1000
TNB	0.26	7.3	250
DNB	0.11	4.0	250
Tetryl	0.60	10.0	650
NB	0.30	7.0	260
TNT	0.12	6.4	250
24DNT	0.04	5.7	250
26DNT	0.10	9.4	260
2NT	0.78	12.0	250
3NT	0.65	7.9	250
4NT	0.65	8.0	250
2ADNT	0.06	12.0	250
4ADNT	0.06	7.0	250

Table 5
Practical Quantitation Limits

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CARBOSORB		LC-CN	
Retention		Retention	
Analyte	Time (min)	Analyte	Time (min)
HMX	4.8	NB	5.1
RDX	7.2	2NT	6.1
TNB	9.7	3NT	6.1
Tetryl	12.6	4NT	6.1
DNB	11.9	DNB	5.8
TNT	15.3	TNB	5.9
NB	13.6	26DNT	6.4
26DNT	17.9	24DNT	6.8
24DNT	18.5	TNT	7.0
2NT	21.7	RDX	9.2
4NT	23.2	Tetryl	11.1
3NT	23.0	HMX	14.0
2A DNT	16.8	2ADNT	8.1
4A DNT	16.0	4ADNT	7.4
SURROGATE			
34DNT	14.6	34DNT	8.1

⇦ Table 6
Retention Times For Analytes On ODS-II and CN Columns

Multilaboratory Precision Of Method For Soil Samples
☞ Table 7

Spiked Soils	Field-Contaminated Soils					
	Mean Concentration (mg/g)	SD	%RSD	Mean Concentration (mg/g)	SD	%RSD
HMX	46.0	1.7	3.7	14 153	1.8 21.6	12.8 14.1
RDX	60.0	1.4	2.3	104 877	12 29.6	11.5 3.4
TNB	8.6 46.0	0.4 1.9	4.6 4.1	2.8 72	0.2 6	7.1 8.3
DNB	3.5	0.14	4	1.1	0.11	9.8
Tetryl	17.0	3.1	17.9	2.3	0.41	18
TNT	40.0	1.4	3.5	7 669	0.61 55	9 8.2
24DNT	5.0	0.17	3.4	1	0.44	42.3

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Spiked Soils	Field-Contaminated Soils					
	Mean Concentration (mg/g)	SD	%RSD	Mean Concentration (mg/g)	SD	%RSD
HMX	46.0	2.6	5.7	14	3.7	26
				153	37.3	24
RDX	60.0	2.6	4.4	104	17.4	17
				877	67.3	7.7
TNB	8.6	0.61	7.1	2.8	0.23	8.2
	460.0	2.97	6.5	72	8.8	12.2
DNB	3.5	0.24	6.9	1.1	0.16	14.5
Tetryl	17.0	5.22	30.7	2.3	0.49	21.3
TNT	40.0	1.88	4.7	7	1.27	18
				669	63.4	9.5
24DNT	5.0	0.22	4.4	1	0.74	74

Table 8
Multilaboratory Error Of Method For Soil Samples

	Sample 1		Sample 2	
	Mean conc. (mg/L)	%RSD	Mean conc. (mg/L)	%RSD
HMX	nd	-	184.0 *	8.4
RDX	431	22.9	2117	29.5
TNB	74.3	3.2	27.6 **	4.2
TNT	10635	59.4	1746	26.8

* 6 Replicate Determinations
** 7 Replicate Determinations

Table 9
Interlaboratory Error Of Method For Water Samples
(10 Replicate Determinations, except where noted)

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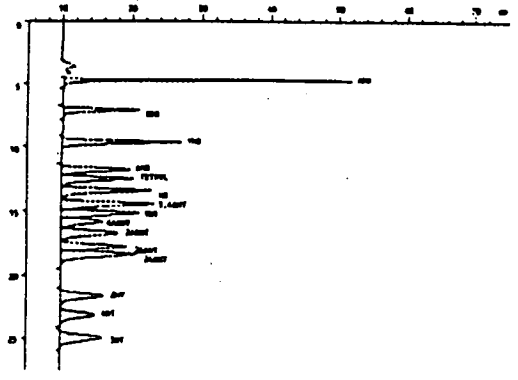
LONG PLOT

Injection F: <HCL> 5 SEE0516.7.1

Sample name.....: EXP L4 SOIL STD
Sample ID.....: 6-Dec-17:LA4
TEST ID: 1013, COL. TYPE: CGS-11, ID: CARB-02, INJ:200,COL:1

Acquired on 14-May-97 at 16:42:30
Reported on 16-May-97 at 17:28:17



INJECTION REPORT

Injection F: <HCL> 5 SEE0516.7.1

Acquired on 14-May-97 at 16:42:30
Modified on 15-May-01 at 16:17:42
Reported on 15-May-97 at 16:30:22

ANALYSIS INFORMATION

Analysis ID.....: EXP. 66 300
Analyst name.....: SS
Comment.....: TEST ID: 1013, COL. TYPE: CGS-11, ID: CARB-02, INJ:200,COL:1
Number of samples..: 64
Calibration file...: MEM0514 Last modified on 15-May-01 at 16:17:06
Method file.....: EXPLOS Last modified on 15-May-01 at 16:16:54
Method title.....: SW846-8330

SAMPLE INFORMATION

Sample name.....: EXP L4 SOIL STD
Sample ID.....: 6-Dec-17:LA4
Sample type.....: Standard
Sample amount.....: 1.0000
Number of injections.....: 1
Bottle number.....: 1

Retention time update.....: No update
Calibration level identifier.....: 6
Response factor update.....: Initialize

Dilution factor amount:
PERCENT MOISTURE.....: 0.000

Conc factors:
Volume (mL).....: 1.000
Dilution.....: 1.000
Amount (g or mL).....: 1.000

PEAK INFORMATION

Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
1	4.775	41808	338950	4.813	HMX
2	7.189	11065	177815	2.053	RDX
3	9.701	16976	288679	1.467	TNB
4	11.872	9938	200473	0.797	DNB
5	12.847	10283	211478	1.835	TETRYL
6	13.499	12790	284967	1.630	NB
7	14.501	13243	289110	2.492	3,4DNT
8	15.200	11216	257817	1.326	TNT
9	15.651	5891	161761	1.329	4ADNT
10	16.709	4090	231448	1.164	2ADNT

Peak	RT mins	Hght uV	Area uVs	ug/mL	Peak name
11	17.781	9476	234648	1.915	26DNT
12	18.411	10701	298173	1.168	24DNT
13	21.675	6152	183003	1.642	2NT
14	23.173	4966	152512	1.634	4NT
15	24.960	6007	195262	1.564	3NT
Total		178312	3508594	26.972	
Residual		0	0	0.000	

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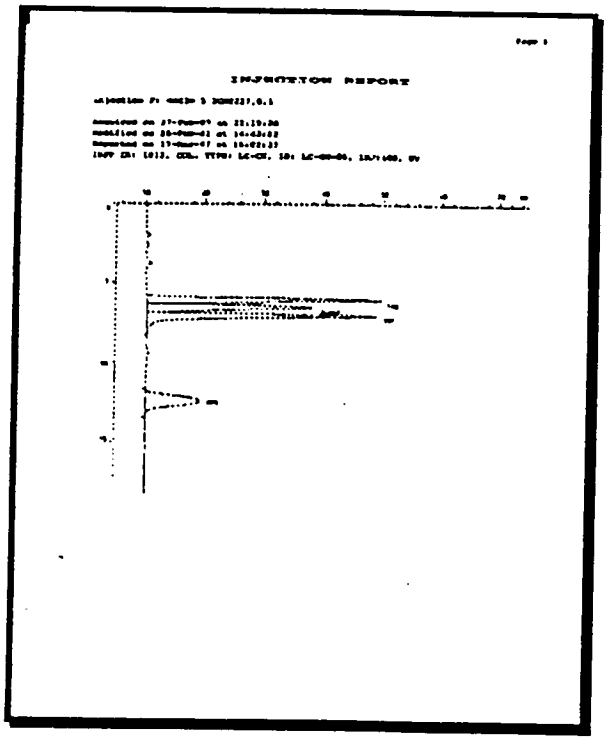
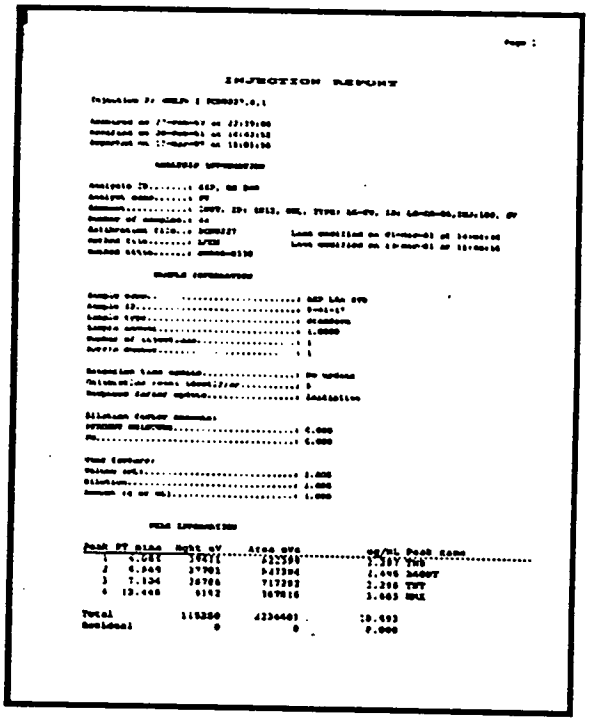

INJECTION REPORT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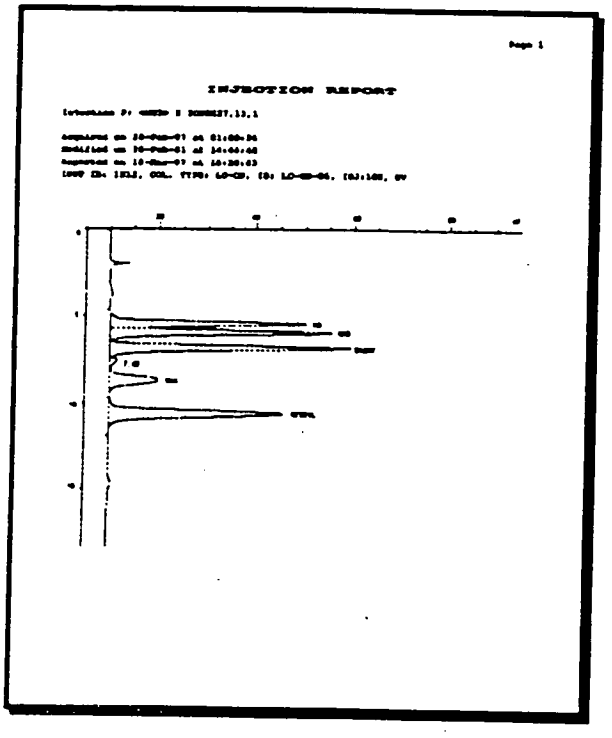
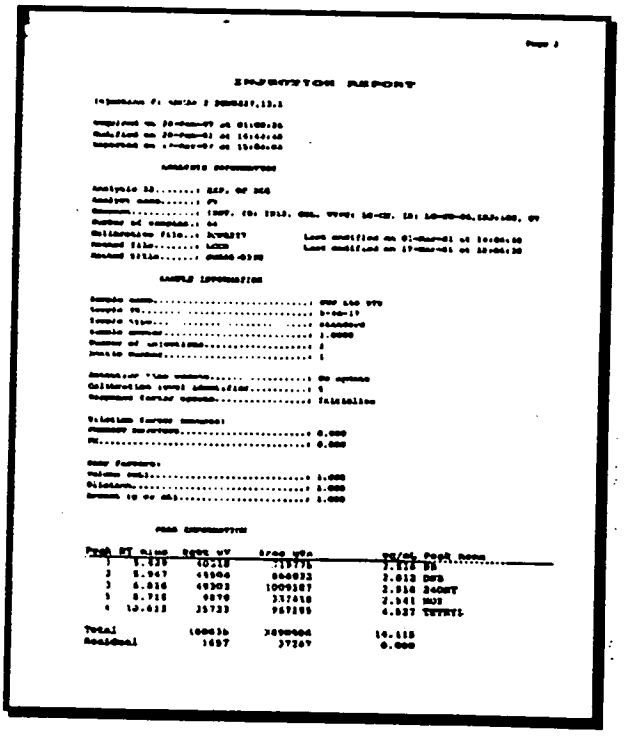

INJECTION REPORT


INJECTION REPORT

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PROCEDURAL DOCUMENTS - SYSTEM TRANSITION

Please note that the following document is in transition from a previous system. The document identification number (listed below) has been assigned in compliance with the SWL SOP titled:

"Preparation, Review, Revision, and Control of Procedural Documents", SWL-GA-101, Rev. 1.0 — 05/14/97

The ID given below integrates the following SOP into the new system set forth in GA-101 prior to its annual review (or conversion of format and numbering). Each ID is unique and a "key" field in the new document control database system. The assignment of a new number is therefore necessary for its tracking in the new system.

Upon revision or review, the following document will be re-issued in full compliance with the guidelines set forth in GA-101, therefore consistent with newly converted or written procedural documents.

Existing ID: MT913B

NEW DOCUMENT IDENTIFICATION NUMBER:

SWL-IN-209

Standard Operating Procedure

"Metals by SW846 Method 6010A
Inductively Coupled Argon Plasma (ICP)"
Rev. No. 1.2
Issued: 4/29/97

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SOUTHWEST LABORATORY OF OKLAHOMA, INC.

TM

Standard Operating Procedure

ICP SW3010A / 3050B Digestion and SW6010B Analysis

Document No.: SWL-IN-205

Rev No. / Date: Rev. 4.0 — 01/26/98

OFFICIAL COPY RED STAMP INDICATES ORIGINAL

APPROVALS	DATES
Procedure Prepared By	Date 1/26/98
Inorganic Program Manager	Date 1/26/98
Laboratory Safety Officer	Date 1/27/98
Laboratory QA/QC Officer	Date 1/27/98

(Effective Date is 5 calendar days after the last signature above - QA/QC Officer)

Document Status

[ ] Controlled DC No. _____ Issued to: _____ Date: _____

[✓] Official Copy OC No.: 2033 Issued to: Alan Fosdick Date: 2/6/98 mm

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ICP SW3010A / 3050B DIGESTION AND SW6010B ANALYSIS

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1.0 SCOPE AND APPLICATION

1.1 This SOP will give the digestion procedure for soil matrices for SW 3050B. These digestion procedures will be used for the preparation of sediments, sludges and solid waste samples for analysis by ICP spectroscopy.

1.2 This SOP will give the digestion procedure for aqueous samples for the SW 3010A. These digestion procedures will be used for the preparation of aqueous samples, drinking water, surface water, domestic and industrial wastewater, and wastes that contain suspended solids for analysis by ICP spectroscopy.

1.3 The Inorganics supervisor /or designee will designate the proper method to be used per batch of samples. If the method is not clearly identified on the internal custody forms, check with the Project Officer responsible for the samples before digestion is started.

1.4 This SOP will be used for the determination of dissolved, suspended or total metals in a variety of matrices by SW 6010B. Table 4 lists elements for which this method applies, with current wavelengths used on each of the ICP units. Elements and wavelengths will be added as new methods are developed or new instruments are acquired.

2.0 SUMMARY OF METHOD

2.1 A representative 1-2 g (wet weight) of sample is digested in nitric acid and hydrogen peroxide. The digestate is then refluxed with hydrochloric acid. The sample is then brought up to a volume of 100 ml.

2.2 A mixture of nitric acid and 100 ml of the sample is refluxed in a fleaker. After two hours or the volume is brought to 25 ml, it is refluxed with hydrochloric acid and brought up to volume.

2.3 Procedure Overview

2.3.1 Each morning the Inorganics supervisor /or designee will designate the day's work by sample batch and method to be completed. This will be done by giving the metals digestion technician a written list of batches to be done that day.

2.3.2 The technician will then locate samples in the walk-in refrigerator, log all samples out, and bring them back to the metal digestion area.

2.3.3 The technician will then make the computer generated digestion log according to the "Digestion/Distillation Log" SOP SWL-IN-105.

2.3.4 Follow the appropriate digestion method (for aqueous or soil matrices).

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2.4 Sample analysis - This method describes the technique for the simultaneous multi-element determination of trace metals in solution using a Thermal-Jarrel Ash ICAP-61 and ICAP61E Trace. The basis of this method is the measurement of atomic emission by an optical spectroscopic technique. Samples are nebulized and the aerosol that is produced is transported to the plasma torch where excitation occurs. Characteristic atomic-line emission spectra are produced by a radio-frequency generated inductively coupled plasma. The spectra are dispersed by a grating spectrometer and the intensities of the lines are monitored by photomultiplier tubes. The photo currents from the photomultiplier tubes are processed and controlled by the Thermo-Spec Software via the computer system.

3.0 HEALTH AND SAFETY

3.1 Refer to the current version of the "Laboratory Safety Plan" SOP (SWL-GA-111).

3.2 Each chemical and sample should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. This may be done by wearing gloves, eye protection, and laboratory coat during distillation and analysis.

3.3 The following chemicals have the potential to be highly toxic or hazardous, consult MSDS: Hydrochloric acid and Nitric acid.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

4.1 Samples should be collected in plastic or glass bottles. All bottles must be thoroughly cleaned and rinsed with reagent water.

4.2 Water samples must be preserved with nitric acid to a pH < 2. Soil samples must be maintained at 4° C.

5.0 INTERFERENCES AND POTENTIAL PROBLEMS

5.1 Spectral Interference

5.1.1 Overlap of a spectral line from another element is compensated with interelement corrections by the instrument.

5.1.2 Unresolved overlap of molecular band spectra may require selection of an alternate wavelength (if available).

5.1.3 Background contribution from continuous or recombination phenomena.

5.1.4 Background contribution from stray light from the line emission of high concentration elements.

Note: Number 5.1.3 and 5.1.4 interference's can usually be compensated by a background correction adjacent to the analyte line.

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5.2 Physical Interference - affects associated with the sample nebulization and transport processes. Dilution may be necessary.

6.0 EQUIPMENT / APPARATUS

6.1 Corning fleakers - 150 ml, or equivalent.

6.2 Whattman grade 41 filter paper or equivalent.

6.3 Analytical Balance - Capable of weighing to the nearest 0.01 g.

6.4 Disposable plastic funnels.

6.5 Electric Hotplate or equivalent - adjustable and capable of maintaining a temperature of 90-95^0 C.

6.6 Disposable Fisher polypropylene 100 ml vials or equivalent.

6.7 pH paper with a range of 1-6.

6.8 Eppendorfs & Oxford pipettes (or equivalent) – various ranges.

6.9 Ribbed watch glass or equivalent cover.

6.10 Class A volumetric flasks – various capacities.

6.11 Teflon or glass beads.

6.12 100 ml graduated cylinders.

6.13 Thermal-Jarrel Ash ICAP-61 and ICAP-61E Trace optical emission spectrophometer.

6.14 IBM PC/AT compatible computer with Thermospec operating software, with automated background correction and interelement corrections.

6.15 Radio frequency power supply with 2500 watts capacity.

6.16 Liquid argon supply, 4300 cu. Ft.

6.17 Thermo Jarrell Ash Autosampler with 300 sample positions and 19 QA/QC position.

6.18 Printer

6.19 Sampler tubes for autosampler

6.20 Peristaltic Pump (TJA 61)

6.21 Peristaltic Pump tubing.

7.0 REAGENTS

7.1 Acids used in the preparation of standards and for sample processing must conform to specifications of the Committee on Analytical Reagents of the American Chemical Society (ACS) and be of the Mallinckrodt ArSelect grade or the equivalent.

7.1.1 Nitric Acid (HNO_3), concentrated

7.1.2 Nitric Acid (1:1) - Add 500 ml of nitric acid to 400 ml of ASTM Type II water and dilute to 1 L.

7.1.3 Hydrochloric Acid (HCl), concentrated

7.1.4 Hydrochloric Acid (1+1) - Add 500 ml of hydrochloric acid to 400 ml of ASTM Type II water and dilute to 1 L.

7.1.5 Hydrogen Peroxide (30%), H_2O_2

7.2 Water - For all sample preparations and dilutions, ASTM Type II water will be used. This water is prepared by passing distilled water through a mixed bead of anion and cation exchange resins.

7.3 Standard Stock Solutions - Spex Industries, Inc., High Purity Standards, Inc. Inorganic Ventures, Inc. or equivalent traceable source- prepared from 99.995% - 99.999% pure elements assayed by wet chemical methods dissolved in 18 mega-ohm, double deionized water and electronic grade acids. Date solutions are received and opened are posted on each bottle. All standards should be traceable to national standards such as NIST.

7.4 Spiking Standards - All mixed and individual elemental standards must be NIST traceable or equivalent.

7.4.1 SS1 - A stock standard used to make a working spike for the matrix spikes, LCS, & LCSD. See Table 1 for elements, concentration requirements, and shelf life.

7.4.2 SS2 - A stock standard used to make a working spike for the matrix spikes, LCS, & LCSD. See Table 1 for elements, concentration requirements, and shelf life.

7.4.3 SS3LS - A stock standard used to make a working spike for the LCS & LCSD. See Table 1 for elements, concentration requirements, and shelf life.

7.4.4 SS3MS - A stock standard used to make a working spike for the matrix spikes. See Table 1 for elements, concentration requirements, and shelf life.

7.4.5 SS4 - A stock standard used to make a working spike for the matrix spikes, LCS, & LCSD. See Table 1 for elements, concentration requirements, and shelf life.

7.4.6 SS5 - A stock standard used to make a working spike for the matrix spikes, LCS, & LCSD. See Table 1 for elements, concentration requirements, and shelf life.

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7.4.7 SS7 - A stock standard used to make a working spike for the matrix spikes, LCS, & LCSD. See Table 1 for elements, concentration requirements, and shelf life.

7.5 Calibration Solutions - Calibration Solution (STD0, STD1....STDn)- These standards are prepared in a 5% HNO_3 solution at concentrations recommended by the instrumentation manufacturer or as specified by the client contract and brought to volume with ASTM Type II water. Any Stock Solutions that meet the requirements as listed in Section 7.3 can be used to prepare these solutions.

7.5.1 STD0- The STD0 calibrating solution is prepared by the following - dilute 5.0 ml of HNO_3 (conc.) to 100 ml with ASTM Type II water.

7.5.2 STD1- The STD1 calibrating solution is prepared at the concentrations listed in Table 5 and Table 6. Prepare (at minimum) every two months.

7.5.3 STD2- The STD2 calibrating solution is prepared at the concentrations listed in Table 5 and Table 6. Prepare (at minimum) every two months.

7.6 Calibration Check Standards (ICV, ICSA, ICSAB, CRI and CCV):

7.6.1 Initial Calibration Verification standard (ICV)-Prepared from a source of standard solutions other than those used for the calibration solutions and preferably from a different vendor. Prepare (at minimum) every two months.

7.6.2 Interference Standards A and AB (ICSA, ICSAB)-The interference solutions are prepared at the concentrations listed in Table 7. Prepare (at minimum) every two months.

7.6.3 SW846 Contract Required Analysis Standard (CRI)-The CRI solution is prepared at the levels listed in Table 8 (approximately 2x reporting level). Prepare (at minimum) every two months.

7.6.4 Continuing Calibration Verification solution (CCV)- Use STD2 (section 7.5.3) as this standard.

7.6.5 Calibration Blanks (ICB / CCB)- Use STD0 (section 7.5.1) as this standard.

7.6 All above standards (7.3 through 7.5) must be recorded in the Multi-Standard Preparation Log. Refer to the current version of the "Inorganic Traceablity of Standards" SOP SWL-IN-104.

8.0 PROCEDURE

8.1 Digestion of Aqueous Matrices (Method 3010A)

8.1.1 Take the pH of all the samples with dual range pH paper. Record as <2 or >2 on your digestion log in the pH column. If any sample is >2 pH the digestion supervisor and the Project Officer must be immediately notified for a decision on how to proceed with sample. Note in comments section actions taken.

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8.1.2 Shake the sample thoroughly to achieve homogeneity.

8.1.3 Pre-label all disposable polypropylene 100 ml vials and 150 ml digestion fleakers, with the assigned laboratory ID number.

8.1.4 Pour sample up to the 100 ml mark on the polypropylene vial and then transfer to the 150 ml digestion fleaker (labeled with the same laboratory ID number) – rinsing vial out three times with DI water, pouring rinse into the digestion fleaker.

8.1.4 Make the appropriate QA/QC samples as described in Section 10.0.

8.1.5 Add 3 ml of concentrated HNO_3.

8.1.6 Heat on a hot plate or equivalent heating source and cautiously evaporate to a low volume (less than 15 ml), making certain that the sample does not boil and that no portion of the bottom of the fleaker is allowed to go dry. All samples in a batch must be located on the same hotplate.

8.1.7 Cool the fleaker. Add another 3 ml portion of concentrated HNO3. Cover fleaker with a nonribbed watch glass and return to hotplate.

8.1.8 Increase the temperature of the hot plate so that a gentle reflux action occurs.

Note: If a sample is allowed to go to dryness, low recoveries will result. Should this occur, discard the sample and reprepare.

8.1.9 Continue heating, adding additional acid as necessary (in 3 ml aliquots), until the digestion is complete (generally indicated when the digestate is light in color or does not change in appearance with continued refluxing).

8.1.10 Rinse watch glass into the fleaker with ASTM Type II water. Uncover the fleaker, and evaporate to a low volume (less than 10 ml), not allowing any portion of the bottom of the fleaker to go dry.

8.1.11 Cool fleaker. Add 10 ml 1:1 HCl, cover the fleaker, and reflux for an additional 15 minutes to dissolve any precipitate or residue resulting from evaporation.

8.1.12 Rinse watch glass into the fleaker with ASTM Type II water. Cool the sample and filter into the appropriate named polypropylene vial to remove insoluble material that could clog the ICP nebilizer. Rinse fleaker into the filter three times with ASTM Type II water. NOTE: Filtering of the sample is not required for samples that do not contain insoluble material.

8.1.13 After the filtering is complete adjust the volume to 100 ml mark with ASTM Type II water.

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8.2 Digestion of Soil Matrices (Method 3050B)

8.2.1 Mix the sample thoroughly to achieve homogeneity. Follow SOP SWL-GA-140, "Homongenzation of Soil, Sedimemt, and Sludge samples".

8.2.2 Weigh 1.00 - 2.00 gram portion of sample and transfer to a fleaker. Record to the nearest 0.01 g.

8.2.3 Make the appropriate QA/QC samples as described in Section 10.0.

8.2.4 Add 10 ml of (1+1) HNO_3 mix and cover with a watch glass.

8.2.5 Heat the sample to 95^0 C ± 5^0 C and reflux for 10-15 minutes <u>without</u> boiling. All samples in a batch must be located on the same hotplate.

8.2.6 Allow the sample to cool, then add 5 ml of HNO_3. Replace the watch glass and reflux for two hours or until the solution evaporates to approximately 5 ml. Maintain a covering of solution over the bottom of the vessal at all times.

8.2.7 Allow the sample to cool, then add 2 ml ASTM Type II water and 3 ml of 30% hydrogen peroxide (H_2O_2).

8.2.8 Return the beaker for heating to start the peroxide reaction. Heat until effervescence subsides.

8.2.9 Continue to add 30% H_2O_2 in 1.0 ml aliquots with warming until the effervescence is minimal. Do not add more than a total of 10 ml 30% H_2O_2.

8.2.10 Cover sample with ribbed watch glass and continue heating the acid-peroxide digestate until the volume has been reduced to approximately 5 ml or heat at 95^0 C ± 5^0 C without boiling for two hours. Maintain a covering of solution over the bottom of the vessel at all times.

8.2.11 Allow the sample to cool. Add 10.0 ml of concentrated HCl and cover with watch glass. Heat and reflux sample at 95^0 C ± 5^0 C and reflux for 15 minutes <u>without</u> boiling.

8.2.12 Rinse watch glass into the fleaker with ASTM Type II water. Cool the sample and filter to remove insoluble material that could clog the ICP nebulizer. Rinse fleaker into the filter three times with ASTM Type II water.

8.2.13 After the filtering is complete adjust the volume to 100 ml with ASTM Type II water. This will be done by measuring reagent water into a 100 ml Class "A" volumetric flask and then transferring the water into a disposable Fisher polypropylene 100 ml vial or equivalent. This vial will then be set side by side with the sample to be brought up to volume for use as a measuring device.

8.3 Digestion of Soil Matrices (Method 3050B - alternative) – This procedure for soil digestion will be used to meet client requirements. The below procedure is used to improve the solubilities and recoveries of antimony, barium, lead, and silver.

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8.3.1 Mix the sample thoroughly to achieve homogeneity. Follow SOP SWL-GA-140, "Homongenzation of Soil, Sedimemt, and Sludge samples".

8.3.2 Weigh 1.00 - 2.00 gram portion of sample and transfer to a fleaker. Record to the nearest 0.01 g.

8.3.3 Make the appropriate QA/QC samples as described in Section 10.0.

8.3.4 Add 2.5 ml of conc. HNO_3 and 10 ml conc. HCl. Cover with a watch glass. Heat the sample to 95^0 C ± 5^0 C and reflux for 10-15 minutes <u>without</u> boiling. All samples in a batch must be located on the same hotplate.

8.3.5 Filter the digestate and collect filtrate in into a disposable Fisher polypropylene 100 ml vial or equivalent. Wash the filter paper, while still in the funnel, with no more than 5 ml of hot HCl (~95^0 C), then with 20 ml of hot DI water (~95^0 C). Collect washings in the same container.

8.3.6 Remove the filter and residue from the funnel, and place them back in the fleaker. Add 5 ml of conc. HCl and heat the sample to 95^0 C ± 5^0 C until the filter paper dissolves.

8.3.7 Filter the residue and collect the filtrate in the same 100 ml container. Allow filtrate to cool, then dilute to 100 ml.

8.3.8 If a precipitate forms on the bottom of a flask, add up to 10 ml of conc. HCl to dissolve the precipitate. After precipitate is dissolved, dilute to volume with reagent water.

8.4 General Procedures for all Digestions.

8.4.1 All designated dissolved metal analysis will be taken through the digestion procedure, unless directed otherwise. If the dissolved metals have not been field filtered, the technician will filter the sample through Whatman Grade 4. filter paper (or equivalent) before any digestion steps are taken.

8.4.2 Document all distillation details in the Sample Distillation Record Log, the analyst must then sign and date the log. Refer to "Digestion/Distillation Log" SOP SWL-IN-105 for procedure and forms. Record the Standards Batch ID # on each Digestion Log that is currently used for recording the reagent lot #'s for ICP digestions (see Figure 1)

8.4.3 Place all samples and QA/QC samples in a container with a copy of the digestion log and give to ICP group for analysis.

8.5 Instrument Preventive Maintenance - Each instrument has a Preventive Maintenance Logbook, that list items that need to be checked. Follow the current version of SOP SWL-INPREMA.

8.6 General Start-Up for all instruments

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8.6.1 Check that the argon tank main valve is open and the pressure is set to at least 90 psi.

8.6.2 Check that the drain tube is inserted into a plastic water bottle containing at least 8 inches of water.

8.6.3 Replace old pump tubing if necessary.

8.7 Start up procedure for TJA ET1 and TJAET2 (TJA Trace units):

8.7.1 Engage reset from back of ICP.

8.7.2 Engage reset on front panel of ICP.

8.7.3 Reset and turn on the computer with ThermoSPEC software.

8.7.4 Access Plasma "control panel" under setup, and select "Startup".

8.7.5 When Plasma ignites, adjust optimize nebulizer pressure (usually at ~ 25 PSI) and pump speed at ~ 130 let plasma stabilize 30 minutes to one hour.

8.7.6 Profile with a 1000 ug/l Arsenic solution as instructed in instrument manual.

8.8 Start up procedure for TJA61E:

8.8.1 Turn on argon switches to torch, auxiliary, and sample. Close needle valve on sample argon.

8.8.2 Set auxiliary to 1.5 LPM and purge 10-15 minutes.

8.8.3 Ensure automatic power control is set to manual position and power dial is fully counter-clockwise.

8.8.4 Turn on R.F. power, increase power with power dial until approximately 0.5 kilowatt.

8.8.5 Press ignitor button and slowly increase power until plasma forms.

8.8.6 Switch power control from manual to automatic.

8.8.7 Decrease auxiliary argon flow to 0.4 LPM, and let plasma stabilize for 20 minutes.

8.8.8 Turn on peristaltic pump and open sample argon needle value to 0.6 LPM.

8.8.9 Close argon auxiliary flow switch.

8.8.10 Check flow of peristaltic pump and nebulizer operation.

8.8.11 Let plasma stabilize for one hour.

8.8.12 Profile with a 1000 ug/l Copper solution as instructed in Instrument Manual.

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8.9 Analysis

8.9.1 Set up the instrument with proper operating parameters already established. The instrument must be allowed to become thermally stable before beginning. This usually requires at least 30 minutes of operation prior to calibration.

8.9.2 Initiate appropriate method file configuration on computer.

8.9.3 Setup the analytical sequence on the ICP Autosampler table, following the sequence on Table 9.

8.9.4 Load samples into the Autosampler according to the autosampler table (see Figure 2). Review before you place each sample and sample position that it matches with the stated position in autosampler table. After the sample has been placed into place double check for accuracy.

8.9.5 Start the analysis of the analytical sequence.

8.9.6 When run is complete, copy electronic version of analytical run to the Inorganic Database.

8.9.7 Using the Inorganic Database printout "Run Log" (see Figure 3) and "Re-run Log" (see Figure 4), and verify QC checks on the analytical sequence and Prep Batch sequence.

8.9.8 Review Data according requirements in Section 10.0 and 12.0 of this SOP, and SOP SWL-IN-106; "1st and 2nd review of ICP, GFAA, Mercury & Cyanide".

8.9.9 The current standard list is then attached to the run (see ICP Standard Batch Form, Figure 5), raw data, and Reanalysis, or Non-conformance forms (if needed) are presented for a secondary or peer review.

9.0 CALCULATIONS

9.1 Calculations: The below calculation is used to calculate the final Concentration of the various stock spiking standards. All units must be the same for C_I & C_F, also for V_I & V_F for the formula to be valid.

$$C_F = C_I \times V_I / V_F$$

where:

C_F = Final Concentration of Standard

C_I = Initial Concentration of Standard

V_I = Final Volume of Standard

V_F = Initial Volume of Standard

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Units normally used for concentration are the following:

ug/l or PPB = 1/1000 mg/l

mg/l or PPM = ug/mL = 1000 ug/L

9.2 If a dilution was performed, the appropriate factor must be applied to sample values.

9.3 Data will be reported in ug/L off the raw data for all water samples.

9.4 Soils: mg/kg (wet weight) = (C x V) / W

Where:

C	=	Concentration in ug/l
V	=	Volume of digestate in Liters
W	=	Weight of sample in grams

9.5 Report significant figures as follows:

Result <10 to 2 significant figures

Result >10 to 3 significant figures

9.6 Report all results to the PQL. Results between the MDL and PQL will be flagged with a "B" on CLP- LIKE packages.

10.0 QUALITY ASSURANCE / QUALITY CONTROL

10.1 The following QA/QC samples will be designated according to the following format:

"XXXYYMMDDTZ" where:

"XXX"- designates the type of sample (PBW, PBS, LCW, LCS, LDW or LDS)

"YY" - is the last two digits in the current year

"MM" - is the month

"DD" - is the day of the month

"T" - designates the type of digestion ("F" for GFAA)

"Z" - references the incremental batch letter done that day ("A" being the first batch of the day, "D" being the fourth)

10.2 Aqueous Digestion QA/QC:

10.2.1 PBW - Is the Method Blank. Bring ASTM Type II water to the 100 ml mark on the fleaker.

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10.2.2 LCW - Is the Laboratory Control Sample and is made from the following:

Add 1 ml of SS1, SS2, & SS3LS. If any sample in batch requires As, Pb, Se, or Tl by Trace spike the sample with 1 ml SS1, SS3LS, and SS5. Then dilute to the 100 ml mark on the fleaker with ASTM Type II water.

10.2.3 LDW - Is the Laboratory Control Sample Duplicate and is made from the following:

Add 1 ml of SS1, SS2, & SS3LS. If any sample in batch requires As, Pb, Se, or Tl by Trace spike the sample with 1 ml SS1, SS3LS, and SS5. Then dilute to the 100 ml mark on the fleaker with ASTM Type II water.

10.2.4 Matrix Spike (MS) - is made from the following:

Spike the sample with 1 ml of SS1, SS2, & SS3MS. If any sample in the batch requires As, Pb, Se, or Tl by Trace ICP, spike the sample with 1 ml SS1, SS3MS, and SS5.

10.2.5 Matrix Spike Duplicate (MSD) - is made from the following:

Spike the sample with 1 ml of SS1, SS2, & SS3MS. If any sample in the batch requires As, Pb, Se, or Tl by Trace ICP, spike the sample with 1 ml SS1, SS3MS, and SS5.

10.2.6 Matrix Duplicate (D): This sample is made by digesting a second aliquot of this sample to determine the precision of the method.

10.3 Soil Digestion QA/QC:

10.3.1 PBS - Is the Method Blank. Add 1.0 ml of water into the fleaker

10.3.2 LCS - Laboratory Control Sample and is made from the following:

Add 1.0 gram of glass beads (use Teflon beads if silicon is required) and 1 ml of SS1, SS2, & SS3LS. If any sample in batch requires As, Pb, Se, or Tl by Trace ICP spike the sample with 1 ml SS1, SS3LS, SS5, and SS7.

10.3.3 LDS - Is the Laboratory Control Sample Duplicate and is made from the following:

Add 1.0 gram of glass beads (use Teflon beads if silicon is required) and 1 ml of SS1, SS2, & SS3LS. If any sample in batch requires As, Pb, Se, or Tl by Trace ICP spike the sample with 1 ml SS1, SS3LS, SS5, and SS7.

10.3.4 Matrix Spike (MS) - is made from the following:

Spike directly on the sample (before any acid has been added) with 1 ml of SS1, SS2, SS3MS and SS7. If any sample in the batch requires As, Pb, Se, or Tl by Trace ICP, spike the sample with 1 ml SS1, SS3MS, SS5, and SS7.

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10.3.5 Matrix Spike Duplicate (MSD) - is made from the following:

Spike directly on the sample (before any acid has been added) with 1 ml of SS1, SS2, SS3MS, and SS7. If any sample in the batch requires As, Pb, Se, or Tl by Trace ICP, spike the sample with 1 ml SS1, SS3MS, SS5, and SS7.

10.3.6 Matrix Duplicate (D) - This sample is made by digesting a second aliquot of the same sample to determine the precision of the method.

10.4 Digestion QA/QC Frequency Requirements:

10.4.1 A PBW, LCW & LDW (or PBS, LCS, & LDS) are required for every batch of samples. (maximum number of samples in a batch is 20 samples).

10.4.2 A MS/MSD (or a MS/D if the client request a duplicate instead of a MSD) are required for every batch samples. (maximum number of samples in a batch is 20 samples).

Note: The final concentrations of the LCS, LDS, matrix spike, and matrix spike duplicate is noted in Tables 2 and 3. Before use, the spiking stock standards must be screened on the ICP and have a 90 - 110% Recovery of the true value.

10.5 Demonstration of Performance

10.5.1 MDL Study - Follow the current version of SOP SWL-GA-113, "Definition and Determination of Detection Limits".

10.5.2 Linear Dynamic Range (LDR) - The upper limit of the LDR must be established for each wavelength utilized. The LDR will be determined by analyzing succeedingly higher standard concentrations (minimum of three standards) of the analyte until the observed analyte concentration is no more than ± 5% the true value. The LDR will be verified on a bi-annually basis (document with the EPA LRS study), or a change in instrument hardware or operating conditions would dictate they be re-determined. If an element is over 90% of the LDR, it will be required to be diluted.

10.5.3 Interelement Corrections - Interelement corrections are determined by analyzing standards at or near the linear range of the interferants and determining the effect on the analytes (a correction is required if the analyte falls ± the reporting limit). Recalculation of Interelement correction factors are required at a minimum every six months, or when a change in instrument hardware or operating conditions would dictate they be re-determined.

10.6 Calibration

10.6.1 Calibration is required every 24 hours.

10.6.2 Calibration will consist of STD0 and STD1

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10.7 Calibration Verification Checks

10.7.1 Initial Calibration Verification (ICV) - After the ICP has been calibrated, the accuracy of the initial calibration is verified and documented for every analyte by the analysis of Initial Calibration Verification Solution at each wavelength used for analysis. If the measurements exceed the control limits of 90-110% R of the true value, the analysis must be terminated, the problem corrected, the instrument re-calibrated, and the calibration re-verified for those analytes.

10.7.2 Continuing Calibration Verification (CCV) - To ensure calibration verification during the analysis run, a continuing calibration verification standard is analyzed for every analyte at each wavelength at a frequency of at least every 10 samples or every 2 hours during an analysis run, whichever is more frequent. The standard is also analyzed for every wavelength used for analysis at the beginning of the run and after the last analytical sample. If the measurements exceed the control limits of 90-110 % R of the true value, the instrument must be re-calibrated and all of the samples since the last successful CRI, ICSA, ICSAB, CCV, CCB set analyzed must be reanalyzed.

10.7.3 Initial and Continuing Calibration Blank (ICB and CCB) - A calibration blank is analyzed at each wavelength used for analysis immediately after every initial and continuing calibration verification, at a frequency of 10% or every 2 hours during the run, whichever is more frequent. If the absolute value of the blank exceeds 3x IDL, or the PQL whichever is the lowest value, terminate analysis, correct the problem, re-calibrate and reanalyze all analytical samples for those analytes.

10.7.4 Interference Check Samples (ICSA and ICSAB) - To verify interelement and background correction factors an ICP interference check sample is analyzed at the beginning and end of each analysis run or a minimum of twice per 8 hour working shift, whichever is more frequent. The ICS consists of two solutions, solution ICSA consists of the interferants and solution ICSAB consists of the analytes mixed with the interferants. An ICS analysis consists of analyzing both solutions consecutively for all wavelengths used.

Note: Results for the analyses of solution ICSAB must fall within the control limits of ± 20% of the true value for the analytes. If not, terminate the analysis, correct the problem, re-calibrate and reanalyze the analytical samples analyzed since the last good ICS.

Note: Results for the ICSA solution should be within 20 % of the true values for the interferants. For all other elements the result should be within ± 2x PQL. If not, the analyst should review data and re-analyze samples with suspect results. Acceptance of data not meeting the above requirements will require a Non-conformance report, explaining why data is valid.

10.7.5 Contract Required Analysis standard (CRI) - To verify linearity near the lower end of the curve an ICP standard at 2x the PQL is analyzed at the beginning and end of each sample analysis, or a minimum of every 8 hours, whichever is more frequent. Control limits are 70-130 % recovery. If not, the analyst should review data and re-analyze samples with suspect results. Acceptance of data not meeting the above requirements will require a Non-conformance report, explaining why data is valid.

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10.8 Method Performance Checks

10.8.1 Preparation Blank (PBW or PBS) - At least one preparation blank must be prepared and analyzed with every Sample Delivery Group (SDG), or with each batch of samples digested, whichever is more frequent. This blank is used to ascertain whether sample concentrations reflect contamination in the following manner:

10.8.1.1 If the absolute value of the blank is less than the PQL no corrective action is necessary.

10.8.1.2 If any analyte concentration in the blank is above the PQL the lowest concentration of that analyte in the associated samples must be 10x the blank concentration. If not, all samples less than 10x the blank concentration and above the PQL must be re-digested and reanalyzed for that analyte.

10.8.1.3 If the concentration of the blank is below the -PQL, all samples associated with the blank must be re-digested and reanalyzed if they are not greater than 10x the absolute value of the preparation blank.

10.8.2 Laboratory Control Sample (LCS/LDS) - A LCS is analyzed for each analyte using the same sample preparations, analytical methods and QA/QC procedures employed for the samples. One LCS/LDS is prepared and analyzed for every group of similar matrix samples in a batch of samples digested. If the percent recovery for the LCS falls outside the control limits of 80-120%, Client requirement, or in-house control limits the analyses must be terminated and the samples associated with that LCS re-digested and reanalyzed.

10.8.3 ICP Serial Dilution (L) - The ICP Serial Dilution analysis is performed on each batch of samples of a similar matrix type and concentration. This sample should be flagged with an "L". Samples identified as field blanks cannot be used for serial dilution analysis. If the analyte concentration is at least a factor of 50 above the method detection limit in the original sample, an analysis of a 5 fold dilution must agree within 10% of the original determination. If not, flag data with an "E".

10.8.4 Matrix Spike/Matrix Spike Duplicate Sample (MS / MSD) - The spike solution is added before digestion to provide information about the effect of the sample matrix on the digestion and measurement methodology. If the spike recovery is not within the limits of 75-125%, the data of all samples received associated with that spike sample and determined by the same analytical method will be flagged with the letter "N". A Post Digestion Spike will be done for that element using the sample that was originally associated with the sample for the matrix spike. An exception to this is when the sample concentration exceeds the spike concentration by a factor of four or more. Samples identified as field blanks cannot be used for spiked sample analysis.

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10.8.5 Post Digested Spike (A) - When the pre-digested spike recovery falls outside the control limits and the sample result does not exceed 4x the spike added, a post-digested spike is performed. The sample ID should be flagged with an "A". Spike the unspiked aliquot of the sample at 2x the indigenous level or 2x PQL whichever is greater.

10.8.6 Duplicate Sample (D) - A duplicate sample is analyzed only when requested by the client. Samples identified as field blanks cannot be used for duplicate sample analysis. A control limit of 20% for relative percent difference (RPD) is used for original and duplicate sample values greater than or equal to 5x PQL. A control limit of ±PQL is used for sample values less than 5x PQL. Samples not meeting this criteria will be flagged.

11.0 **WASTE DISPOSAL** - Refer to the current version of the SOP SWL-GA-114 ,"Hazardous Waste Management Plan".

12.0 METHOD PERFORMANCE

12.1 The current MDL or IDL studies. Summaries are provided in the Red Book and see the Inorganic Program Manager for raw data.

12.2 The LRS studies - see the Inorganic Program Manager.

12.3 See summaries of the LCS control charts in the "Blue book".

12.4 IEC calculations - see the Inorganic Program Manager.

13.0 REFERENCES

13.1 *U.S. EPA SW846*, Method 3010A, Revision 1.0, July 1992

13.2 *U.S. EPA SW846*, Method 3050B, Revision 2.0, December 1996

13.3 *U.S. EPA SW846*, Method 6010B, Revision 2.0, December 1996

13.4 Southwest Laboratory of Ok. SOP's as listed in body of text.

14.0 DEFINITIONS

14.1 Material Safety Data Sheet (MSDS): Information provided by commercial vendors for the chemical products prepared by their company. The information on the MSDS includes but is not limited to composition, physical properties, hazards identification, first aid measures, emergency clean-up and /or response, exposure and storage.

14.2 Method Detection Limit (MDL): A detection limit derived from seven samples taken through the entire method.

14.3 Practical Detection Limit (PQL): SWL reporting limit based on a factor times the MDL.

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14.4 Sample Batch: Consists of a maximum of twenty samples per matrix.

14.5 Preparation Blank (water-PBW, soil-PBS): Method blank, consisting of 0.25 N NaOH, taken through digestion and analysis to check for contamination in the system.

14.6 Laboratory Control Sample (water-LCW, soil-LCS): A spiked sample (usually water) taken through digestion and analysis to check the system's accuracy.

14.7 Laboratory Duplicate Control Sample (water-LDW, soil-LDS): A spiked sample (usually water) taken through digestion and analysis to check the system's accuracy and precision.

14.8 Matrix Spike (MS): A spiked sample taken through digestion and analysis to check the accuracy of the matrix. This is designed to provide information about the effect of the sample matrix on the digestion and/or measurement methodology

14.9 Matrix Spike Duplicate (MSD): A spiked sample taken through digestion and analysis to check the accuracy and precision of the matrix. This is designed to provide information about the effect of the sample matrix on the digestion and/or measurement methodology

14.10 Duplicate Sample: A sample taken through digestion and analysis to check the precision of the matrix.

14.11 Redbook – A listing of all IDL, MDL, and PQL for the laboratory.

14.12 Bluebook – Summary listing of in-house control limits.

14.13 Initial Calibration Verification standard (ICV)-Prepared from a source of standard solutions other than those used for the calibration solutions and preferably from a different vendor. Used to verify calibration of instrument.

14.14 Interference Standards A and AB (ICSA, ICSAB)-The interference solutions are prepared at the concentrations listed in Table 7. Used to verify the IEC calculations are being performed correctly.

14.15 SW846 Contract Required Analysis Standard (CRI)-The CRI solution is prepared at the levels listed in Table 8 (approximately 2x reporting level), used to verify results at the reporting levels.

14.16 Continuing Calibration Verification solution (CCV)- A mid-range standard used to verify calibration throughout analysis.

14.17 Calibration Blanks (ICB / CCB) – A standard blank used to verify calibration throughout analysis.

15.0 ATTACHMENTS

15.1 Table 1 - Concentration of SS1, SS2, SS3LS, SS3MS, SS4, and SS5 Stock Standards

15.2 Table 2 - Final Concentration of LCS/LDS

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15.3 Table 3 - Final Concentration of MS/MSD

15.4 Table 4 - Elements and Corresponding Wavelengths by Instrument

15.5 Table 5 - ICP (TJA 61) Calibration Standard Concentrations

15.6 Table 6 - ICP (Trace) Calibration Standard Concentrations

15.7 Table 7 - ICP ICSA / ICSAB Standard Concentrations

15.8 Table 8 - ICP CRI values

15.9 Table 9 - ICP Analytical Sequence

15.10 Figure 1 - Standard Batch Form for ICP Digestions

15.11 Figure 2 - ICP Autosampler Table

15.12 Figure 3 - ICP Run Log

15.13 Figure 4 – ICP Re-Run Log

15.14 Figure 5 – ICP Standard Batch Form

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TABLE 1

Concentration of SS1, SS2, SS3LS, SS3MS, SS4, and SS5 Stock Standards

ELEMENT	SS1 (mg/L)	SS2 (mg/L)	SS3LS (mg/L)	SS3MS (mg/L)	SS4 (mg/L)	SS5 (mg/L)	SS7 (mg/L)
Al	200						
Sb	50						
As		200			4	8	
Ba			25	200			
Be	5						
B			25	100			
Cd	5						
Ca			500	2000			
Cr	20						
Co	50						
Cu	25						
Fe	100						
Pb		50			2	8	
Mg			500	2000			
Mn	50						
Mo			20	100			
Ni	50						
K			1000	2000			
Sc			20	50			
Se		200			1	8	
Si			100	100			
Ag							5
Na			1000	2000			
Sr			20	100			
Tl		200			5	8	
Ti			25	100			
Sn			25	100			
V	50						
Zn	50						

Note: All above standards will have a maximum shelf life of 6 months.

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TABLE 2

Final Concentration of LCS/LDS

ELEMENT	ICP LCS/LDS (ug/L)	ICP LCS/LDS (mg/Kg)	TRACE LCS/LDS (ug/L)	TRACE LCS/LDS (mg/Kg)*
Al	2,000	200	2,000	200
Sb	500	50	500	50
As	2,000	200	80	8
Ba	250	25	250	25
Be	50	5	50	5
B	250	25	250	25
Cd	50	5	50	5
Ca	5,000	500	5,000	500
Cr	200	20	200	20
Co	500	50	500	50
Cu	250	25	250	25
Fe	1,000	100	1,000	100
Pb	500	50	80	8
Mg	5,000	500	5,000	500
Mn	500	50	500	50
Mo	200	20	200	20
Ni	500	50	500	50
K	10,000	1,000	10,000	1,000
Sc	200	20	200	20
Se	2,000	200	80	8
Si	1,000	100	1,000	100
Ag	50	5	50	5
Na	10,000	1,000	10,000	1,000
Sr	200	20	200	20
Tl	2,000	200	80	8
Ti	250	25	250	25
Sn	250	25	250	25
U	--	--	500	50
V	500	50	500	50
Zn	500	50	500	50

** Values in mg/kg are based on 1g to 100ml*

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TABLE 3

Final Concentration of MS/MSD

ELEMENT	ICP MS/MSD (ug/L)	ICP MS/MSD (mg/Kg)	TRACE MS/MSD (ug/L)	TRACE MS/MSD (mg/Kg)*
Al	2,000	200	2,000	200
Sb	500	50	500	50
As	2,000	200	80	8
Ba	2,000	200	2,000	200
Be	50	5	50	5
B	1,000	100	1,000	100
Cd	50	5	50	5
Ca	20,000	2,000	20,000	2,000
Cr	200	20	200	20
Co	500	50	500	50
Cu	250	25	250	25
Fe	1,000	100	1,000	100
Pb	500	50	80	8
Mg	20,000	2,000	20,000	2,000
Mn	500	50	500	50
Mo	1,000	100	1,000	100
Ni	500	50	500	50
K	20,000	2,000	20,000	2,000
Sc	500	50	500	50
Se	2,000	200	80	8
Si	1,000	100	1,000	100
Ag	50	5	50	5
Na	20,000	2,000	20,000	2,000
Sr	1,000	100	1,000	100
Tl	2,000	200	80	8
Ti	1,000	100	1,000	100
Sn	1,000	100	1,000	100
U	--	--	500	50
V	500	50	500	50
Zn	500	50	500	50

** Values in mg/kg are based on 1g to 100ml*

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TABLE 4

Elements and Corresponding Wavelengths by Instrument

ELEMENT	*TJA ET1* *Wavelength (nm)*	*TJA ET2* *Wavelength (nm)*	*TJA 61* *Wavelength (nm)*
Antimony	206.838*	206.831*	217.581
Antimony	206.839*	206.832*	n/a
Aluminum	308.22	308.20	308.215
Arsenic	189.04	189.04	193.606
Barium	493.41	493.41	493.409
Beryllium	313.04	313.04	313.042
Bismuth	223.06	n/a	n/a
Boron	249.68	249.68	249.678
Cadmium	226.50	226.50	228.802
Calcium	317.93	317.93	317.933
Chromium	267.72	267.72	267.716
Cobalt	228.62	228.62	228.616
Copper	324.75	324.75	324.754
Iron	271.44	271.44	259.940
Lead	220.354*	220.351*	220.353
Lead	220.355*	220.352*	n/a
Magnesium	279.08	279.08	279.079
Manganese	257.61	257.61	257.610
Molybdenum	202.03	n/a	202.030
Nickel	231.6	231.60	231.604
Potassium	766.49	766.49	766.491
Potassium	404.72**	n/a	n/a
Scandium	361.38	361.38	n/a
Selenium	196.027*	196.021*	196.026
Selenium	196.028*	196.022*	n/a
Silicon	288.16	n/a	288.158
Silver	328.07	328.07	328.068
Sodium	588.99	588.99	588.995
Sodium	330.23**	n/a	n/a
Strontium	421.55	421.55	n/a
Thallium	190.87	190.86	190.864
Tin	189.99	189.99	189.989
Titanium	334.94	334.94	334.941
Uranium	385.96	n/a	n/a
Vanadium	292.40	292.40	292.402
Yttrium	371.03	371.03	n/a
Zinc	213.86	213.86	213.856

* Dual channel required for these elements on trace ICP

** Used only a line to verify dilution factor needed

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TABLE 5

ICP (TJA 61) Calibration Standard Concentrations

ELEMENT	*STD 1 (ug/L)*	*STD 2 (ug/L)*	*STD 3 (ug/L)*
Antimony	4,000	2,000	1,000
Aluminum	4,000	2,000	1,000
Arsenic	4,000	2,000	1,000
Barium	4,000	2,000	1,000
Beryllium	4,000	2,000	1,000
Boron	4,000	2,000	1,000
Cadmium	4,000	2,000	1,000
Calcium	40,000	20,000	10,000
Chromium	4,000	2,000	1,000
Cobalt	4,000	2,000	1,000
Copper	4,000	2,000	1,000
Iron	4,000	2,000	1,000
Lead	4,000	2,000	1,000
Magnesium	40,000	20,000	10,000
Manganese	4,000	2,000	1,000
Molybdenum	4,000	2,000	1,000
Nickel	4,000	2,000	1,000
Potassium	40,000	20,000	10,000
Selenium	4,000	2,000	1,000
Silicon	4,000	2,000	1,000
Silver	4,000	2,000	1,000
Sodium	40,000	20,000	10,000
Thallium	4,000	2,000	1,000
Tin	4,000	2,000	1,000
Titanium	4,000	2,000	1,000
Vanadium	4,000	2,000	1,000
Zinc	4,000	2,000	1,000

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TABLE 6

ICP (Trace) Calibration Standard Concentrations

ELEMENT	*STD 1* *(ug/L)*	*STD 2* *(ug/L)*	*STD 3* *(ug/L)*
Antimony	1,000	500	250
Aluminum	10,000	5,000	2,500
Arsenic	1,000	500	250
Barium	1,000	500	250
Beryllium	1,000	500	250
Boron	1,000	500	250
Cadmium	1,000	500	250
Calcium	10,000	5,000	2,500
Chromium	1,000	500	250
Cobalt	1,000	500	250
Copper	1,000	500	250
Iron	10,000	5,000	2,500
Lead	1,000	500	250
Magnesium	10,000	5,000	2,500
Manganese	1,000	500	250
Molybdenum	1,000	500	250
Nickel	1,000	500	250
Potassium	10,000	5,000	2,500
Selenium	1,000	500	250
Scandium	1,000	500	250
Strontium	1,000	500	250
Silicon	1,000	500	250
Silver	1,000	500	250
Sodium	10,000	5,000	2,500
Thallium	1,000	500	250
Tin	1,000	500	250
Titanium	1,000	500	250
Uranium	1,000	500	250
Vanadium	1,000	500	250
Zinc	1,000	500	250

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TABLE 7

ICP ICSA / ICSAB Standard Concentrations

ELEMENT	*TJA61 ICSA (ug/L)*	*TJA61 ICSAB (ug/L)*	*TRACE ICSA (ug/L)*	*TRACE ICSAB (ug/L)*
Antimony	0	600	0	600
Aluminum	500,000	500,000	500,000	500,000
Arsenic	0	500	0	100
Barium	0	500	0	500
Beryllium	0	500	0	500
Bismuth	0	500	0	500
Boron	0	500	0	500
Cadmium	0	1000	0	1,000
Calcium	500,000	500,000	500,000	500,000
Chromium	0	500	0	500
Cobalt	0	500	0	500
Copper	0	500	0	500
Iron	200,000	200,000	200,000	200,000
Lead	0	1,000	0	50
Magnesium	500,000	500,000	500,000	500,000
Manganese	0	500	0	500
Molybdenum	0	500	0	500
Nickel	0	1,000	0	1,000
Potassium	0	0	0	0
Scandium	0	500	0	500
Selenium	0	500	0	50
Silicon	0	1,000	0	1,000
Silver	0	200	0	200
Sodium	0	0	0	0
Strontium	0	500	0	500
Thallium	0	500	0	100
Tin	0	1,000	0	1,000
Titanium	0	500	0	500
Uranium	n/a	n/a	0	500
Vanadium	0	500	0	500
Yttrium	0	500	0	500
Zinc	0	1,000	0	1,000

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TABLE 8

ICP CRI Values

ELEMENT	*TJA61 SW846 (ug/L)*	*TRACE SW846 (ug/L)*
Antimony	120	16
Aluminum	200	100
Arsenic	320	20
Barium	20	4
Beryllium	10	4
Bismuth	500	500
Boron	200	200
Cadmium	10	6
Calcium	1,000	500
Chromium	20	10
Cobalt	40	10
Copper	50	14
Iron	200	200
Lead	200	6
Magnesium	500	500
Manganese	20	6
Molybdenum	40	14
Nickel	70	8
Potassium	10,000	600
Scandium	N/A	2
Selenium	500	10
Silicon	900	600
Silver	20	10
Sodium	1,000	300
Strontium	N/A	2
Thallium	500	20
Tin	200	140
Titanium	20	8
Vanadium	40	12
Yttrium	50	50
Zinc	40	40

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TABLE 9

ICP Analytical Sequence

1.....SO
2.....S3
3.....S2
4.....S1
5.....ICV
6.....ICB
7.....ICSA
8.....ICSAB
9.....CRI
10.....CCV
11 .....CCB
12.....10 SAMPLES*
13 CCV
14.....CCB
15.....10 SAMPLES*
16.....CCV
17.....CCB
18.....ICSA
19.....ICSAB
20 .....CRI
21.....CCV
22 .....CCB

*NOTE: Samples include any samples or QC in a digestion Batch (i.e. PBS, LCS, LDS, MS, DUP, or MSD).

NOTE: You may repeat steps 13-18 until the 8 hour time limit (for the ICSA/AB and CRI) is up.

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Figure 1

Standard Batch Form for ICP Digestions

ICP(Dig.) Standard Batch Form, SWL-IN-205

ICP Digestion Standard Batch Form (205-FIG1.DOC) SWLO rev.# _____

*Standards Batch ID. #:*_____ *Date:*_____ *Analyst:*_____

Applicable SOPs: SWL-IN- _____ / SWL-IN-_____ / SWL-IN-_____ / SWL-IN-_____

The following is a list of reagent lot # used for this group of digestions, and will have the Standards Batch ID. # placed upon the digestion sheet:

Reagent	*Lot #*
Nitric Acid	_____
Hydrochloric Acid	_____
Hydrogen Peroxide	_____

NOTE: Refer to each specific SOP for reagents that are used in digestions of different matrices.

The following is a list of unique identification numbers for the assoicated standards:

Standard	*SWOK Identification #*
SS1	_____
SS2	_____
SS3LS	_____
SS4MS	_____
SS4	_____
SS5	_____
ICV-1	_____
ICV-2	_____
ICV-3	_____
ICV-4	_____
LCS (soil EPA)	_____
_____	_____
_____	_____

Southwest Laboratory of Oklahoma, Inc

Reviewed By:_____

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Figure 2

ICP Autosampler Table

AutoSampler Report Table: EPA Mon 01-26-98 09:44:45 AM page 1

Table Name: EPA
Sample Positions: 279/300 Autosampler Type: TJA 300
QC Positions: 11/19 # Sets: 1
Rinse Station location is rack -1, pos. -1.

--- Racks ---

Rack #	Type	Usage	#Pos Left	Analyses/Pos
1	Aux. (L) Rack	STD/QC/BLANK	11	4
2	Sample (13mm)	Samples	54	1
3	Sample (13mm)	Samples	75	1
4	Sample (13mm)	Samples	75	1
5	Sample (13mm)	Samples	75	1

--- Sample Sets ---

Set#	Type	Prepare?	Description	Method	#Pos	Rack#	StartPo
1	Normal	No		61ET2	21	2	1

--- Preparation Info ---

Set#	Uptake	Uptake#2	Final	Dil.Factor

No Samples Prepared.

Rack #1

Pos	Row	Col	Sample Name	Set #	#Used	Type
1	1	1	STD1	-NA-	1	Standard
2	1	2	STD0	-NA-	1	Standard
3	1	3	ICV	-NA-	1	QC Standard
4	1	4	ICB	-NA-	1	QC Standard
5	1	5	CCV	-NA-	3	QC Standard
6	1	6	CCB	-NA-	3	QC Standard
7	1	7	CCV	-NA-	1	QC Standard
8	1	8	CCB	-NA-	1	QC Standard
(9...19	Not Used)					

Rack #2

Pos	Row	Col	Sample Name	Set #	#Used	Type
1	1	1	CRI	1	-NA-	Sample
2	1	2	ICSA	1	-NA-	Sample
3	1	3	ICSAB	1	-NA-	Sample
4	1	4	PBW980123C	1	-NA-	Sample
5	1	5	LCW980123C	1	-NA-	Sample
6	1	6	LCW980123C(2)	1	-NA-	Sample
7	1	7	LDW980123C	1	-NA-	Sample
8	1	8	32452.01	1	-NA-	Sample
9	1	9	32452.02	1	-NA-	Sample
10	1	10	32452.03	1	-NA-	Sample
11	1	11	32452.04	1	-NA-	Sample
12	1	12	32452.05	1	-NA-	Sample
13	1	13	ZZZZZZ	1	-NA-	Sample

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Figure 3

ICP Run Log

Inorganics Department Southwest Laboratories of Oklahoma Run Log

File Name: T2012422.ASC Instrument: TJA ET2 Book: 6 Page: 176

Analyst : JA Date: 01/24/1998 Initials: JP Date: 1/25/98
Method : ILM04.0P Sop: SWL-IN-203 1.1
QC Ref : 277534 Standard Batch ID: SB4-3-26

ELEMENTS CIRCLED IN STD0 USED THROUGHOUT RUN UNLESS noted

Client ID	LAB ID	Time	DF	Analytes:
	STD0	22:11	1.00	Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Ni, K, Se, Ag, Na, Tl, V, Zn, Sn JP 1/26/98
	STD1	22:17	1.00	Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Ni, K, Se, Ag, Na, Tl, V, Zn, Sn
	ICV	22:22	1.00	Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Ni, K, Se, Ag, Na, Tl, V, Zn, Sn
	ICB	22:28	1.00	Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Ni, K, Se, Ag, Na, Tl, V, Zn, Sn
	CRI	22:34	1.00	Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Ni, K, Se, Ag, Na, Tl, V, Zn, Sn
	ICSA	22:39	1.00	Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Ni, K, Se, Ag, Na, Tl, V, Zn, Sn
	ICSAB	22:45	1.00	Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Ni, K, Se, Ag, Na, Tl, V, Zn, Sn
	CCV	22:51	1.00	Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Ni, K, Se, Ag, Na, Tl, V, Zn, Sn
	CCB	22:56	1.00	Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Ni, K, Se, Ag, Na, Tl, V, Zn, Sn
	PBS980121C	23:02	1.00	Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Ni, K, Se, Ag, Na, Tl, V, Zn, Sn
	LCS980121C	23:08	1.00	Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Ni, K, Se, Ag, Na, Tl, V, Zn, Sn
	LDS980121C	23:14	1.00	Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Ni, K, Se, Ag, Na, Tl, V, Zn, Sn
MSSB-22T	32460.01	23:19	1.00	Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Ni, K, Se, Ag, Na, Tl, V, Zn, Sn

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Figure 4

ICP Re-Run Log

Inorganics Department Southwest Laboratories of Oklahoma Re-Run Log

File Name: T2012422.ASC Instrument: TJA ET2 Book: Page: 1

Analyst : JA Date: 01/24/1998 Initials: JP Date: 1/25/98
Method : ILM04.0P Sop: SWL-IN-203 1.1
QC Ref : 277534 Standard Batch ID: SB4-3-26
The following elements are not verified:

Client ID	LAB ID	Time	DF	Analytes:
	STD0	22:11	1.00	
	STD1	22:17	1.00	
	ICV	22:22	1.00	
	ICB	22:28	1.00	
	CRI	22:34	1.00	
	ICSA	22:39	1.00	
	ICSAB	22:45	1.00	
	CCV	22:51	1.00	
	CCB	22:56	1.00	
	PBS980121C	23:02	1.00	
	LCS980121C	23:08	1.00	
	LDS980121C	23:14	1.00	
MSSB-22T	32460.01	23:19	1.00	
MSSB-22B	32460.02	23:25	1.00	
MSSB-23T	32460.03	23:31	1.00	
MSSB-23B	32460.04	23:36	1.00	
MSSB-24T	32460.05	23:42	1.00	
MSSA-14	32460.06	23:48	1.00	
	ZZZZZZ	23:53	1.00	

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Figure 5

ICP Standard Batch Form

ICP(Analysis) Standard Batch Form, SWL-IN-205

ICP Digestion Standard Batch Form (205-FIG2.DOC) SWLO rev.# _____

*Standards Batch ID. #:*_____ *Date:*_____ *Analyst:*_____

Applicable SOPs: SWL-IN- _____ / SWL-IN-_____ / SWL-IN-_____ / SWL-IN-____

The following is a list of current standards used for analysis on the ICP:

Standard List for (circle one): TJA ET1 / TJA ET2 / TJA 61E

Standard Name	*SWLO ID#*	*Date Prepped*	*Expiration Date*
S, STD-1	MP-7-48	11/21/97	1/21/98
STD-2 (CCV)	MP-7-49	11/21/97	1/21/98
ICV (SWLO)	MP-7-19	11/03/97	1/03/98
ICSA (SWLO)	MP-7-31	11/11/97	01/11/98
ICSAB (SWLO)	MP-7-22	11/03/97	01/03/98
ICV (EPA only)	MP-7-41	11/18/97	01/18/98
ICSA (EPA only)	MP-7-58	12/01/97	02/01/98
ICSAB (EPA only)	MP-7-59	12/01/97	02/01/98
CRI (ILM04.0 protocol)	MP-7-15	10/25/97	12/25/97
CRI (SW846 protocol)	MP-7-25	11/05/97	01/05/98
CRI (EPA 200.7)			

Reviewed By:_____

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NOTE:

PAGES C2-83 to C2-86

ARE NOT INCLUDED

SOUTHWEST LABORATORY OF OKLAHOMA, INC.

Standard Operating Procedure

Mercury SW846 7470A/7471A Digestion and Analysis

Document No.: SWL-IN-207

Rev No. / Date: Rev. 2.0 — 06/06/97

APPROVALS	DATES
Procedure Prepared By	06/06/97 Date
Inorganic Program Manager	06/06/97 Date
Del Alston for Mike Butler Laboratory Safety Officer	6/6/97 Date
Chuck Hoover Laboratory QA/QC Officer.	6/6/97 Date

(Effective Date is 5 calendar days after the last signature above - QA/QC Officer)

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Mercury SW846 7470A/7471A Digestion and Analysis

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Attachment 1 MERCURY STANDARD BATCH FORM 11

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1.0 - SCOPE AND APPLICATION

1.1 This SOP will give the SW846 methods 7470A/7471A digestion and analysis procedures for water and soil matrices. These procedures will be used for the preparation and analysis for mercury.

1.2 The digestion supervisor will designate the proper method to be used per batch of samples. If the method is not clearly identified on the internal custody forms, check with the Project Officer responsible for the samples before digestion is started.

2.0 - METHOD SUMMARY

2.1 Prior to analysis, the solid, semi-solid, or water samples must be prepared according to the procedures discussed in this method.

2.2 This method is a cold-vapor atomic absorption method, based on the absorption of radiation at the 254 nm wavelength by mercury vapor. The mercury is reduced to the elemental state 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 (peak height) is measured as a function of mercury concentration.

3.0 - HEALTH AND SAFETY

3.1 Refer to the current version of SOP SWL-GENSAFT.

3.2 Mercury compounds are highly toxic if swallowed, inhaled, or absorbed through the skin. Analysis should be conducted in an exhaust hood. The analyst should use chemical resistant gloves when handling any mercury standards or potential samples containing mercury. During and after the procedure there should be a intensive cleanup to prevent human contact and cross contamination.

4.0 - SAMPLE PRESERVATION, CONTAINERS, HANDLING & STORAGE

4.1 Water samples must be preserved to a pH <2. If not, notify the PO and Inorganic Supervisor, acidify sample with HNO_3 and proceed with analysis. Comment action on digestion sheet, fill out a "Non-Conformance Report" (refer to SOP SWL-GA-105").

4.2 The sample must be analyzed within 28 days of collection.

4.3 Care must be taken in glassware preparation, digestion, and analysis to ensure minimal cross contamination.

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5.0 - INTERFERENCES AND POTENTIAL PROBLEMS

5.1 Potassium permanganate is added to eliminate possible interference from sulfide. Concentrations as high as 20 mg/kg or 10 mg/l, as sodium sulfate, do not interfere with the recovery of added inorganic mercury in reagent water.

5.2 Copper has also been reported to interfere; however, copper concentrations as high as 10 mg/kg or 10 mg/l had no effect on recovery of mercury from spiked samples.

5.3 Samples high in chlorides require additional permanganate (as much 25 ml) because, during the oxidation step chlorides are converted to free chlorine, which also absorbs radiation of 253 nm. Therefore, care must be taken to ensure that free chlorine is absent before the mercury is reduced and swept into the cell. This may be accomplished by using an excess of hydroxylamine sulfate reagent (25 ml). In addition, the dead air space in the glass container must be purged before adding stannous sulfate.

5.4 Certain volatile organic materials that absorb at this wavelength may also cause interference. A preliminary run without reagents should determine if this type of interference is present.

5.5 Low level mercury sample preparation, digestion, and analysis may be subject to environmental contamination if performed in areas with high ambient backgrounds where mercury was previously employed as an analytical reagent in analyses such as total Kjeldahl nitrogen (TKN) or chemical oxygen demand (COD). Care should be taken to avoid performing digestion or analysis in these areas.

6.0 - EQUIPMENT/APPARATUS

6.1 8 oz glass jars with septa lids

6.2 Stainless steel scoops for soils

6.3 Class A volumetric flasks (100 ml)

6.4 Volumetric Flasks (2000 ml)

6.5 Eppendorfs (appropriate volume ranges) with tips

6.6 Eppendorf repeater pipette dispenser with tips

6.7 Leeman PS200 Mercury analyzer with computer and autosampler

6.8 Tubing for autosampler pump

6.9 Water Bath capable of 95°C

6.10 Balance (top loader) capable of weighing 0.2 g

6.11 Laboratory exhaust hood (for preparation)

6.12 Exhaust Hood for Instrument

6.13 Argon High Purity Gas

7.0 - REAGENTS

7.1 Acids used in the preparation of standards and for sample processing must conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society (ACS) and be of the Mallinckrodt ArSelect grade or the equivalent.

7.2 Reagents

7.2.1 Reagent water: ASTM Type II (Millipore) water. All references to water in this method refer to reagent water unless otherwise specified.

7.2.2 Sulfuric acid, conc.: trace metals analysis grade.

7.2.3 Sulfuric acid, 0.5 N: Dilute 14.0 ml of conc. sulfuric acid to 1.0 liter.

7.2.4 Nitric acid, conc.: trace metals analysis grade.

7.2.5 Hydrochloric acid, conc.: trace metals analysis grade.

7.2.6 Stannous chloride: Add 25 grams stannous chloride to 250 ml 25% HCl. Filter solution through a Grade 4, 20-25 um retention filter paper.

7.2.7 Sodium chloride - hydroxylamine hydrochloride solution: Dissolve 12 grams of NaCl and 12 grams hydroxylamine hydrochloride in reagent water and dilute to 100 ml. Volume of reagent may be increase as long as the grams of reagent is also increased by the same factor (ex. 2000 ml will take 240 grams of each reagent).

7.2.8 Potassium permanganate, 5% solution, w/v: Dissolve 5 grams of potassium permanganate in 100 ml of reagent water. Volume of reagent may be increase as long as the grams of reagent is also increased by the same factor (ex. 2000 ml will take 100 grams of reagent).

7.2.9 Potassium persulfate, 5% solution, w/v: Dissolve 5 grams of potassium persulfate in 100 ml of reagent water. Volume of reagent may be increase as long as the grams of reagent is also increased by the same factor (ex. 2000 ml will take 100 grams of reagent).

7.3 Standard Preparation

7.3.1 Stock solutions for mercury are prepared solutions from NIST approved standards containing 1,000 mg/l mercury.

7.3.2 An intermediate stock solution of 10.0 mg/l mercury is prepared by diluting 1.0 ml of stock solution and 1.0 ml of conc. HNO_3 to 100 ml with ASTM Type II water. This standard is prepared weekly.

7.3.3 A second intermediate solution of 0.1 mg/l mercury is prepared each day of analysis by diluting 1.0 ml of the 10.0 mg/l intermediate stock solution and 1.0 ml of conc. HNO3 to 100 ml with ASTM Type II water.

7.3.4 Calibration standards (prepared daily) are prepared by diluting the 0.1 mg/l intermediate standard to 100 ml with ASTM Type II water according to the following schedule:

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Standard Calibration ug/l	*ml of 0.1 mg/l Intermediate Standard*
0.0	0.0
0.2	0.2
0.5	0.5
1.0	1.0
5.0	5.0
10.0	10.0

7.3.5 Initial Calibration Verification (ICV): This standard is made from a standard obtained from a NIST certified standard that has a different source than the calibration standards. The true value of this standard must be within the calibration range of the system.

7.3.6 CRA standard: Use the 0.2 ug/l calibration standard.

7.3.7 Continuing Calibration Verification (CCV): Use the 5.0 ug/l calibration standard.

7.3.8 Initial and Continuing Calibration Blanks (ICB and CCB): Use 0.0 ug/l calibration standard.

8.0 - PROCEDURE

8.1 Each day of sample preparation, the standards in Section 7.3.4-8 will be brought through the Aqueous digestion procedure.

8.2 Aqueous Digestion Procedure

8.2.1 Mix the sample, by shaking the sample bottle vigorously, to assure the sample is homogenized and a representative sample will be easily taken. Transfer 100 ml of well mixed sample to a 250 ml glass bottle.

8.2.2 Add 5 ml of conc. sulfuric acid and 2.5 ml of conc. nitric acid to each bottle.

8.2.3 Add 15 ml of $KMnO_4$ solution to each bottle and allow to stand at least 15 minutes.

8.2.4 Add 8 ml of potassium persulfate to each bottle.

8.2.5 Cap with Septa Lid.

8.2.6 Heat for 2 hours in a water bath at 90-95°C.

8.2.7 Cool and add 6 ml of sodium chloride-hydroxylamine sulfate solution to reduce the excess permanganate.

8.3 Soil Digestion Procedure

8.3.1 Homogenize sample by mixing sample with metal scoop. Fully mix the sample within the container before taking a representative sample out for analysis.

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8.3.2 Weigh a representative 0.6 g portion (three 0.2 g of sample in different areas) of wet sample in a 250 ml glass bottle.

8.3.3 Add 5.0 ml of ASTM type II water.

8.3.4 Add 5 ml of concentrated sulfuric acid and 2.5 ml of concentrated nitric acid, mixing after dilution.

8.3.5 Heat 2 minutes in a water bath at 95°C.

8.3.6 Cool and add 45 ml of ASTM type II water.

8.3.7 Add 15 ml $KMnO_4$ and allow to stand for 15 minutes.

8.3.8 Add 8 Ml of potassium persulfate to each bottle.

8.3.9 Mix thoroughly and heat at 95°C for 30 minutes in the water bath.

8.3.10 Cool and add 6 ml of sodium chloride-hydroxylamine sulfate solution to reduce the excess permanganate.

8.3.11 Add 50 ml of ASTM type II water.

8.4 Instrument Parameters

8.4.1 Wavelength: 254.0 nm

8.4.2 Approximate Rinse Time:≈50 seconds (instrument varies rinse time according to mercury level in sample).

8.4.3 Pump Speed: ≈5mL/min.

8.4.4 Readout mode: Absorbance

8.4.5 Integration time: ≈10.0 seconds

8.4.6 Gas Flow: ≈0.3L/min.

8.5 Instrument Calibration

8.5.1 Set up instrument according to Section 8.4 and manufacturers recommendations.

8.5.2 Make sure the stannous chloride line is correctly attached to the Pump Assembly.

8.5.3 Fill water bath with 5% H_2SO_4 and 2.5% HNO_3.

8.5.4 Rinse system using macro "warmstart" for a minimum of 40 minutes before starting calibration.

8.5.5 Check aperature reading. If $> \pm$ 100, adjust, wait 10 minutes, and recheck.

8.5.6 Place fresh drying tube containing Magnesium Perchlorate into system.

8.5.7 Place calibration standards into the correct autosampler tubes.

8.5.8 Press F2 macro. Type STANDARD, then press enter key.

8.5.9 Tabulate the calibration standard concentration versus absorbance response for each calibration standard, which is performed by instrument.

8.5.10 Analyze data by linear regression. The correlation coefficient must be greater than 0.995 before proceeding with analysis.

8.5.11 If data is acceptable, press "A" to accept calibration curve.

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8.6 Instrument Analysis

8.6.1 Set up instrument parameters as discussed in Section 8.4.

8.6.2 Calibrate instrument as discussed in Section 8.5.

8.6.3 Add samples to the autosampler (refer to instrument manual), double checking sample & position, as stated in the autosampler table (see Appendix 1.1). Press F8 sample to start analysis run.

8.6.4 Instrument must be put into "overnite" mode during the week and "weekend" mode on Fridays. Refer to instrument manual.

8.6.5 Soak water bath in 1:1 HNO_3 overnight.

8.7 Write-up & Review Procedure

8.7.1 Record digestions according to the current version of SOP "Digestion / Distillation Log", SWL-IN-105.

8.7.2 Record calibration standards, run QC standards, and reagent lot #s on the Mercury Standard Batch Form (attachment 1). Calibration standards, run QC standards, and reagent lot #s associated with a preparation date must be analyzed with all of the digestion batches made that day. Reagents must be from the same lot # for all QC samples and client samples.

8.7.1 When run is complete, copy electronic version of analytical run to the Inorganic Database.

8.7.2 Follow the procedure as explained in the current version of SOP "Inorganic Database", SWL-IN-108, to process the run and print out the runlog.

8.7.3 Review Data according requirements in Section 10.0 of this SOP, and the current version of SOP "1st and 2nd Review of ICP, GFAA, Mercury, and Cyanide", SWL-IN-106.

8.7.5 If needed, fill out a "Non-Comformance Report" (see SOP SWL-IN-106).

8.7.6 If needed, fill out a Re-analysis Form (see SOP SWL-IN-106).

8.7.7 The Digestion Sheet(s) is then attached to the raw data, and above forms (if needed) are presented for a secondary or peer review.

8.7.8 The run has been accepted the data will be automatically uploaded into LIMS by the Inorganic Database, and QC sheets (if necessary) will be filled out.

9.0 - CALCULATIONS

9.1 Instrument reports all results as ug/l.

9.2 Report all soils as mg/kg (wet weight) by the following:

$(C \times V) / W =$ mg/kg of Hg

Where:

C = concentration , ug/l

V = final volume in liters after sample preparation

W = weight in grams of wet sample in preparation

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9.3 Report mercury concentrations as follows:

9.3.1 Below 10 ug/l = two significant figures

9.3.2 Above 10 ug/l = three significant figures

10.0 - QUALITY ASSURANCE / QUALITY CONTROL

10.1 Initial Demonstration of Performance.

10.1.1 Linear Calibration Range(LCR) - The LCR must be determined initially and verified every six months or whenever, in the judgement of the analyst, a change in analytical performance caused by either a change in instrument hardware of operating conditions would dictate they be redetermined. The verification on linearity must use a minimum of a blank and three standards. If any verification data exceeds the initial values by +/- 10%, linearity must be re-established. If any portion of the range is shown to be nonlinear, sufficient standards must be used to clearly define the nonlinear portion.

10.1.2 Method Detection Limit (MDL) -- MDL's must be determined, according to the current version of SOP "Defination and Determination of Detection Limits", SWL-GA-113, on a yearly basis.

10.2 Digestion QA/QC Requirements:

10.2.1 The following QA/QC samples will be designated as XXXYYMMDDHGZ. XXX designates the type of sample (PBS, LCS, or LDS). YY is the last two digits in the current year, MMDD is the month and day, HG designates a mercury digestion. Z is the batch letter done that day (if located in batch YYMMDDHG1-Z = "A; if located in batch YYMMDDHG4-Z="D").

10.2.1.1 Preparation Blanks (PBW or PBS) - prepare by using the following volumes of ASTM type water:

PBW = 100ml

PBS = 10ml

Result must be between the current -PQL and PQL (refer to the SWL Redbook).

10.2.1.2 Laboratory Control Sample (LCW, LCS) is prepared from the following:

LCW samples: spike 99 ml of ASTM Type II water with 1.0 ml of 0.1 mg/l mercury standard (see Section 7.3.3)

LCS samples: add 1.0 ml of 0.1 mg/l mercury standard and proceed with digestion procedure.

10.2.1.3 Laboratory Control Sample Duplicate (LDW, LDS) is prepared from the following:

LDW samples: Same as the LCW.

LDS samples: Same as the LCS.

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NOTE Control limits for the Laboratory Control Samples are ± 20% recovery and <20 % RPD, unless stated otherwise by the Inorganic Manager.*

10.2.1.4 Matrix Spike & Matrix Spike Duplicates:, and has a control limt of 75-125 % recovery. Spike the water or soil sample with 1.0 ml of 0.1 mg/l mercury standard (see Sec 7.3.3). If sample fails the control limt of ± 25% recovery of true value, flagged all assoicated data with a "N" . The MSD also has a control limit of < 20 % RPD. If the control limit is not met, flagged all assoicated data with a "*".

10.2.1.5 Matrix Duplicate: This sample is made by digesting a second aliquot of the same sample to determine the precision of the method, and has a control limit of < 20 % RPD. If the control limit is not met, flagged all assoicated data with a "*".

10.2.1.6 Frequency Requirements - A PBS, LCS, and LDS are required for every batch of samples (maximum number of samples in a batch is 20 samples). The matrix duplicate, spike, and spike duplicate will be done on request per client basis.

10.2.1.7 Digestion QC failures: If the Preparation Blank or Laboration Control Sample do not meet the required control limits, the samples must be redigested.

10.3 Calibration Checks

10.3.1 Initial Calibration Verification (ICV) - After the system has been calibrated, analysis of the ICV is required. The measurement must be within ± 10% (± 5% for EPA 245.1 and 245.5) of the true value or the analysis must be terminated, the problem corrected, the instrument recalibrated and the calibration reverified.

10.3.2 Initial Calibration Blank (ICB) - Immediately after the analysis of the ICV the ICB must be analyzed. If the absolute result is greater than the current PQL, terminate analysis, correct the problem and recalibrate.

10.3.3 CRA Standard - Must be run after the ICB. The acceptable deviation of the CRDL is ± 50 of the true value.

10.3.4 Continuing Calibration Verification (CCV) - Will be analyzed at the beginning, a frequency of 10% during the run, and after the last analytical sample. The mercury concentrations will be at the mid-range level of the calibration curve. If the deviation of the continuing calibration verification is not within ± 20% (± 10% for EPA 245.1 and 245.5) of the true value, the instrument will be recalibrated and the proceeding samples since the last calibration verification will be reanalyzed.

10.3.5 Continuing Calibration Blank (CCB) - A calibration blank is analyzed immediately after every CCV. Blanks are to be reported down to the instrument detection limit. If the blank result is greater than the current PQL, terminate analysis, correct the problem and recalibrate.

11.0 - WASTE DISPOSAL

11.1 Refer to the current version of SOP "Hazardous Waste", SWL-GA-114.

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12.0 - METHOD PERFORMANCE

12.1 Refer to Section 13 in Method 245.1 of Methods for Chemical Analysis of Water and Wastes.

12.2 Refer to Section 10 in Method 245.5 of Methods for Chemical Analysis of Water and Wastes.

13.0 - METHOD REFERENCES

13.1 SW846 Methods 7000A (rev 1.0 - July 1992), 7470A (rev 1 - September 1994), and 7471A (rev 1 - September 1994) - modified.

13.2 Leeman PS200 instrument manual.

13.3 "Corrective Action" SOP, SWL-GA-105

13.4 "Laboratory Safety Plan" SOP, SWL-GENSAFT

13.5 "Digestion / Distillation Log" SOP, SWL-IN-105.

13.6 "Inorganic Database" SOP, SWL-IN108.

13.7 "1st and 2nd Reveiw of ICP, GFAA, Mercury, and Cyanide" SOP, SWL-IN-106.

14.0 - DEFINATIONS

14.1 Initial Demonstration of Performance - Items that must be performed for analysis of method.

14.2 Linear Calibration Range (LCR) - The linear range in which the method is valid.

14.3 Method Detection Limit (MDL) -- MDL's as defined in the current version of SOP SWL-GENMDLS.

14.4 Digestion QC Requirements - QC samples required for each digestion batch.

14.5 Preparation Blanks (PBW or PBS) - Method blanks to ensure contamination of analyte is not present in distillation procedure.

14.6 Laboratory Control Sample (LCW, LCS) - A laboratory spike on a clean matrix to ensure distillation procedure is in control through % recovery.

14.7 Laboratory Control Sample Duplicate (LDW, LDS) - A laboratory spike on a clean matrix to ensure digestion procedure is in control through % recovery and % RPD in relation to LCW or LCS.

14.8 Matrix Spike & Matrix Spike Duplicates (MS / MSD) - This is made from digesting the sample spiked with an known amount and then comparing it with the original sample. This is designated to provide information about the effect of the sample matrix on the digestion and/or measurement methodolgy.

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14.9 Matrix Duplicate: This sample is made by digesting a second aliquot of the same sample to determine the precision of the method.

14.10 Calibration Checks - QC samples performed during analysis to assure instrument is responding correctly.

14.11 Initial Calibration Verification (ICV)- A standard, that is made from an independent source in relation to the calibration standards. Analyzed to assure calibration accuracy of the system.

14.12 Initial Calibration Blank (ICB) - A instrument blank sample to assure no contamination in system or no memory effects.

14.13 CRA Standard - A low range standard carried through the digestion procedure, to verify the ability to detect results at the reporting limit.

14.14 Continuing Calibration Verification (CCV) - Mid-range standard analyzed to assure calibration accuracy during each analysis run.

14.15 Continuing Calibration Blank (CCB)- A instrument blank sample to assure not contamination in system or no memory effects.

14.16 Red Book - A Complilation of current MDLs/PQLs for SWL.

15.0 - ATTACHMENTS

15.1 Attachment 1 - Mercury Standard Batch Form

16.0 - UNIQUE METHOD INFORMATION

16.1 Soil digestion method has been modified from the use of 5ml of aqua regia to 5 ml of sulfuric acid and 2.5 mls of nitric acid. This was done to eliminate a possible source of false positves due to chlorine and the negative effects aqua regia has on our instrumentation.

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ATTACHMENT 6

Mercury Standard Batch Form

Mercury Standard Batch Form, SWL-IN-207

Sample Re-Analysis Form (207-ATT1.DOC) SWLO rev.# _____

Page _____

*Standards Batch ID. #:*_____

Applicable SOPs: SWL-IN- _____ / SWL-IN-_____ / SWL-IN-_____ / SWL-IN-_____
(above is only valid if the digestion procedure is the same in each of the stated SOPs)

Date:_____ Analyst:_____

The following samples were taken through the aqueous digestion procedure:

Name of Standard	*mls of 0.1 mg/l Std.*	*Final Volume (mls)*	*Final Conc. (ug/l)*	*Analyst Initials*
SO/ICB/ICB	0.0	100	0.0	_____
S0.2/CRA	0.2	100	0.2	_____
S0.5	0.5	100	0.5	_____
S1	1.0	100	1.0	_____
S5/CCV	5.0	100	5.0	_____
S10	10.0	100	10.0	_____

The following 0.1 mg/l standard was used in the preparation of the above standards:
0.1 mg/l standard ID: _____ Expiration: Daily Date Prepared: _____

Standard	*Parent*	*Parent Ex. Date*	*Parent Conc. (mg/l)*	*Parent Volume (ml)*	*Final Volume (ml)*	*Final Conc. (ug/l)*	*Analyst Initials*
_____	_____	_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____	_____	_____

All Digestion Batches and above standards were analyzed and digested together. The following is a list of reagent lot # used for this group of digestions and analysis:

Reagent	*Lot #*	*Reagent*	*Lot #*
Sulfuric Acid	_____	Sodium Chloride	_____
Nitric Acid	_____	Hydroxylamine hydrochloride	_____
Hydrochloric Acid	_____	Potassium permanganate	_____
Stannous Chloride	_____	Potassium Persulfate	_____

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SOUTHWEST LABORATORY OF OKLAHOMA, INC.

TM

Standard Operating Procedure

Toxicity Characteristic Leaching Procedure Non-Volatile Extractions

Document No.: SWL-IN-700

Rev No. / Date: Rev. 3.1 — 12/02/97

APPROVALS	DATES
Chuck Hoover	12-02-97
Procedure Prepared By	Date
[signature]	12/02/97
Laboratory Manager or Director	Date
Mike Kuttle	12/2/97
Laboratory Safety Officer	Date
Chuck Hoover	12-02-97
Laboratory QA/QC Officer	Date

(Effective Date for is 5 calendar days after the last signature above - QA/QC Officer)

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[✓] Official Copy OC No.: 2028 Issued to: Alan Fosdick Date: 2/4/98 Initials: MM

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TOXICITY CHARACTERISTIC LEACHING PROCEDURE NON-VOLATILE EXTRACTIONS

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1.0 SCOPE AND APPLICATION

1.1 The purpose of this SOP, Toxicity Characteristic Leaching Procedure (TCLP) is to determine the mobility of both organic and inorganic analytes in liquid, solid, and multiphasic wastes.

1.2 If a total analysis of the waste demonstrates that individual analytes are not present in the waste, or that they are present but at such low concentrations that the appropriate regulatory levels could not be exceeded, the TCLP need not be run.

1.3 If an analysis of any one of the liquid fractions of the TCLP extract indicates that a regulated compound is present at such high concentrations that, even after accounting for dilution from the other fractions of the extract, the concentration would be above the regulatory level for that compound, then the waste is hazardous and it is not necessary to analyze the remaining fractions of the extract.

2.0 METHOD SUMMARY

2.1 For liquid wastes (i.e., those containing less than 0.5% dry solid material), the waste, after filtration through a 0.6 to 0.8 um glass fiber filter, is defined as the TCLP extract.

2.2 For wastes containing greater than or equal to 0.5% solids, the liquid, if any, is separated from the solid phase and stored for later analysis. The solid phase is extracted with an amount of extraction fluid equal to 20 times the weight of the solid phase. Following extraction, the liquid phase is separated from the solid phase by filtration through a 0.6 to 0.8 um glass fiber filter.

2.3 If compatible, the initial liquid of the waste is added to the liquid extract, and these are analyzed together. If incompatible, the liquids are analyzed separately and the results are mathematically combined to yield a volume-weighted average concentration.

3.0 HEALTH AND SAFETY — Refer to the Lab Safety Plan (SWL-GA-111) for precautions.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING & STORAGE — N/A

5.0 **INTERFERENCES.** Potential interferences that may be encountered during analysis are discussed in the individual analytical SOP's.

6.0 APPARATUS

6.1 Agitation apparatus: Associated Design and MFG. Co., Model No. 3740-24-BRE-TM. Capable of maintaining 30 +/-2 rpm.

6.2 Bottle Extraction Vessel. Two liter borosilicate glass jar.

6.3 Filtration Device.

6.4 Filters. Glass fiber filters with a pore size of 0.6 to 0.8 um. When analyzing for metals, the filters are rinsed with 1N nitric acid, followed by three consecutive rinses with deionized water.

6.5 pH meter. Accumet Model 915.

6.6 Top Loading Balance. Mettler PM2000, or equivalent — All measurements shall be ± 0.1g of the desired weight. All balances used shall be able to read to 0.01g.

6.7 Beakers. 250 ml glass.

6.8 Watchglass.

6.9 Magnetic stirrer.

7.0 REAGENTS

7.1 Reagent Water. Millipore – ASTM Type II (by conductivity).

7.2 Hydrochloric acid (1N). Add 83 ml conc. HCl to 500 ml distilled water, dilute to 1 L.

7.3 Nitric acid (1N). Add 64 ml conc. HNO_3 to 500 ml distilled water, dilute to 1 L.

7.4 Sodium hydroxide (1N). Dissolve 40 g of NaOH in 500 ml distilled water, dilute to 1 L.

7.5 Glacial acetic acid, conc. , ACS reagent grade.

7.6 Extraction fluid #1. Add 5.7 ml glacial acetic acid(7.5) to 500 ml reagent water(7.1), add 64.3 ml of 1N NaOH(7.4), and dilute to 1 liter. When correctly prepared, the pH of this solution will be 4.93+/- 0.05. If pH is outside these limits the fluid will be discarded and fresh extraction fluid prepared.

7.7 Extraction fluid #2. Dilute 5.7 ml glacial acetic acid(7.5) with reagent water(7.1) to a volume of 1 L. When correctly prepared, the pH of this fluid will be 2.88 +/- 0.05. If pH is outside limits the fluid will be discarded and fresh fluid extraction fluid prepared.

8.0 PROCEDURE

8.1 Preliminary determination of percent solids:

8.1.1 If the waste will obviously yield no liquid when subjected to pressure filtration proceed to Section 8.2.

8.1.2 Pre-weigh the filter and the container that will receive the filter. Record weight on TCLP Percent Moisture Log (Attachment 1).

8.1.3 Assemble the filter holder and filter. Place the filter on the support screen and secure.

8.1.4 Weigh out a subsample of the waste (100 gram minimum) and record the weight.

8.1.5 Allow slurries to stand to permit the solid phase to settle. Transfer the waste sample to the filter holder and gradually apply vacuum of 1-10 psi, until air moves through the filter. Slowly increase the vacuum in 10 psi increments to 50 psi. When liquid flow ceases stop the filtration.

8.1.6 The material in the filter holder is defined as the solid phase and the filtrate is defined as the liquid phase.

8.1.7 Some wastes such as oily wastes and paints will not filter and is defined as a solid. Use only one filter for this procedure and do not replace.

8.1.8 Determine the weight of the liquid phase by subtracting the weight of the filtrate container from the total weight of the filtrate-filled container. Determine the weight of the solid phase by subtracting the weight of the liquid phase from the weight of the total waste sample.

8.1.9 Determine the percent solids as follows:

$$\text{Percent solids} = \frac{\text{weight of solid}}{\text{total weight of waste}} \times 100$$

8.1.10 If the percent solid is equal to or greater than 0.5%, then proceed to Section 8.2 to determine if particle size reduction is necessary or to Section 8.1.11 if it's noticed that water is entrapped in the solid material. If the percent solid is less than 0.5%, then the filtrate will be the TCLP extract.

8.1.11 Remove the solid phase and filter from the filtration apparatus.

8.1.12 Dry the filter and solid phase at 100 +/- 20^0 C until the two successive weighings yield the same value within 1%. Record the final weight.

8.1.13 Calculate the percent solids as follows:

$$\text{Percent solids} = \frac{\text{(Wt. of dry waste + filter) - tared wt. of filter}}{\text{Initial weight of waste}} \times 100$$

8.1.14 If the percent solid is less than 0.5 % then the filtrate from Section 8.1.5 will be the TCLP extract. If the percent dry solid is equal to or greater than 0.5%,

return to Section 8.1.1 with a fresh portion of waste and determine if particle size reduction is necessary.

8.2 Determination if particle size reduction is required.

8.2.1 Particle size reduction is not required if the waste will pass through a 9.5 mm (0.375 in.) sieve. Proceed to Section 8.3.1.

8.2.2 If the waste will not pass through a 9.5 mm sieve then prepare the waste for extraction by cutting, crushing or grinding to reduce the particle size. When complete proceed to Section 8.3.1.

8.3 Determination of appropriate extraction fluid. If the solid content of the waste is greater than or equal to 0.5% determine the appropriate fluid for extraction as follows:

8.3.1 Weigh out a small subsample of the solid phase, reduce the solid, if necessary, to a particle size of 1mm in diameter or less. Transfer 5.0 g of the waste to a 250 ml beaker.

8.3.2 Add 96.5 ml of reagent water to the beaker, cover with a watchglass, and stir vigorously for 5 minutes on a magnetic stirrer. Measure and record the pH on the TCLP pH Run Log (Attachment 1). If the pH is <5.0 use extraction fluid #1 and proceed to section 8.4.1.

8.3.3 If the pH was greater than 5.0, add 3.5 ml 1N HCl, cover with a watch glass, heat to 50^0 C, and hold at 50^0 C for 10 minutes.

8.3.4 Let the solution cool to room temperature and record the pH. If the pH is <5.0, use extraction fluid #1. If the pH is >5.0, use extraction fluid #2.

8.4 Extraction procedure

8.4.1 Enough solids should be generated for extraction such that the volume of TCLP extract will be sufficient to support all of the analyses required. A good rule of thumb is 1-L per analyses.

8.4.2 Determine the amount of the raw sample needed to provide combined filtrate and extract totaling 2000mL. The following formula should be used to determine this amount:

$$(1-S/100)\,(RS) + 20(S/100)\,(RS) = 2000$$

where: S = % solids
RS = raw sample wieght

e.g. if the raw sample were 20% solids:

$$(1-20/100)\,(RS) + 20(20/100)\,(RS) = 2000$$
$$= 0.8RS + 4RS = 2000$$
$$= RS = 417 \text{ grams} \quad \pm .1 \text{ g}$$

8.4.3 Determine the amount of the extraction fluid to add to the extractor vessel a follows:

$$\text{Amount of extraction fluid} = \frac{20 \times \text{percent solids (8.1)} \times \text{weight of waste filtered}}{100}$$

8.4.4 Add the solids and appropriate amount of extraction fluid to the extractor vessel. Record information on TCLP non-volatile extraction log (Attachment 1).

8.4.5 Close the extractor bottle tightly, secure in the rotary agitation device, and rotate at 30 +/- 2 rpm for 18 +/- 2 hours. Ambient temperature shall be maintained at 23 +/- 2^{0}C during the extraction. Record both the time start and temperature at start on extraction log.

8.4.6 Following the 18 hour extraction, separate the material in the extractor vessel into its component liquid and solid phase by filtering through a new glass fiber filter as outlined in Section 8.1. For final filtration of the TCLP extract, the glass fiber filter may be changed, if necessary, to facilitate filtration. Filters shall be acid-washed (6.4) if evaluating the mobility of metals.

8.5 Prepare the TCLP extract as follows:

8.5.1 If the waste contained no initial liquid phase, the filtered liquid material is the TCLP extract.

8.5.2 If compatible, combine the filtered liquid obtained in Step 8.4 with the initial liquid phase of the waste obtained in Section 8.1. This combined liquid is defined as the TCLP extract.

8.5.3 If the initial liquid phase of the waste, obtained in Section 8.1, is not compatible with the filtered liquid resulting from Section 8.4, do not combine these liquids. Analyze these liquids, collectively defined as the TCLP extract, and combine the results mathematically. Determine the volume of the individual phases and combine the results by using the volume-weighted average:

$$\text{Final Analyte Concentration} = \frac{(V_1)(C_1) + (V_2)(C_2)}{V_1 + V_2}$$

where:
V_1 = The volume of the first phase (liters).
C_1 = The concentration of the analyte of concern in the first phase (mg/l).
V_2 = The volume of the second phase (liters).
C_2 = The concentration of the analyte of concern in the second phase (mg/l).

8.5.4 Following collection of the TCLP extract, the pH of the extract should be recorded.

8.5.5 Immediately aliquot and preserve the extract for analysis. Metals aliquots must be acidified with nitric acid to pH <2. Metal matrix spike samples are to be spiked prior to the addition of HNO_3. All other aliquots must be stored at 4^0C until analyzed.

9.0 CALCULATIONS — Not Applicable

10.0 QUALITY ASSURANCE

10.1 A minimum of one blank (using the same extraction fluid as used for samples) must be analyzed for every 20 extractions that have been conducted in an extraction vessel.

10.2 A matrix spike shall be performed for each waste type. A minimum of one matrix spike must be analyzed for each analytical batch. Matrix spikes are to be added after filtration of the TCLP extract and before preservation. Matrix spikes should be added at a concentration equivalent to the corresponding regulatory limit.

10.3 The method of standard additions (MSA) shall be used for metals if:

10.3.1 Recovery of the matrix spike for an analyte is < 50% and the concentration does not exceed the regulatory limit.

10.3.2 The concentration of the contaminant measured in the extract is within 20% of the regulatory level.

10.4 All quality control measures described in the appropriate analytical methods shall be followed.

11.0 WASTE DISPOSAL — Not Applicable

12.0 METHOD PERFORMANCE — Not Applicable

13.0 REFERENCES

- USEPA, SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods. Method 1311.

14.0 DEFINITIONS — Not Applicable

15.0 ATTACHMENTS — TCLP WORKSHEET

TCLP Extraction Log, Ref: SWL-IN-700

TCLP Extraction Log (700-ATT1.DOC) SWLO SOP rev. #____ Book # IN700-LOG-1 Page _____

Type of Extraction (circle): TCLP / Zero Head Space

Analyst Initials:_____ Date:_____ Time:_____

Batch ID:

#	SWL ID#	Particle Reduction Necessary?	INITIAL pH	ACID pH	FINAL pH	AMOUNT EXTRACTED	START TEMP & TIME	END TEMP & TIME
1								
2								
3								
4								
5								
6								
7								
8								
9								
10								
11								
12								
13								
14								
15								
16								
17								
18								
19								
20								
21								
22								
23								
24								
25								

*COMMENTS:*_____

Southwest Laboratory of Oklahoma, Inc.

Verified:_____

ATTACHMENT 1
TCLP Worksheet

Request for New or Revised Procedure, SWL-GA-101

SWL SOP Rev. # 1.0 - 05/14/97; Form ID: GA101-DOCREQ-F: Location/File ID: F:\SOP\GA\101-ATT5

REVISED (includes Forms/Logs)

Submitted By: CHH Date: 12/1/97

Document Title: Toxicity Characteristic Leaching Procedure

Document No.: SWL-IN-700 Rev. No. 3.1 - 12102197

Reference: Page 6 of 7 Section: 8.5 ; Subpart: 8.5.5

Attachment No. _____ Title: Attach 1 - New Worksheet

Brief Description (need for revision): Metal Matrix Spike Samples are to be spiked prior to the addition of HNO_3

Approved: _____ Dated: _____ Denied: ☐ PM/SS Initials: _____
Program Manager/Section Supervisor (see attached explanation)

NEW (Procedural Document)

Submitted By: _____ Date: _____

Justification for New Procedure: _____

Approved: _____ Dated: _____ Denied: ☐ PM/SS Initials: _____
Program Manager/Section Supervisor (see attached explanation)

Author: _____ Assigned By: _____ Date: _____

Review/Approval by Manager/Supervisor

Draft No. ___ Author's Initials: ___ Date: ___
Reviewed: ☐ Revise: ☐ Approved: ☐
PM/SS Initials ___ Date ___ (revise as marked)

Draft No. ___ Author's Initials: ___ Date: ___
Reviewed: ☐ Revise: ☐ Approved: ☐
PM/SS Initials ___ Date ___ (revise as marked)

Draft No. ___ Author's Initials: ___ Date: ___
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SOUTHWEST LABORATORY OF OKLAHOMA, INC.

Standard Operating Procedure

Analysis of TS, TDS, TSS, TVS and Fixed Solids

Document No.: SWL-IN-600

Rev No. / Date: Rev. 3.0 — 09/10/97

APPROVALS	DATES
Procedure Prepared By	09/16/97 Date
Program Manager or Section Supervisor	09/16/97 Date
Laboratory Safety Officer	9/17/97 Date
Laboratory QA/QC Officer	9/17/97 Date

(Effective Date is 5 calendar days after the last signature above - QA/QC Officer)

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ANALYSIS OF TS, TDS, TSS, TVS, AND FIXED SOLIDS

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1.0 SCOPE AND APPLICATION

1.1 Total Solids, Total Suspended Solids, and Total Dissolved Solids.

1.1.1 This SOP is applicable for drinking, surface and saline water, domestic and industrial wastes.

1.1.2 The practical range of the determinations vary from 4mg/l to 20,000mg/l for Total Suspended Solids (TSS) to 10mg/l to 20,000mg/l for Total Solids (TS) and Total Dissolved Solids (TDS).

1.2 Total Volatile Solids (TVS) and Fixed Solids.

1.2.1 This method determines the weight of solid material combustible at 550°C.

1.2.2 The determination of volatile solids is useful in control of wastewater treatment plant operation because it offers a rough approximation of the amount of organic matter present in the solid fraction of wastewater, activated sludge and industrial wastes.

2.0 METHOD SUMMARY

2.1 Total Solids — A well-mixed sample is dried to constant weight at 103°C to 105°C.

2.2 Total Suspended Solids and Total Dissolved Solids — A well-mixed sample is filtered through a glass fiber filter. The residue retained on the filter is dried to constant weight at 103°C to 105°C for Total Suspended Solids, while the filtrate is dried to constant weight at 180°C for Total Dissolved Solids.

2.3 Total Volatile and Fixed Solids – The residue from the solids analysis is ignited to constant weight at 500°C ± 50°C. The remaining solids represent the fixed total dissolved and suspended solids, while the weight lost on ignition is the volatile solids.

3.0 HEALTH AND SAFETY

3.1 All samples should be considered hazardous and handled as set forth in the current version of SOP, SWL-GA-111 Laboratory Safety Plan.

3.2 Crucibles and evaporating dishes will be extremely hot after ignition. The use of tongs and heavy-duty gloves are required, when performing the total volatile solids test.

4.0 SAMPLE PRESRVATION, CONTAINERS, HANDLING AND STORAGE

4.1 Non-representative particulate such as leaves, sticks, fish and lumps of fecal matter should be excluded from the sample.

4.2 Preservation of the sample is not practical; analysis should begin as soon as possible. Refrigeration to 4°C minimizes microbiological decomposition of the solids.

4.3 Samples may be collected in pre-cleaned glass or plastic containers.

4.4 Holding time is 7 days from sample collection.

5.0 INTERFERENCES AND POTENTIAL PROBLEMS

5.1 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 of total dissolved solids.

5.2 Too many residues in the evaporating dish may form a water-trapping crust, which will entrap water that will not be driven off during drying. Residue should be limited to about 200mg for total dissolved solids.

5.3 Samples containing high concentrations of bicarbonate will require careful and possibly prolonged drying at 180°C for dissolved solids to ensure that all the bicarbonate is converted to carbonate.

5.4 For the analysis of total suspended solids, samples high in dissolved solids such as saline waters, brines and some waters, may be subject to a positive interference. Care must betaken in selecting the filtering apparatus so that washing of the filter and any dissolved solids in the filter minimizes this potential interference.

5.5 Negative errors in the volatile solids may be produced by loss of volatile matter during drying.

6.0 EQUIPMENT/APPARATUS

6.1 Evaporating dishes, porcelain: 100ml volume.

6.2 Filter supports: Gooch Crucible and holder.

6.3 Vacuum flask, 1L.

6.4 Gooch Crucibles, 25ml.

6.5 Drying oven, 103°-105°C

6.6 Drying oven, 180°C.

6.7 Analytical balances, Mettler H80 or S/P180.

6.8 Desiccator

6.9 Muffle Furnace, capable of temperatures of 500 ± 50°C.

6.10 Tongs

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6.11 Gloves – Temperature Resistant

7.0 REAGENTS - Not Applicable

8.0 PROCEDURE

8.1 Total Solids

8.1.1 Prepare the evaporating dishes by heating the clean dish to 103°C to 105°C for one hour. Cool in desiccator and store until needed.

8.1.2 Weigh the dish on an analytical balance, recording the weight in the Solids Logbook (see Attachment) to four decimal places.

8.1.3 Shake the sample vigorously and rapidly transfer 100ml to the evaporating dish by means of a 100ml graduated cylinder. Rinse the graduated cylinder with three portions of ASTM Type II water, putting each rinse into the evaporating dish.

8.1.4 Transfer the evaporating dish to the drying oven and evaporate to dryness without splattering. Dry the evaporated sample for at least 1 hour in an oven at 103°C to 105°C. Cool the dish in a desiccator to balance temperature and weigh. Record the weight to four decimal places in the solids logbook.

8.1.5 Repeat the cycle of drying, cooling, desiccating and weighing until a constant weight is obtained, or until weight loss is less than 4% of previous weight or 0.5mg, whichever is less.

8.2 Total Dissolved Solids

8.2.1 Prepare the glass fiber disc by placing the disc on the bottom of a Gooch Crucible. While vacuum is applied, wash the disc with three successive 20ml volumes of distilled water. Remove all traces of water by continuing to apply vacuum. Discard washings.

8.2.2 Prepare the evaporating dish by heating the dish to 180°±2°C for one hour. Cool in a desiccator and store until needed.

8.2.3 Immediately before use weigh the dish on an analytical balance and record the weight to four decimal places in the solids logbook.

8.2.4 Assemble the filtering apparatus and begin suction. Shake the sample vigorously and rapidly transfer 100ml to the funnel by means of a 100ml-graduated cylinder.

8.2.5 Filter the sample through the glass fiber filter. Rinse the cylinder and Gooch crucible with three portions of distilled water and continue to apply vacuum for about 3 minutes after filtration is complete to remove as much water as possible.

8.2.6 Transfer the filtrate to the weighed evaporating dish and evaporate to dryness in the oven at a temperature less than 100°C.

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8.2.7 When the dish is dry, increase temperature to 180°C and heat for at least one hour at 180°C ± 2°C cool in a desiccator to balance temperature and weigh. Record weight to four decimal places in the solids logbook.

8.2.8 Repeat cycle of drying, cooling, desiccating and weighing until a constant weight is obtained or until weight loss is less than 4% of previous weight or 0.5mg, whichever is less.

8.3 Total Suspended Solids

8.3.1 Insert a glass fiber filter into the bottom of a Gooch crucible with the wrinkled surface up.

8.3.2 Apply vacuum, and then wash the filter with three successive 20ml volumes of distilled water. Remove all traces of water by continuing to apply vacuum after water has passed through.

8.3.3 Place the crucible and filter in a drying oven and dry at 103-105°C for at least one hour. Remove to a desiccator and store until needed.

8.3.4 Immediately before use weigh the crucible and filter on an analytical balance. Handle the crucible with forceps or tongs only. Record the weight of the crucible and filter to four decimal places in the solids logbook.

8.3.5 Assemble the filtering apparatus and begin suction. Wet the filter with a small volume of distilled water to seat it against the fritted support.

8.3.6 Shake the sample vigorously and transfer 100ml of the sample to the filter using a graduated cylinder. If the sample has a lot of particulate matter the analyst should use their judgement on how small of volume to use. Remove all traces of water by continuing to apply vacuum after sample has passed through.

8.3.7 With the suction on, wash the graduated cylinder and Gooch crucible wall with three portions of distilled water allowing complete drainage between washing. Remove all traces of water by continuing to apply vacuum after water has passed through.

8.3.8 Using tongs, remove the crucible and filter and place in a drying oven at 103-105°C for at least one hour. Cool in a desiccator to balance temperature, then weigh and record weight in solids logbook.

8.3.9 Repeat the cycle of drying, desiccating and weighing until a constant weight is obtained or until the weight loss is less than 4% of the previous weight or 0.5mg, whichever is less.

8.4 Total Volatile Solids

8.4.1 Turn on muffle furnace and let stabilize at 500 ± 50°C.

8.4.2 Place residue produced from either the total, suspended or dissolved solids test into the muffle furnace for ignition.

8.4.3 Let the sample ignite for 15 to 20 minutes. Remove with tongs and let the dish or crucible cool partially in air until most of the heat has dissipated. Transfer to a dessicator for final cooling in a dry atmosphere.

8.4.4 Weigh dish as soon as it has cooled to balance temperature and record weight in column D (Dish or Crucible + Residue) of the solids logbook (see attachment 1).

8.4.5 Repeat the cycle of igniting, cooling, desiccating and weighing until a constant weight is obtained or until weight change is less than 4% or 0.5 mg, whichever is less.

8.5 Write up and Review

8.5.1 Fill out the appropriate information on the Analysis Log (see attachment 1).

8.5.2 Review data in accordance with section 10 and current revision of SWL-IN-107.

8.5.3 Enter results into LIMS and place a copy of the analysis log with the LIMS Episode work sheet.

9.0 CALCULATIONS

9.1 Total Solids:

$$\text{Total Solids, mg/l} = \frac{(A - B) \times 1000}{C}$$

Where:
A = Weight of evaporating dish + residue in mg.
B = Weight of evaporating dish in mg.
C = ml of sample used

9.2 Total Dissolved Solids

$$\text{Total Dissolved Solids, mg/l} = \frac{(A - B) \times 1000}{C}$$

Where:
A = Weight of evaporating dish + residue in mg.
B = Weight of evaporating dish in mg.
C = ml of sample used

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9.3 Total Suspended Solids

$$\text{Total Suspended Solids, mg/l} = \frac{(A - B) \times 1000}{C}$$

Where:
A = Weight of filter and crucible + residue in mg.
B = Weight of filter and crucible in mg.
C = ml of sample used

9.4 Total Volatile Solids

$$\text{Total Volatile Solids, mg/l} = \frac{(A - D) \times 1000}{C}$$

Where:
A = Weight of residue + dish before ignition, mg.
D = Weight of residue + dish after ignition, mg.
C = ml of sample used

9.5 Total Fixed Solids

$$\text{Total Fixed Solids, mg/l} = \frac{(D - B) \times 1000}{C}$$

Where:
B = Dish or crucible weight, mg.
D = Weight of residue + dish after ignition, mg.
C = ml of sample used

NOTE: A and D (sections 9.1 - 9.5) indicates the lowest weight value.

10.0 QUALITY ASSURANCE/QUALITY CONTROL

10.1 A blank of 100mls of distilled water must be run with every batch of ≤20 samples.

10.2 A Laboratory Control Sample (LCS) must be analyzed with every batch of ≤20 samples. Not required for total volatile solids and fixed solids.

10.3 A duplicate sample will be analyzed with every batch of ≤20 samples.

10.4 Filter the QC samples after filtering each batch of samples to show that there is no contamination or carry-over between samples.

11.0 WASTE DISPOSAL

11.1 All samples will be disposed of as instructed in the current version SOP SWL-GA-114, Hazardous Waste Management Plan.

12.0 METHOD PERFORMANCE

12.1 Blanks must be less than the reporting limit of 4mg/l for total suspended solids and 10mg/l for total, total dissolved solids, and total volatile solids.

12.2 The LCS must be ± 15% recovery of the true value.

12.3 Duplicate precision should be within ± 20% recovery.

13.0 REFERENCES

13.1 USEPA, Methods for Chemical Analysis of Water and Wastes, Methods 160.1, 106.2, 160.3, and 160.4.

13.2 Standard Methods for the Examination of Water and Wastewater, 19th Edition, Methods 2540B, 2540C, 2540D and 2540E.

14.0 DEFINITIONS

14.1 Solids: ..... Matter suspended or dissolved in water or wastewater. Total Solids includes "Total Suspended Solids", the portion of total solids retained by a filter, and "Total Dissolved Solids", the portion that passes through the filter.

14.2 Fixed Solids..... Residue of total, suspended or dissolved solids remaining after ignition for a specified time of a specified temperature.

14.3 Volatile Solids..... The weight loss on ignition.

14.4 Sample Batch: ..... Consists of a maximum of twenty samples per matrix.

14.5 Blank:..... Method Blank, consisting of 100ml of distilled water taken through the analysis to check for contamination.

14.6 Laboratory Control Sample:... A sample of known concentration taken through the analysis to check the method accuracy.

14.7 Sample Duplicate: ..... A sample taken through the analysis to check the matrix effect on method precision.

15.0 ATTACHMENTS:

- Attachment 1: Solids Logbook

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ATTACHMENT 1
Solids Logbook

Solids Analysis Log, Ref: SWL-IN-600

Analysis Batch Log (600-ATT2.DOC) SWLO SOP rev. #_____ Book # IN600-LOG-1 Page _____

OVEN Temp:_____ Type of Analysis (circle): TS, TDS, TSS, TVS, Fixed Solids

Analyst Initials: Date: Time: Batch ID:

#	SWL ID#	COMMENTS	Dish or Crucible Wt. (B) g	Sampl Vol (C) ml	Crucible + Residue #1 Wt. (A) g	Crucible + Residue #2 Wt. (A) g	Crucible After Ignit. #1 Wt. (D) g	Crucible After Ignit. #2 Wt. (D) g	A-B A-D D-B	Final Result mg/l	%R
1											
2											
3											
4											
5											
6											
7											
8											
9											
10											
11											
12											
13											
14											
15											
16											
17											
18											
19											
20											

Calculations: TS, TDS, TSS = ((A-B) x 1000) / C TVS = ((A-D) x 1000) / C Fixed Solids = ((D-B) x 1000) / C

Southwest Laboratory of Oklahoma, Inc.

Verified:_____

C2-119

Request for New or Revised Procedure, SWL-GA-101

SWL SOP Rev. # 1.0 - 05/14/97; Form ID: GA101-DOCREQ-F: Location/File ID: F:\SOP\GA\101-ATT5

REVISED (includes Forms/Logs) Submitted By: CP Date: 9/17/97

Document Title: ~~Analysis of~~ Total Dissolved Solids (TDS

Document No.: SWL-I.N-600 Rev. No. 2.2 — 05/15/97

Reference: Page ____ of ____ Section: ___.___; Subpart: _____

Attachment No. _____ — Title: _____

Brief Description (need for revision): New Title: Analysis of TS, TDS, TSS, TVS and Fixed Solids Place in new format incorporate + Retire SOP's SWL-IN-601, 602, 604, 605

Approved: _____ Dated: 09/17/97 Denied: ☐ PM/SS Initials: _____
Program Manager/Section Supervisor (see attached explanation)

NEW (Procedural Document) Submitted By: _____ Date: _____

Justification for New Procedure: _____

Approved: _____ Dated: _____ Denied: ☐ PM/SS Initials: _____
Program Manager/Section Supervisor (see attached explanation)

Author: _____ Assigned By: _____ Date: _____

Review/Approval by Manager/Supervisor

Draft No. ___ Author's Initials: ____ Date: ____
Reviewed: ☐ Revise: ☐ Approved: ☐
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SOUTHWEST LABORATORY OF OKLAHOMA, INC.

TM

Standard Operating Procedure

Chemical Oxygen Demand Analysis

(IN090)

Document No.: SWL-IN-403

Rev No. / Date: Rev. 3.0 — 07/28/97

APPROVALS	DATES
Procedure Prepared By	08/01/97 Date
Inorganic Program Manager	08/01/97 Date
Laboratory Safety Officer	8/1/97 Date
Laboratory QA/QC Officer	8/1/97 Date

(Effective Date is 5 calendar days after the last signature above - QA/QC Officer)

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CHEMICAL OXYGEN DEMAND

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1.0 SCOPE AND APPLICATION

1.1 This SOP gives two procedures for the determination of COD. Both methods cover COD in surface waters, domestic and industrial wastes.

1.2 The applicable range of the low level method is 5-150ppm, samples above 150ppm may be diluted, then run using same standard curve, or may be run using the mid-high range method.

1.3 The applicable range of the mid-high level method is 10-1500ppm, samples above 1500ppm may be diluted, then run using same standard curve.

NOTE: The mid-high level will be used unless client asks for the Low Level Method.

2.0 METHOD SUMMARY

— Sample, blanks and standards in sealed Hach tubes are heated in a block digestor at 150°C for a minimum of two hours. After heating period, the tubes are removed from the digestor, cooled and measured spectrophotometrically at 600nm.

3.0 HEALTH AND SAFETY

— Refer to the current version of Laboratory Safety Plan, SOP SWL-GA-111.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING & STORAGE

4.1 Collect the samples in glass bottles if possible. Use of plastic containers is permissible if it is known that no organic contaminants are present in the containers.

4.2 Samples should be preserved with sulfuric acid to a pH <2 and maintained at 4°C until analysis.

4.3 Holding Time: 28 days from collection, if preserved. Non-preserved samples should be run as soon as possible from collection date.

5.0 INTERFERENCES AND POTENTIAL PROBLEMS

— Chloride are quantitatively oxidized by dichromate and represent a positive interference. Mercuric sulfate has been added to the Hach tubes to complex the chlorides.

6.0 EQUIPMENT/APPARATUS

6.1 Block digestor, 150°C

6.2 Hach COD tubes 0-1500ppm (mid-high level).

6.3 5.0ml x 1/10 Disposable Serological pipets.

6.4 Spectrophotometer.

6.5 Hach COD tubes 0-150ppm (low-level).

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7.0 REAGENTS

7.1 Hach COD Tubes (appropriate level), each tube contains:

7.1.1 H_2SO_4 98% concentration

7.1.2 Mercuric Sulfate

7.1.3 Chromic Acid

7.1.4 Ag_2SO_4

7.1.5 DI water

7.2 Stock potassium acid phthalate (KHP): Dissolve 1.700g of dried (2 hours at 120°C) KHP in 800mls of distilled water and dilute to 1 liter. 1ml = 2mg of COD; 2000mg/l COD. This standard is required to be refrigerated, and has a shelf life of 4 months.

8.0 PROCEDURE

8.1 Calibration

8.1.1 Low Level Method — Prepare six standard solutions; i.e. 5, 25, 50, 100, and 125mg/l, by diluting appropriate volumes of the stock standard (see Section 7.2).

8.1.2 Mid Level Method — Prepare six standard solutions, i.e. 10, 50, 100, 500, 1000, and 1500mg/l by diluting appropriate volumes of stock standard (see Section 7.2).

8.1.3 Analyze DI water as a reagent blank with each standard curve.

8.1.4 Follow steps 8.2-8.9 for digestion and analysis. Keep in mind the Mid-High and Low method's have separate tubes used specifically for that method.

8.2 Turn on block digestor.

8.3 Mix both unknowns and standards by inversion.

8.4 Add with 5.0ml disposable serological pipet 2.0mls of sample to each tube. Use same volume for both standards and unknowns.

8.5 Use DI water for a reagent blank with each run of unknowns to set zero absorbance on spectrophotometer.

8.6 Cap tightly and mix tubes by inversion to blend both phases.

8.7 Place tubes in block digestor at 150°C ± 5°C for two hours. At end of two hours , turn on spectrophotometer with wavelength set at 600 nm.

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8.8 Remove tubes from digestor, place in Nalgene rack, and allow to cool for 10 minutes. Mix each tube by inversion twice and allow any precipitate to settle and for tubes to reach room temperature, about 20 minutes.

8.9 Measure and record absorbency at 600nm, making sure that the Hach label on the tubes faces to the right in the spectrophotometer.

8.10 Write-up and Review

8.10.1 Enter the appropriate information on the Analysis Log (see Attachment 1).

8.10.2 Review data according to Section 10.0 of this SOP, and SOP SWL-IN-107.

8.10.3 Attach a copy of the Analysis Log to the LIMS worksheet, report QC samples on appropriate QuatroPro sheet, and report results in LIMS.

9.0 CALCULATIONS

9.1 Prepare a standard curve by plotting absorbency against known concentrations of standards, of the appropriate method.

9.2 Compute concentration of unknowns by comparing sample response to standard curve.

9.3 Reporting Limits are 5.0mg/l (low-level) and 10.0mg/l (mid-high level).

10.0 QUALITY ASSURANCE/QUALITY CONTROL

10.1 The calibration coeffient will be ≥0.995. The calibration will be required with every new lot # of tubes.

10.2 PBW must be analyzed with each batch of 20 samples or less. The result must be below 5.0 for low-level and 10.0 for mid-high level. Re-prep the batch of samples if PBW fails control limits. Zero the instrument on this sample (if instrument was calibrated on a previous day) recording the reading it first read.

10.3 LCW/LDW (ICV): must be analyzed with each batch of 20 samples or less. The true value will be 100-400 mg/L (mid-high level) or 25-50 mg/L (low level). In-house control limits for the LCS shall be used, and may be found in the Blue Book. If in-house control limits are not available, then use 90-110 percent recovery. Re-prep the batch of samples if LCW/LDW fails control limits.

10.5 MS/MSD will be done with each batch of 20 samples or less. For the low level; spike the sample with 0.05ml of the 2000mg/L standard (Section 7.2) giving a true value of 50mg/L COD. For the mid-high level; spike the sample with 0.1ml of the 2000mg/L standard (Section 7.2) giving a true value of 100mg/L COD. MS and MSD have control limits of 75-125 percent recovery and 20% RPD. If control limits are exceeded flag data.

10.6 Duplicates will be done on client request. The duplicates have a control limit of 20% RPD and data will be flagged, if control limits are exceeded.

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ATTACHMENT 1
COD Analysis Log

COD Analysis Log, Ref: SWL-IN-403

COD Analysis Log (403-ATT1.DOC) SWLO SOP rev. #____ Book # . IN403-COD-1 Page _____

Low Level /or Mid-High Level

Analyst:_____ Date:_____ Prep Batch ID:_____

Tube Lot #:_____ Last Calibration Performed on Book# IN403-COD-____ Page._____

#	SWL ID#	Sample Vol. (ml)	Absorb. Reading	Calibration CurveValue (mg/l)	Final Value (mg/l)	%R	Comments
1							
2							
3							
4							
5							
6							
7							
8							
9							
10							
11							
12							
13							
14							
15							
16							
17							
18							
19							
20							

C2-126

PROCEDURAL DOCUMENTS - SYSTEM TRANSITION

Please note that the following document is in transition from a previous system. The document identification number (listed below) has been assigned in compliance with the SWL SOP titled:

"Preparation, Review, Revision, and Control of Procedural Documents", SWL-GA-101, Rev. 1.0 — 05/14/97

The ID given below integrates the following SOP into the new system set forth in GA-101 prior to its annual review (or conversion of format and numbering). Each ID is unique and a "key" field in the new document control database system. The assignment of a new number is therefore necessary for its tracking in the new system.

Upon revision or review, the following document will be re-issued in full compliance with the guidelines set forth in GA-101, therefore consistent with newly converted or written procedural documents.

Existing ID: IN025

NEW DOCUMENT IDENTIFICATION NUMBER:

SWL-IN-415

Standard Operating Procedure

"Biochemical Oxygen Demand in Water"
Rev. No. 1.1
Issued: 10/28/96

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C2-127

SOUTHWEST LABORATORY OF OKLAHOMA, INC.

STANDARD OPERATING PROCEDURE FOR THE ANALYSIS OF BIOCHEMICAL OXYGEN DEMAND (BOD) IN WATER

IN025
REV 1.1 — 10/28/96

Document Control # SWL-IN025-2028 2/4/98 MM
Alan Fosdick

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RED STAMP INDICATES ORIGINAL

APPROVALS BY:

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I. SCOPE & APPLICATION

A. This method is an empirical determination of the oxygen requirement for the biochemical degradation of organic and inorganic material in water and wastewater.

B. The method is most applicable to measurement of biochemical oxygen demand above 10 mg/l.

II. SUMMARY OF METHOD

A. A series of dilutions of sample are incubated for 5 days in airtight bottles. The dissolved oxygen content of each bottle is measured prior to and after incubation. The difference in dissolved oxygen demand is calculated.

III. SAMPLE HANDLING & PREPARATION

A. Samples should be stored at 4°C until time of analysis.

B. Holding times are 48 hours from sample collection.

IV. INTERFERENCES

A. Toxic Materials/Heavy Metals

V. APPARATUS

A. Orion D.O. meter

B. 300 ml BOD bottles with stopper and caps

C. Incubator

VI. REAGENTS

A. Distilled water

B. Hach Nutrient/Buffer Pillow

C. Hach Nitrification inhibitor

D. Glucose-glutamic acid solution

E. Seed water

F. Sulfuric Acid

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G. NaOH

VII. PROCEDURE

A. Preparation of Dilution Water

1. Fill a 3 liter glass jug with distilled water and add contents of one nutrient pillow, nitrification inhibitor, and 5 ml of seed, and begin to aerate jug with compressed air.

2. Check dilution water before using by incubating a bottle for 5 days. The D.O. uptake should not be more than 0.2 mg/l. If it is over 0.2 mg/l, prepare a new batch of dilution water.

B. Glucose-glutanic Acid Check

1. Take a mixture of 300 mg glucose per liter and 300 mg glutanic acid per liter, using Hach Bod Standard.

2. Determine the BOD of a 3% dilution of glucose-glutanic acid standard check solution using same procedure as samples.

3. If the BOD value of the check is outside the range of 300 $\pm$ 37, reject any BOD determinations made with the seed and dilution water, and seek the cause of the problem.

C. Sample Preparation

1. Sample should be allowed to come to room temperature, and pH of sample adjusted to 6.5 to 7.0 with sulfuric acid or sodium hydroxide.

2. Begin to aerate sample with compressor air.

D. Set-up of Bottles

1. Arrange numbered bottles and put 1, 5, 50, and 100 mL of aerated sample (or other appropriate quantities) into the bottles.

2. Fill bottles with dilution water to completely fill bottle.

E. Initial D.O. Reading

1. Calibrate instrument per manufacturing instructions

2. Determine initial DO reading with probe for each bottle and record.

3. Seal each bottle with ground glass stopper and plastic cap.

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F. Incubation

1. Place filled BOD bottles in incubator and incubate at 20°C for five days. The bottles should remain in the dark during the incubation period.

G. Post DO Reading

1. Calibrate instrument per manufacturing instructions.
2. After five days, remove bottles and determine DO for each bottle.

H. Calculations

$$BOD\ (mg/l) = (D_1 - D_2) - (B_1 - B_2)\ f/p$$

where: D_1 = initial DO of sample

D_2 = Post DO of sample

p = Decimal fractions of sample used

B_1 = Initial DO of Blank

B_2 = Post DO of Blank

f = Ratio of seed in diluted sample to seed in Blank

Notes: An acceptable dilution will yield at least 2 mg/l.

VIII. QUALITY CONTROL

A. Analyze one duplicate sample for every 10 samples.

IX. REFERENCE STANDARD METHOD 5210B

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NOTE:

PAGES C2-132 TO C2-135

ARE NOT INCLUDED

SOUTHWEST LABORATORY OF OKLAHOMA, INC.

Standard Operating Procedure

Analysis of Polychlorinated Dibenzo-P-Dioxins and Polychlorinated Dibenzofurans by HRGC/HRMS (Method 8290)

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ANALYSIS OF POLYCHLORINATED DIBENZO-P-DIOXINS AND POLYCHLORINATED DIBENZOFURANS BY HRGC/HRMS (METHOD 8290)

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1.0 SCOPE AND APPLICATION

1.1 This method is appropriate for the detection and quantitative measurement of 2,3,7,8-tetrachlorinated dibenzo-p-dioxin (2,3,7,8-TCDD) and total tetra-, penta-, hexa-, hepta-, and octachlorinated dibenzo-p-dioxins (PCDDs) and dibenzofurans (PCDFs) in a variety of matrices and at part-per-trillion (ppt) to part-per-quadrillion (ppq) concentrations. The analytical method requires the use of high resolution gas chromatography and high resolution mass spectrometry (HRGC/HRMS) on purified sample extracts that have been subjected to specified cleanup procedures.

1.2 This method is designed for use only by analysts experienced with residue analysis and skilled in HRGC/HRMS.

1.3 Because of the extreme toxicity of these compounds, the analyst must take necessary precautions to prevent exposure of personnel to materials known or believed to contain PCDDs/PCDFs.

2.0 METHOD SUMMARY

2.1 This procedure is derived from the U.S. EPA "Test Methods For Evaluating Solid Waste," (SW-846 Method 8290, September, 1994, Update II).

2.2 This procedure uses a matrix-specific extraction, analyte-specific cleanup, and high-resolution capillary column gas chromatography/high resolution mass spectrometry (HRGC/HRMS) techniques.

2.3 If interferents are encountered, the method provides selected cleanup procedures to aid the analyst in their elimination. The cleanup flow chart is shown in Figure 1.

2.4 A specified amount of soil, sediment, fly ash, water, sludge (including paper pulp), still bottom, fuel oil, chemical reactor residue, fish tissue, or human adipose tissue is spiked with a solution containing specified amounts of each of the nine isotopically labeled PCDDs/PCDFs listed in Column 1 of Table 4. The sample is then extracted according to a matrix specific extraction procedure. The extraction procedures are:

2.4.1 Toluene: Soxhlet extraction for soil, sediment, fly ash, and paper pulp samples;

2.4.2 Methylene chloride: liquid-liquid extraction or separatory funnel extraction for water samples;

2.4.3 Toluene: Dean-Stark extraction for fuel oil and aqueous sludge samples;

2.4.4 Toluene extraction for still bottom samples;

2.4.5 Hexane/methylene chloride or methylene chloride: Soxhlet extraction for fish tissue samples; and

2.4.6 Methylene chloride extraction for human adipose tissue samples.

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2.4.7 As an option, all solid samples (wet or dry) may be extracted with toluene using a Soxhlet/Dean Stark extraction system.

2.4.8 The decision for the selection of an extraction procedure for chemical reactor residue samples is based on the appearance (consistency, viscosity) of the samples. Generally, they can be handled according to the procedure used for still bottom (or chemical sludge) samples.

2.5 The extracts are submitted to an acid-base washing treatment and dried. Following a solvent exchange step, the extracts are cleaned up by column chromatography on alumina, silica gel, and activated carbon.

2.5.1 The extracts from adipose tissue samples are treated with silica gel impregnated with sulfuric acid before chromatography on acidic silica gel, neutral alumina, and activated carbon.

2.5.2 Fish tissue and paper pulp extracts are subjected to an acid wash treatment only, prior to chromatography on alumina and activated carbon.

2.6 The preparation of the final extract for HRGC/HRMS analysis is accomplished by adding 20µL (depending on the matrix) of a nonane solution containing 50 pg/µL of the recovery standards 13C12-1,2,3,4-TCDD and 13C12-1,2,3,7,8,9-HxCDD (Table 4). The former is used to determine the percent recoveries of tetra- and pentachlorinated PCDD/PCDF congeners, while the latter is used to determine the percent recoveries of the hexa-, hepta- and octachlorinated PCDD/PCDF congeners.

2.7 Two µL of the concentrated extract are injected into an HRGC/HRMS system capable of performing selected ion monitoring at resolving powers of at least 10,000 (10% valley definition).

2.8 The identification of OCDD and nine of the fifteen 2,3,7,8-substituted congeners (Table 3), for which a 13C-labeled standard is available is based on their elution at their exact retention time (within 0.005 retention time units measured in the routine calibration), and the simultaneous detection of the two most abundant ions in the molecular ion region. The remaining six 2,3,7,8-susbtituted congeners for which no carbon-labeled internal standards are available in the sample fortification solution, and all other PCDD/PCDF congeners, are identified when their relative retention times fall within their respective PCDD/PCDF retention time windows, as established from the routine calibration data, and the simultaneous detection of the two most abundant ions in the molecular ion region. The identification of OCDF is based on its retention time relative to 13C12-OCDD and the simultaneous detection of the two most abundant ions in the molecular ion region. Identification also is based on a comparison of the ratios of the integrated ion abundance of the molecular ion species to their theoretical abundance ratios.

2.9 Quantitation of the individual congeners, total PCDDs and total PCDFs is achieved in conjunction with the establishment of a multipoint (five points) calibration curve for each homologue, during which each calibration solution is analyzed once.

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3.0 HEALTH AND SAFETY

3.1 The 2,3,7,8-TCDD isomer has been found to be acnegenic, carcinogenic, and teratogenic in laboratory animal studies. Other PCDDs and PCDFs containing chlorine atoms in positions 2,3,7, and 8 are known to have toxicities comparable to that of 2,3,7,8-TCDD.

3.2 Because of the extreme toxicity of the PCDD and PCDF compounds, necessary precautions must be taken to prevent the exposure of laboratory personnel to materials known or believed to contain PCDDs and PCDFs. All samples, standards, and solvents are treated as potentially hazardous and exposure to them is minimized. Any spills that occur are cleaned up immediately. Refer to the current version of "Laboratory Safety Plan" SWL-GA-1111 and section 8.5 of this SOP (OD205) for frequency and procedure for wipe tests.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, & STORAGE

4.1 Water Samples - Each water sample received will consist of at least two (2) 1-liter (or quart) amber glass bottles. Do not freeze. After a portion of the sample is removed for analysis, the unused portion of the sample is stored in a locked, limited access area for at least 60 days from the date of data submission.

4.2 Soil/Chemical Waste Samples - When received, each sample will be contained in a 1-pint glass jar surrounded by vermiculite in a sealed metal paint can. Until a portion is removed for analysis, the sealed sample must be stored in a locked limited access area where ambient temperature is maintained above freezing. After a portion is removed for analysis, the unused portion of the sample is returned to its original container and stored at ambient temperature for at least 60 days from the date of data submission.

4.3 Grinding or Blending of Fish Samples – If not otherwise specified, the whole fish (frozen) should be blended or ground to provide a homogeneous sample. In some circumstances, analysis of fillet or specific organs of fish may be requested. If so requested, the above whole fish requirement is superseded.

4.4 Storage and Holding Times – All samples, except fish and adipose tissue samples, must be stored at 4°C in the dark, extracted within 30 days and completely analyzed within 45 days of extraction. Fish and tissue samples must be stored at –20°C in the dark. Whenever samples are analyzed after the holding time expiration date, the results should be considered to be minimum concentrations and must be identified as such.

NOTE: The holding times listed in Sec. 4.4 are recommendations. PCDDs and PCDFs are very stable in a variety of matrices, and holding times under the conditions listed in Sec. 4.4 may be as high as a year for certain matrices. Sample extracts, however, should always be analyzed within 45 days of extraction.

4.5 Phase Separation - This is a guideline for phase separation for very wet (>25 percent water) soil, sediment and paper pulp samples. Place a 50g portion in a suitable centrifuge bottle and centrifuge for 30 minutes at 2,000 rpm. Remove the bottle and mark the interface level on the bottle. Estimate the relative volume of each phase. With a disposable pipette, transfer the liquid layer into a clean bottle. Mix the solid with a

stainless steel spatula and remove a portion to be weighed and analyzed (percent dry weight determination, extraction). Return the remaining solid portion to the original sample bottle (empty) or to a clean sample bottle that is properly labeled, and store it as appropriate. Analyze the solid phase by using only the soil, sediment and paper pulp method. Take note of, and report, the estimated volume of liquid before disposing of the liquid as a liquid waste.

4.6 Soil, Sediment, or Paper Sludge (Pulp) Percent Dry Weight Determination - The percent dry weight of soil, sediment or paper pulp samples showing detectable levels (see note below) of at least one 2,3,7,8-substituted PCDD/PCDF congener is determined according to the following procedure. Weigh a 10g portion of the soil or sediment sample (+/- 0.5g) to three significant figures. Dry it to constant weight at 105-110°C in an adequately ventilated oven. Allow the sample to cool in a desiccator. Weigh the dried solid to three significant figures. Calculate and report the percent dry weight. Do not use this solid portion of the sample for extraction, but instead dispose of it as hazardous waste.

NOTE: Until detection limits have been established, the lower MCLs (Table 1) may be used to estimate the minimum detectable levels.

$$\% \text{ dry weight} = \frac{\text{g of dry sample}}{\text{g of sample}} \times 100$$

CAUTION: Finely divided soils and sediments contaminated with PCDDs/PCDFs are hazardous because of the potential for inhalation or ingestion of particles containing PCDDs/PCDFs (including 2,3,7,8-TCDD). Such samples should be handled in a confined environment (i.e., a closed hood or a glove box).

4.7 Lipid Content Determination

4.7.1 Fish Tissue - To determine the lipid content of fish tissue, concentrate 125mL of the fish tissue extract (Sec. 8.2.2), in a tared 200 mL round bottom flask, using a KD apparatus with vacuum, until a constant weight (W) is achieved.

$$\text{Percent lipid} = \frac{100\,(W)}{10}$$

Dispose of the lipid residue as a hazardous waste if the results of the analysis indicate the presence of PCDDs or PCDFs.

4.7.2 Adipose Tissue - Details for the determination of the adipose tissue lipid content are provided in Sec. 8.3.3.

5.0 INTERFERENCES AND POTENTIAL PROBLEMS

5.1 Any compound that yields ions of PCDDs/PCDFs and also elutes within the retention time window of the corresponding homologue is a potential interference. PCDDs/PCDFs

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are often associated with other chlorinated compounds such as polychlorinated biphenyls (PCBs) and polychlorinated diphenyl ethers (PCDPEs). These compounds may be found at concentrations several orders of magnitude higher than that of the analytes of interest, and may otherwise interfere with the analysis of PCDDs/PCDFs.

5.2 Solvents, reagents, glassware, and other sample processing hardware may yield discrete artifacts and/or elevated baselines which may cause misinterpretation of chromatographic data. All of these materials shall be demonstrated to be free from interferents under the conditions of analysis by performing laboratory method blanks. Analysts should avoid using PVC gloves.

5.3 The use of high purity reagents and solvents helps to minimize interference problems. Purification of solvents by distillation in all glass systems may be necessary.

5.4 High resolution capillary columns are used to resolve as many PCDD/PCDF isomers as possible; however, no single column is known to resolve all of the isomers.

6.0 EQUIPMENT/APPARATUS

6.1 High Resolution Gas Chromatograph/High Resolution Mass Spectrometer/Data System (HRGC/HRMS/DS) – The GC must be equipped for temperature programming, and all required accessories must be available, such as syringes, gases, and capillary columns.

6.1.1 GC Injection Port – The GC injection port must be designed for capillary columns. The use of splitless injection techniques is recommended. A 2µL injection volume is used consistently in this method.

6.1.2 Gas Chromatograph/Mass Spectrometer (GC/MS) Interface – The GC/MS interface components should withstand 350°C. The interface must be designed so that the separation of 2,3,7,8-TCDD from the other TCDD isomers achieved in the gas chromatographic column is not appreciably degraded. It is recommended that the GC column be fitted directly into the mass spectrometer ion source without being exposed to the ionizing electron beam.

6.1.3 Mass Spectrometer – The static resolving power of the instrument must be maintained at a minimum of 10,000 (10% valley).

6.1.4 Data System – A dedicated data system is employed to control the rapid multiple-ion monitoring process and to acquire the data. Quantitation data and SIM traces must be acquired during the analyses and stored. Quantitations may be reported based upon computer generated peak areas or upon measured peak heights. The data system must be capable of acquiring data at a minimum of 10 ions in a single scan. The data system should be able to provide hard copies of individual ion chromatograms for selected gas chromatographic time intervals. It should also be able to acquire mass spectral peak profiles and provide hard copies of peak profiles to demonstrate the required resolving power.

6.2 GC Columns – In order to have an isomer specific determination for 2,3,7,8-TCDD and to allow the detection of OCDD/OCDF within a reasonable time interval in one

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HRGC/HRMS analysis, use of the 60m DB-5 fused silica capillary column is recommended.

6.2.1 Minimum acceptance criteria must be demonstrated and documented. At the beginning of each 12-hour period during which sample extracts or concentration calibration solutions will be analyzed, column operating conditions must be attained for the required separation on the column to be used for samples.

6.2.2 Isomer specificity for all 2,3,7,8-substituted TCDFs cannot be achieved on the 60 m DB-5 column. In order to determine the proper concentrations of the individual 2,3,7,8-TCDF, the sample extract must be reanalyzed on another GC column that resolves the isomers such as the SP-2331 fused silica capillary column or equivalent.

6.3 Miscellaneous Equipment and Materials – The following list of items does not necessarily constitute an exhaustive compendium of the equipment needed for this analytical method.

6.3.1 Nitrogen blowdown apparatus.

6.3.2 Analytical balance.

6.3.3 Water bath.

6.3.4 Glove box.

6.3.5 Centrifuge.

6.3.6 Drying oven.

6.3.7 Extraction jars.

6.3.8 Kuderna-Danish Apparatus (KD) with a provision for vacuum.

6.3.9 Disposable Pasteur pipets.

6.3.10 Disposable serological pipets.

6.3.11 Mini vials.

6.3.12 Funnels.

6.3.13 Concentrator tubes.

6.3.14 Separatory funnels.

6.3.15 Continuous liquid-liquid extractor.

6.3.16 Boiling chips.

6.3.17 Buchner funnel.

6.3.18 Filtration flask.

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6.3.19 Chromatography columns: 300mm x 10.5mm glass chromatographic column fitted with Teflon ™ stop cock.

6.3.20 Soxhlet apparatus, 500ml flask, all glass: Complete with glass extraction thimbles, heating mantle, and variable transformer for heat control.

6.3.21 Dean Stark water separator apparatus: With Teflon ™ stopcock. Must fit between Soxhlet extractor body and condenser.

6.3.22 Turbovap

NOTE: Reuse of glassware should be minimized to avoid the risk of using contaminated glassware. All glassware that is reused shall be scrupulously cleaned, applying the following procedure, as soon as possible after use. Rinse glassware with the last solvent used in it. Wash with hot water containing detergent. Rinse with copious amounts of tap water and several portions of distilled water. Drain dry. Rinse with high-purity acetone and hexane. After glassware is dry, store inverted or capped with aluminum foil in a clean environment. Do not bake reusable glassware after cleaning, as repeated baking may cause active sites on the glass surface that will irreversibly adsorb PCDDs/PCDFs.

7.0 REAGENTS

7.1 Column Chromatography Reagents

7.1.1 Alumina, neutral, 80/200 mesh (Super 1, Woelm®, or equivalent). Store in a sealed container at room temperature, in a desiccator, over self-indicating silica gel.

7.1.2 Alumina, acidic AG4, (Bio Rad Laboratories catalogue #132-1240) or equivalent - Soxhlet extract with methylene chloride for 24 hours and activate by heating in a foil covered glass container for 24 hours at 190°C. Store in a glass bottle sealed with a Teflon ™ lined screw cap.

7.1.3 Silica gel, high purity grade, type 60, 70-230 mesh – Soxhlet extract with methylene chloride for 24 hours, and activate by heating in a foil covered glass container for 24 hours at 190°C. Store in a glass bottle sealed with a Teflon ™ lined screw cap.

7.1.4 Silica gel impregnated with sodium hydroxide – Add one part (by weight) of 1 M NaOH solution to two parts (by weight) silica gel (extracted and activated), mix with a glass rod until free of lumps. Store in a glass bottle sealed with a Teflon™ lined screw cap.

7.1.5 Silica gel impregnated with 40 percent (by weight) sulfuric acid – Add two parts (by weight) concentrated sulfuric acid to three parts (by weight) silica gel (extracted and activated), mix with a glass rod until free of lumps. Store in a glass bottle sealed with a Teflon ™ lined screw cap.

7.1.6 Celite 545 ® (Supelco) , or equivalent.

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7.1.7 Charcoal carbon – Activated carbon, Carbopak C (Supelco) or equivalent. Prewash with methanol and dry in vacuo at 110°C.

NOTE: AX-21 (Anderson Development Company) carbon is no longer available.

7.2 Reagents

7.2.1 Sulfuric acid, H_2SO_4, concentrated, ACS grade, specific gravity 1.84.

7.2.2 Potassium Hydroxide. ACS Grade, Prepare a 20 percent (w/v) solution in distilled water.

7.2.3 Sodium chloride, NaCl, analytical reagent, 5 percent (w/v) in distilled water.

7.2.4 Potassium carbonate, K_2CO_3, anhydrous, analytical reagent.

7.3 Desiccating agent

7.3.1 Sodium sulfate (powder, anhydrous), Na_2SO_4 – Purify by heating at 400°C for 4 hours in a shallow tray, or by precleaning the sodium sulfate with methylene chloride.

7.4 Solvents

7.4.1 Methylene chloride, CH_2CL_2 – High purity, distilled in glass

7.4.2 Hexane, C_6H_{14} – High purity, distilled in glass

7.4.3 Methanol, CH_3OH – High purity, distilled in glass

7.4.4 Nonane, C_9H_{20} – High purity, distilled in glass

7.4.5 Toluene, $C_6H_5CH_3$ – High purity, distilled in glass

7.4.6 Cyclohexane, C_6H_{12} – High purity, distilled in glass

7.4.7 Acetone, CH_3COCH_3 – High purity, distilled in glass

7.5 High Resolution Concentration Calibration Solutions (Table 5) – Five nonane solutions containing unlabeled (totaling 17) and carbon-labeled (totaling 11) PCDDs and PCDFs at known concentrations are used to calibrate the instrument. The concentration ranges are homologue dependent, with the lowest values for the tetrachlorinated dioxin and furan and the highest values for the octachlorinated congeners. Store the concentration calibration solutions in 1 mL minivials at room temperature in the dark.

7.6 GC Column Performance Check Solution – This solution contains the first and last eluting isomers for each homologous series from tetra- through heptachlorinated congeners (Table 12). The solution also contains a series of other TCDD isomers for the purpose of documenting the chromatographic resolution.

7.7 Sample Fortification Solution – This nonane solution contains the nine internal standards at the nominal concentrations that are listed in Table 4. The solution contains at least one

carbon-labeled standard for each homologous series, and it is used to measure the concentrations of the native substances.

7.8 Recovery Standard Solution – This nonane solution contains two recovery standards, 13C12-1,2,3,4-TCDD and 13C12-1,2,3,7,8,9-HxCDD, at a nominal concentration of 50 pg/µL per compound. 20µL of this solution will be spiked into each sample extract before HRGC/HRMS analysis.

7.9 Matrix Spike Fortification solution – Solution used to prepare the MS and MSD samples. It contains all unlabeled analytes listed in Table 5 at concentrations corresponding to the HRCC 3. Addition of 1ml acetone solution to the sample gives concentration listed in Table 13.

8.0 PROCEDURE

8.1 Internal standard addition

8.1.1 Use a portion of 1 g to 1000 g (+/- 5 percent) of the sample to be analyzed. Typical sample size requirements for different matrices are given in Sec. 8.4 and in Table 1. Transfer the sample portion to a tared flask and determine its weight.

8.1.2 Except for adipose tissue, add an appropriate quantity of the sample fortification mixture (Sec. 7.7) to the sample. All samples should be spiked with the sample fortification mixture to give internal standard concentrations as indicated in Table 4. As an example, for 13C12-2,3,7,8-TCDD, a 10 g soil sample requires the addition of 1000 pg of 13C12-2,3,7,8-TCDD to give the required 100 ppt fortification level. The fish tissue sample (20 g) must be spiked with two times the amount of the internal standard solution, because half of the extract will be used to determine the lipid content (Sec. 4.7.1).

8.1.2.1 For the fortification of soil, sediment, fly ash, water, fish tissue, paper pulp and wet sludge samples, mix the sample fortification solution with 1.0 mL acetone to give a final concentration of 1000 pg/mL.

8.1.2.2 Do not dilute the nonane solution for the other matrices.

8.1.2.3 The fortification of adipose tissue is carried out at the time of homogenization (Sec. 8.3.2.3).

8.2 Extraction and Purification of Fish and Paper Pulp Samples

8.2.1 Add 60 g anhydrous sodium sulfate to a 20 g portion of a homogeneous fish sample (Sec. 4.3) and mix thoroughly with a stainless steel spatula. After breaking up any lumps, place the fish/sodium sulfate mixture in the Soxhlet apparatus on top of a glass wool plug. Add 250 mL methylene chloride or hexane/methylene chloride (1:1) to the Soxhlet apparatus and reflux for 16 hours. The solvent must cycle completely through the system five times per hour. Follow the same procedure for the partially dewatered paper pulp sample (using a 10 g sample, 30g of anhydrous sodium sulfate and 200 mL of toluene).

8.2.2 Transfer the fish extract from Sec. 8.2.1 to a 250mL volumetric flask and fill to the mark with methylene chloride. Mix well, then remove 125mL for the determination of the lipid content (Sec. 4.7.1). Transfer the remaining 125mL of the extract, plus two 15 mL hexane/methylene chloride rinses of the volumetric flask, to a KD apparatus equipped with a Snyder column. Quantitatively transfer all of the paper pulp extract to a KD apparatus equipped with a Snyder column and vacuum.

8.2.3 Add a Teflon ™, or equivalent, boiling chip. Concentrate the extract in a water bath to an apparent volume of 10mL. Remove the apparatus from the water bath and allow to cool for 5 minutes.

8.2.4 Add 50mL hexane and a new boiling chip to the KD flask. Concentrate in a water bath to an apparent volume of 5mL. Remove the apparatus from the water bath and allow to cool for 5 minutes.

NOTE: The methylene chloride must have been completely removed before proceeding with the next step.

8.2.5 Remove and invert the Snyder column and rinse it into the KD apparatus with two 1mL portions of hexane. Decant the contents of the KD apparatus and concentrator tube into a 125mL separatory funnel. Rinse the KD apparatus with two additional 5mL portions of hexane and add the rinses to the funnel. Proceed with the cleanup according to the instructions starting in Sec. 8.6.1.1, but omit the procedures described in Sec. 8.6.1.2 and 8.6.1.3.

8.3 Extraction and Purification of Human Adipose Tissue

8.3.1 Human adipose tissue samples must be stored at a temperature of -20°C or lower from the time of collection until the time of analysis. The use of chlorinated materials during the collection of the samples must be avoided. Samples are handled with stainless steel forceps, spatulas, or scissors. All sample bottles (glass) are cleaned as specified in the note at the end of Sec. 6.3. Teflon™ lined caps should be used.

NOTE: The specified storage temperature of -20°C is the maximum storage temperature permissible for adipose tissue samples. Lower storage temperatures are recommended.

8.3.2 Adipose Tissue Extraction

8.3.2.1 Weigh, to the nearest 0.01 g, a 10 g portion of a frozen adipose tissue sample into a culture tube (2.2 x 15 cm).

NOTE: The sample size may be smaller, depending on availability. In such a situation, the analyst is required to adjust the volume of the internal standard solution added to the sample to meet the fortification level stipulated in Table 4.

8.3.2.2 Allow the adipose tissue specimen to reach room temperature (up to 2 hours).

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8.3.2.3 Add 10mL methylene chloride and 1mL of the sample fortification solution. Homogenize the mixture for approximately 1 minute with a tissue homogenizer.

8.3.2.4 Allow the mixture to separate, then remove the methylene chloride extract from the residual solid material with a disposable pipet. Percolate the methylene chloride through a filter funnel containing a clean glass wool plug and 10g anhydrous sodium sulfate. Collect the dried extract in a graduated 100mL volumetric flask.

8.3.2.5 Add a second 10mL portion of methylene chloride to the sample and homogenize for 1 minute. Decant the solvent, dry it, and transfer it to the 100 mL volumetric flask (Sec. 8.3.2.4).

8.3.2.6 Rinse the culture tube with at least two additional portions of methylene chloride (10mL each), and transfer the entire contents to the filter funnel containing the anhydrous sodium sulfate. Rinse the filter funnel and the anhydrous sodium sulfate contents with additional methylene chloride (20 to 40mL) into the 100mL flask. Discard the sodium sulfate.

8.3.2.7 Adjust the volume to the 100mL mark with methylene chloride.

8.3.3 Adipose Tissue Lipid Content Determination

8.3.3.1 Preweigh a clean 1 dram (or metric equivalent) glass vial to the nearest 0.0001 g on an analytical balance tared to zero.

8.3.3.2 Accurately transfer 1.0mL of the final extract (100mL) from Sec. 8.3.2.6 to the vial. Reduce the volume of the extract on a water bath (50-60°C) by a gentle stream of purified nitrogen until an oily residue remains. Nitrogen blowdown is continued until a constant weight is achieved.

NOTE: When the sample size of the adipose tissue is smaller than 10g, then the analyst may use a larger portion (up to 10 percent) of the extract defined in Sec. 8.3.2.7 for the lipid determination.

8.3.3.3 Accurately weigh the vial with the residue to the nearest 0.0001g and calculate the weight of the lipid present in the vial based on the difference of the weights.

8.3.3.4 Calculate the percent lipid content of the original sample to the nearest 0.1 percent as shown below:

$$\text{Lipid content, LC (\%)} = \frac{W(lr) \times V(ext)}{W(at) \times V(al)} \times 100$$

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where:

W(lr) = weight of the lipid residue to the nearest 0.0001g calculated from Sec. 8.3.3.3

V(ext) = total volume (100 mL) of the extract in mL from Sec. 8.3.2.6

W(at) = weight of the original adipose tissue sample to the nearest 0.01g from Sec. 8.3.2.1

V(al) = volume of the aliquot of the final extract in mL used for the quantitative measure of the lipid residue (1.0mL) from Sec. 8.3.3.2

8.3.3.5 Record the lipid residue measured in Sec. 8.3.3.3 and the percent lipid content from Sec 8.3.3.4.

8.3.4 Adipose Tissue Extract Concentration

8.3.4.1 Quantitatively transfer the remaining extract from Sec. 8.3.3.2 (99.0mL) to a 500mL Erlenmeyer flask. Rinse the volumetric flask with 20 to 30mL of additional methylene chloride to ensure quantitative transfer.

8.3.4.2 Concentrate the extract on a KD apparatus with vacuum at 50-60°C until an oily residue remains.

8.3.5 Adipose Tissue Extract Cleanup

8.3.5.1 Add 200mL hexane to the lipid residue in the 500mL Erlenmeyer flask and swirl the flask to dissolve the residue.

8.3.5.2 Slowly add, with stirring, 100g of 40 percent (w/w) sulfuric acid-impregnated silica gel. Stir with a magnetic stirrer for two hours at room temperature.

8.3.5.3 Allow the solid phase to settle, and decant the liquid through a filter funnel containing 10g anhydrous sodium sulfate on a glass wool plug, into another 500mL Erlenmeyer flask.

8.3.5.4 Rinse the solid phase with two 50mL portions of hexane. Stir each rinse for 15 minutes, decant, and dry as described under Sec. 8.3.5.3. Combine the hexane extracts from Sec. 8.3.5.3 with the rinses.

8.3.5.5 Rinse the sodium sulfate in the filter funnel with an additional 25mL hexane and combine this rinse with the hexane extracts from Sec. 8.3.5.4.

8.3.5.6 Prepare an acidic silica column as follows: Pack a 2 cm x 10 cm chromatographic column with a glass wool plug, add approximately 20mL hexane, add 1g silica gel and allow to settle, then add 4 g of 40 percent (w/w) sulfuric acid-impregnated silica gel and allow to settle. Elute the excess hexane from the column until the solvent level reaches the top of the chromatographic packing. Verify that the column does not have any air bubbles and channels.

8.3.5.7 Quantitatively transfer the hexane extract from the Erlenmeyer flask (Secs. 8.3.5.3 through 8.3.5.5) to the silica gel column reservoir. Allow the hexane extract to percolate through the column and collect the eluate in a 500mL KD apparatus.

8.3.5.8 Complete the elution by percolating 50mL hexane through the column into the KD apparatus. Concentrate the eluate on a steam bath to approximately 5mL. Use nitrogen blowdown to bring the final volume to about 100 µL.

NOTE: If the silica gel impregnated with 40 percent sulfuric acid is highly discolored throughout the length of the adsorbent bed, the cleaning procedure must be repeated beginning with Sec. 8.3.5.1.

8.3.5.9 The extract is ready for the column cleanups described in Secs. 8.6.2 through 8.6.3.8.

8.4 Extraction and Purification of Environmental and Waste Samples

8.4.1 Sludge/Wet Fuel Oil

8.4.1.1 Extract aqueous sludge or wet fuel oil samples by refluxing a sample (e.g., 2g) with 50mL toluene in a 125mL flask fitted with a Dean-Stark water separator. Continue refluxing the sample until all the water is removed.

NOTE: If the sludge or fuel oil sample dissolves in toluene, treat it according to the instructions in Sec. 8.4.2 below. If the labeled sludge sample originates from pulp (paper mills), treat it according to the instructions starting in Sec. 8.2, but without the addition of sodium sulfate.

8.4.1.2 Cool the sample, filter the toluene extract through a glass fiber filter, or equivalent, into a 100mL round bottom flask.

8.4.1.3 Rinse the filter with 10mL toluene and combine the extract with the rinse.

8.4.1.4 Concentrate the combined solutions to near dryness on a KD apparatus with vacuum at 50-60°C. Use of an inert gas to concentrate the extract is also permitted. Proceed with Sec. 8.4.4.

8.4.2 Still Bottom/Oil

8.4.2.1 Extract still bottom or oil samples by mixing a sample portion (e.g., 1.0g) with 10mL toluene in a small beaker and filtering the solution through a glass fiber filter (or equivalent) into a 50mL round bottom flask. Rinse the beaker and filter with 10mL toluene.

8.4.2.2 Concentrate the combined toluene solutions to near dryness on a KD apparatus with vacuum at 50-60°C. Proceed with Sec. 8.4.4.

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8.4.3 Fly Ash

NOTE: Because of the tendency of fly ash to "fly", all handling steps should be performed in a hood in order to minimize contamination.

8.4.3.1 Weigh about 10g fly ash to two decimal places and transfer to an extraction jar. Add 1mL sample fortification solution (Sec. 7.7), in acetone, to the sample. Add 150mL of 1 M HCl to the fly ash sample. Seal the jar with the Teflon™ lined screw cap and shake for 3 hours at room temperature.

8.4.3.2 Rinse a glass fiber filter with toluene, and filter the sample through the filter paper, placed in a Buchner funnel, into a 1L flask. Wash the fly ash cake with approximately 500mL organic-free reagent water and dry the filter cake overnight at room temperature in a desiccator.

8.4.3.3 Add 10g anhydrous powdered sodium sulfate, mix thoroughly, let sit in a closed container for one hour, mix again, let sit for another hour, and mix again.

8.4.3.4 Place the sample and the filter paper into an extraction thimble, and extract in a Soxhlet extraction apparatus charged with 200mL toluene for 16 hours using a five cycle/hour schedule.

NOTE: As an option, a Soxhlet/Dean Stark extractor system may be used, with toluene as the solvent. No sodium sulfate is added when using this option.

8.4.3.5 Cool and filter the toluene extract through a glass fiber filter into a 500mL round bottom flask. Rinse the filter with 10mL toluene. Add the rinse to the extract and concentrate the combined toluene solutions to near dryness on a KD apparatus with vacuum at 50-60°C. Proceed with Sec. 8.4.4.

8.4.4 Transfer the concentrate to a 125mL separatory funnel using 15mL hexane. Rinse the flask with two 5mL portions of hexane and add the rinses to the funnel. Shake the combined solutions in the separatory funnel for two minutes with 50mL of 5 percent sodium chloride solution, discard the aqueous layer, and proceed with Sec. 8.6.

8.4.5 Aqueous samples

8.4.5.1 Allow the sample to come to ambient temperature, then measure 1L of sample using a measuring cylinder. Transfer the sample to a separatory funnel and add 1mL of the sample fortification solution (section 7.7).

8.4.5.2 When the sample is judged to contain 1 percent or more solids, the sample must be filtered through a glass fiber filter that has been rinsed with toluene. If the suspended solids content is too great to filter

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through the 0.45um filter, centrifuge the sample, decant, and then filter the aqueous phase.

NOTE: Paper mill effluent samples normally contain 0.02%-0.2% solids, and would not require filtration. However for optimum analytical results, all paper mill effluent samples should be filtered, the isolated solids and filtrate extracted separately, and the extracts recombined.

8.4.5.3 Combine the solids from the centrifuge bottle(s) with the particulates on the filter and with the filter itself and proceed with the Soxhlet extraction as specified in Secs. 8.4.6.1 through 8.4.6.4. Remove and invert the Snyder column and rinse it down into the KD apparatus with two 1 mL portions of hexane.

8.4.5.4 Pour the aqueous filtrate into a 2L separatory funnel. Add 60mL methylene chloride to the sample bottle, seal and shake for 30 seconds to rinse the inner surface. Transfer the solvent to the separatory funnel and extract the sample by shaking the funnel for two minutes with periodic venting.

8.4.5.5 Allow the organic layer to separate from the water phase for a minimum of 10 minutes. If the emulsion interface between layers is more than one third the volume of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation (e.g., glass stirring rod).

8.4.5.6 Collect the methylene chloride into a KD apparatus (mounted with a 10mL concentrator tube) by passing the sample extracts through a filter funnel packed with a glass wool plug and 5g anhydrous sodium sulfate.

8.4.5.7 Repeat the extraction twice with fresh 60mL portions of methylene chloride. After the third extraction, rinse the sodium sulfate with an additional 30mL methylene chloride to ensure quantitative transfer. Combine all extracts and the rinse in the KD apparatus.

NOTE: A continuous liquid-liquid extractor may be used in place of a separatory funnel when experience with a sample from a given source indicates that a serious emulsion problem will result or an emulsion is encountered when using a separatory funnel. Add 60mL methylene chloride to the sample bottle, seal, and shake for 30 seconds to rinse the inner surface. Transfer the solvent to the extractor. Repeat the rinse of the sample bottle with an additional 50 to 100mL portion of methylene chloride and add the rinse to the extractor. Add 200 to 500mL methylene chloride to the distilling flask, add sufficient organic-free reagent water to ensure proper operation, and extract for 18 hours. Allow to cool, then detach the distilling flask. Dry and concentrate the extract as described in Secs. 8.4.5.6 and 8.4.5.8 through 8.4.5.10. Proceed with Sec. 8.4.5.11.

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8.4.5.8 Attach a Snyder column and concentrate the extract on a water bath until the apparent volume of the liquid is 5mL. Remove the KD apparatus and allow it to drain and cool for at least 10 minutes.

8.4.5.9 Remove the Snyder column, add 50mL hexane, add the concentrate obtained from the Soxhlet extraction of the suspended solids (Sec. 8.4.5.3), if applicable, re-attach the Snyder column, and concentrate to approximately 5mL. Add a new boiling chip to the KD apparatus before proceeding with the second concentration step.

8.4.5.10 Rinse the flask and the lower joint with two 5mL portions of hexane and combine the rinses with the extract to give a final volume of about 15mL.

8.4.6 Soil/Sediment

8.4.6.1 Add 10g anhydrous sodium sulfate to the sample portion (e.g., 10 g) and mix thoroughly with a stainless steel spatula. After breaking up any lumps, place the soil/sodium sulfate mixture in the Soxhlet apparatus on top of a glass wool plug (the use of an extraction thimble is optional).

NOTE: As an option, a Soxhlet/Dean Stark extractor system may be used, with toluene as the solvent. No sodium sulfate is added when using this option.

8.4.6.2 Add 200 to 250mL toluene to the Soxhlet apparatus and reflux for 16 hours. The solvent must cycle completely through the system five times per hour.

NOTE: If the dried sample is not of free flowing consistency, more sodium sulfate must be added.

8.4.6.3 Cool and filter the extract through a glass fiber filter into a 500mL round bottom flask for evaporation of the toluene. Rinse the filter with 10mL of toluene, and concentrate the combined fractions to near dryness on a KD apparatus with vacuum at 50-60°C. Remove the flask from the water bath and allow to cool for 5 minutes.

8.4.6.4 Transfer the residue to a 125mL separatory funnel, using 15mL of hexane. Rinse the flask with two additional portions of hexane, and add the rinses to the funnel. Proceed with Sec. 8.6.

8.5 Collection, handling, analysis, and reporting of wipe tests performed within the laboratory – This procedure is designed for the evaluation of potential contamination in the blanks by 2,3,7,8-substituted PCDD/PCDF congeners of the working areas inside the laboratory.

8.5.1 Perform the wipe tests on surface areas of two inches by one foot with a glass fiber paper saturated with distilled in glass acetone using a pair of clean stainless steel forceps. Use one wiper for each of the designated areas. Combine the wipes

to one composite sample in an extraction jar containing 200mL acetone. Place an equal number of unused wipes in a jar containing 200mL acetone for use as a control. Add 1.0mL of the sample fortification solution to each jar containing used or unused wipers (Sec. 7.7).

8.5.1.1 Close the jar containing the wipers and the acetone and extract for 20 minutes using a wrist action shaker. Transfer the extract into a KD apparatus fitted with a concentration tube and a three ball Snyder column. Add two Teflon ™ or Carborundum ™ boiling chips and concentrate the extract to an apparent volume of 1.0mL on a steam bath. Rinse the Snyder column and the KD assembly with two 1mL portions of hexane into the concentrator tube, and concentrate its contents to near dryness with a gentle stream of nitrogen. Add 1.0mL hexane to the concentrator tube and swirl the solvent on the walls.

8.5.1.2 Prepare a neutral alumina column as described in Sec 8.6.2.2 and follow the steps outlined in Secs. 8.6.2.3 through 8.6.2.5.

8.5.2 Concentrate the contents of the vial to dryness. Add 20 µL of the recovery standard solution (Sec. 7.8). Inject 2 µL of each extract (wipe and control) onto a capillary column and analyze for 2,3,7,8-substituted PCDDs/PCDFs as specified in the analytical method in Sec. 8.11. Perform calculations according to Sec 9.0

8.5.3 Report the presence of 2,3,7,8-susbtituted PCDDs and PCDFs as a quantity (pg or ng) per wipe test experiment (WTE). Under the conditions out-lined in this analytical protocol, a lower limit of calibration of 10 pg/WTE is expected for 2,3,7,8-TCDD. A positive response for the blank (control) is defined as a signal in the TCDD retention time window at any of the masses monitored which is equivalent to or above 3 pg of 2,3,7,8-TCDD per WTE. For other congeners, use the multiplication factors listed in Table 1, footnote (a). Also, report the recoveries of the internal standards during the simplified cleanup procedure.

8.5.4 At a minimum, wipe tests should be performed when there is evidence of contamination in the method blanks.

8.5.5 An upper limit of 25 pg per TCDD isomer and per wipe test experiment is allowed. This value corresponds to 2.5 times the lower calibration limit of the analytical method.

8.6 Cleanup

8.6.1 Partition

8.6.1.1 Partition the hexane extract against 10mL of concentrated sulfuric acid. Shake for two minutes. Remove and discard the sulfuric acid layer (bottom). Repeat the acid washing until no color is visible in the acid layer (perform a maximum of four acid washings).

8.6.1.2 Omit this step for the fish sample extract. Partition the extract against 10mL of 5 percent (w/v) aqueous sodium chloride. Shake for two minutes. Remove and discard the aqueous layer (bottom).

8.6.1.3 Omit this step for the fish sample extract. Partition the extract against 10mL of 20 percent (w/v) aqueous potassium hydroxide (KOH). Shake for two minutes. Remove and discard the aqueous layer (bottom). Repeat the base washing until no color is visible in the bottom layer (perform a maximum of four base washings). Strong base (KOH) is known to degrade certain PCDDs/PCDFs, so contact time must be minimized.

8.6.1.4 Partition the extract against 10mL of 5 percent (w/v) aqueous sodium chloride. Shake for two minutes. Remove and discard the aqueous layer (bottom). Dry the extract by pouring it through a filter funnel containing anhydrous sodium sulfate on a glass wool plug, and collect it in a 50mL round bottom flask. Rinse the funnel with the sodium sulfate with two 15mL portions of hexane, add the rinses to the 50mL flask, and concentrate the hexane solution to near dryness on a KD apparatus with vacuum at 50-60°C, making sure all traces of toluene (when applicable) are removed. (Use of blowdown with an inert gas to concentrate the extract is also permitted.)

8.6.2 Silica/Alumina Column Cleanup

8.6.2.1 Pack a gravity column (glass, 30 cm x 10.5 mm), fitted with a Teflon™ stopcock, with silica gel as follows:

Insert a glass wool plug into the bottom of the column. Place 1g silica gel in the column and tap the column gently to settle the silica gel. Add 2g sodium hydroxide-impregnated silica gel, 4g sulfuric acid-impregnated silica gel, and 2g silica gel. Tap the column gently after each addition. A small positive pressure (5psi) of clean nitrogen may be used if needed. Elute with 10mL hexane and close the stopcock just before exposure of the top layer of silica gel to air. Discard the eluate. Check the column for channeling. If channeling is observed, discard the column. Do not tap the wetted column.

8.6.2.2 Pack a gravity column (glass, 300 mm x 10.5 mm), fitted with a Teflon™ stopcock, with alumina as follows:

Insert a glass wool plug into the bottom of the column. Add a 4g layer of sodium sulfate. Add a 4g layer of Woelm(R) Super 1 neutral alumina. Tap the top of the column gently. Woelm(R) Super 1 neutral alumina need not be activated or cleaned before use, but it should be stored in a sealed desiccator. Add a 4 g layer of anhydrous sodium sulfate to cover the alumina. Elute with 10mL hexane and close the stopcock just before exposure of the sodium sulfate layer to air. Discard the eluate. Check the column for

channeling. If channeling is observed, discard the column. Do not tap a wetted column.

NOTE: Optionally, acidic alumina (Sec. 7.1.2) can be used in place of neutral alumina.

8.6.2.3 Dissolve the residue from Sec. 8.6.1.4 in 2 mL hexane and apply the hexane solution to the top of the silica gel column. Rinse the flask with enough hexane (3-4 mL) to complete the quantitative transfer of the sample to the surface of the silica gel.

8.6.2.4 Connect the silica gel column and the alumina column in series. Elute the silica gel column with 40mL of hexane on to the alumina column. Discard the silica gel column.

8.6.2.5 Add 20mL hexane to the alumina column and elute until the hexane level is just below the top of the sodium sulfate. Do not discard the eluted hexane, but collect it in a separate flask and store it for later use, as it may be useful in determining where the labeled analytes are being lost if recoveries are not satisfactory.

8.6.2.6 Add 15mL of 60 percent methylene chloride in hexane (v/v) to the alumina column and collect the eluate in a conical shaped (15 mL) concentration tube. With a carefully regulated stream of nitrogen, concentrate the 60 percent methylene chloride/hexane fraction to about 2 mL.

8.6.3 Carbon Column Cleanup

8.6.3.1 Prepare a Carbopak C/Celite 545® column as follows: Thoroughly mix 5.4 g active carbon Carbopak C and 62.0 g Celite 545® to produce an 8 percent (w/w) mixture. Activate the mixture at 130°C for a minimum of 6 hours and store it in a desiccator.

8.6.3.2 Remove the stop cock from the alumina column and use this column for carbon column cleanup. Insert a glass wool plug at one end, then pack the column with enough Celite 545® to form a 1 cm plug, add 1 g of the Carbopak C/Celite 545® mixture, top with additional Celite 545® (enough for a 1 cm plug), and cap the packing with another glass wool plug.

NOTE: Each new batch of Carbopak C/Celite 545® must be checked as follows: Add 50µL of the continuing calibration solution to 950µL hexane. Take this solution through the carbon column cleanup step, concentrate to 50µL and analyze. If the recovery of any of the analytes is <80 percent, discard this batch of Carbopak C/Celite 545®.

8.6.3.3 Rinse the Carbopak C/Celite 545® column with 5mL of toluene, followed by 2mL of 75:20:5 (v/v) methylene chloride/methanol/toluene, 1mL of 1:1 (v/v) cyclohexane/methylene chloride, and 5mL hexane. The flow rate should be less than

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0.5mL/min. Discard the rinses. While the column is still wet with hexane, add the sample concentrate (Sec. 8.6.2.6) to the top of the column. Rinse the concentrator tube (which contained the sample concentrate) twice with 1mL hexane, and add the rinses to the top of the column.

8.6.3.4 Elute the column sequentially with two 2mL portions of hexane, 2mL cyclohexane/methylene chloride (50:50, v/v), and 2mL methylene chloride/methanol/toluene (75:20:5, v/v). Combine these eluates; this combined fraction may be used as a check on column efficiency.

8.6.3.5 Turn the column upside down and elute the PCDD/PCDF fraction with 20mL toluene. Verify that no carbon fines are present in the eluate. If carbon fines are present in the eluate, filter the eluate through a glass fiber filter (0.45um) and rinse the filter with 2mL toluene. Add the rinse to the eluate.

8.6.3.6 Concentrate the toluene fraction to about 1mL on a KD apparatus with vacuum by using a water bath at 50-60°C. Carefully transfer the concentrate into a 1mL minivial and, again at elevated temperature (50-60°C), reduce the volume to about 100µL using a stream of nitrogen and a sand bath, or by using Turbovap. Rinse the KD apparatus three times with 300 µL of a solution of 1 percent toluene in methylene chloride, and add the rinses to the concentrate. Further concentrate to dryness. Store the sample at room temperature in the dark. Before analysis, add 20 µL of the recovery standard solution (Sec. 7.8) to the sample extract.

8.7 Chromatographic/Mass Spectrometric Conditions and Data Acquisition Parameters

8.7.1 The following GC operating conditions are used and adjusted as needed to meet the relative retention time specification in Table 11.

Injector temp:	300°C
Interface temp:	300°C
Initial temp & time:	150°C, 5 min.
Temp Program:	150° - 215°C at 35°C/min.
	215°C for 5 min.
	215° - 230°C at 1.5°C/min.
	230°C for 6 min.
	230 - 315°C at 7°C/min.
	315°C for 6 min.

NOTE: All portions of the column which connect the GC to the ion source shall remain at the interface temperature specified above during analysis, to preclude condensation of less volatile compounds.

8.7.2 Mass Spectrometer (MS) resolution: Obtain a selected ion current profile (SICP) of each analyte in Table 3 at the two exact masses specified in Table 6 and at ≥

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10,000 resolving power by injecting an authentic standard of the PCDDs and PCDFs either singly or as part of a mixture in which there is no interference between closely eluted components

8.7.2.1 The recommended mass spectrometer tuning conditions are based on the groups of monitored ions shown in Table 6. By using a PFK molecular leak, tune the instrument to meet the minimum required resolving power of 10,000 (10 percent valley) at m/z 304.9824 (PFK) or any other reference signal close to m/z 303.9016 (from TCDF). By using peak matching conditions and the aforementioned PFK reference peak, verify that the exact mass of m/z 380.9760 (PFK) is within 5ppm of the required value. The mass resolution check must be performed at the beginning and end of the 12-hour shift.

8.7.2.2 The analysis time for PCDDs and PCDFs may exceed the long-term mass stability of the mass spectrometer. Because the instrument is operated in the high-resolution mode, mass drifts of a few ppm (e.g., 5 ppm in mass) can have serious adverse effects on the instrument performance. Therefore, a mass-drift correction is mandatory. A lock-mass ion from the reference compound (PFK) is used for tuning the mass spectrometer. The lock-mass ion is dependent on the masses of the ions monitored within each descriptor, as shown in Table 6. The level of the reference compound (PFK) metered into the ion chamber during HRGC/HRMS analyses should be adjusted so that the amplitude of the most intense selected lock-mass ion signal (regardless of the descriptor number) does not exceed 10 percent of the full-scale deflection for a given set of detector parameters. Under those conditions, sensitivity changes that might occur during the analysis can be more effectively monitored.

NOTE: Excessive PFK (or any other reference substance) may cause noise problems and contamination of the ion source resulting in an increase in time lost in cleaning the source.

8.7.3 The mass spectrometer must be operated in a selected ion monitoring (SIM) mode with a total cycle time (including voltage reset time) of one second or less. At a minimum, the ions listed in Table 6 for each of the five SIM descriptors must be monitored. The same set of ions must be maintained for both calibration and sample extract analyses.

8.8 Calibration – Inject the HRCC-1 calibration solution (Table 5) and check for ion abundance ratios, minimum levels, signal-to-noise ratios, and absolute retention times.

8.8.1 Measure the SICP areas for each analyte and compute the ion abundance ratios specified in Table 7. Compare the computed ratio to the theoretical ratio given in Table 7.

8.8.1.1 The groups of m/z's to be monitored are shown in Table 6. Each group or descriptor shall be monitored in succession as a function of

GC retention times to ensure that all PCDDs and PCDFs are detected. The theoretical abundance ratios for the m/z's are given in Table 7, along with the control limits of each ratio.

8.8.1.2 The mass spectrometer shall be operated in a mass drift correction mode, using perfluorokerosene (PFK) to provide lock masses. The lock mass for each group of m/z's is shown in Table 6. Each lock mass shall be monitored and shall not vary by more than ±10 percent throughout its respective retention time window. Variations of the lock mass by more than 10 percent indicate the presence of coeluting interferences that may significantly reduce the sensitivity of the mass spectrometer. Reinjection of another aliquot of the sample extract will not resolve the problem. Additional cleanup of the extract may be required to remove the interferences.

8.8.1.3 Verify that the HRGC/HRMS instrument meets the minimum levels in Table 5. The peaks representing both unlabeled and labeled analytes in the calibration standards must have a signal-to-noise (S/N) greater than or equal to 10; otherwise, the mass spectrometer shall be adjusted and this test repeated until the minimum levels in Table 5 are met.

8.8.2 Compute the percent valley between the GC peaks that elute most closely to the 2,3,7,8-TCDD and TCDF isomers, on their respective columns, per Figure 4.

8.8.2.1 Verify that the height of the valley between the most closely eluted isomers and the 2,3,7,8-isomers is less than 25 percent. If the valley exceeds 25 percent, adjust the analytical conditions and repeat the test or replace the GC column and recalibrate.

8.8.3 Calibration with isotope dilution: Isotope dilution is used for the fifteen 2,3,7,8-substituted PCDDs and PCDFs with internal standards added to the samples prior to the extraction. The reference compound for each unlabeled compound is shown in Table 2.

8.9 Initial Calibration – Initial calibration is required before any samples are analyzed for PCDDs and PCDFs. Initial calibration is also required if any routine calibration (Sec. 8.10) does not meet the required criteria listed in Secs. 8.10.1.3 and 8.10.1.4.

8.9.1 All five high resolution concentration calibration solutions listed in Table 5 must be used for the initial calibration.

8.9.1.1 Tune the instrument with PFK as described in Sec. 8.7.2.

8.9.1.2 Inject 2 µL of the GC column performance check solution (Sec. 7.6) and acquire SIM mass spectral data. The total cycle time must be ≤ 1 second. No further analysis should be performed until it is demonstrated and documented that the criterion in Sec. 8.8.1 and 8.8.2 was met.

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8.9.1.3 Using the same GC and MS conditions that produced acceptable results with the column performance check solution, analyze a 2 µL portion of each of the five concentration solutions once with the following mass spectrometer operating parameters.

- The ratio of integrated ion current for the ions listed in Table 7 (homologous series quantitation ions) must be within the control limits set for each homologous series for all unlabeled calibration standards in Table 5.
- The ratio of integrated ion current for the ions belonging to the carbon-labeled internal and recovery standards (Table 5) must be within the control limits stipulated in Table 7.
- For each selected ion current profile (SICP) and for each GC signal corresponding to the elution of a target analyte and of its labeled standards, the signal-to-noise ratio (S/N) must be better than or equal to 2.5. Measurement of S/N is required for any GC peak that has an apparent S/N of less than 5:1. The result of the calculation must appear on the SICP above the peak in question.

8.9.2 Calculate the relative response factors (RRFn) for the 17 unlabeled target analytes (2,3,7,8-specific PCDD/PCDFs, OCDD and OCDF) relative to their appropriate internal standards, and the RRFis for the 10 labeled standards according to the following formulas:

$$RRFn = \frac{An \times Qis}{Ais \times Qn} \qquad RRFis = \frac{Ais \times Qrs}{Ars \times Qis}$$

where:

An = Sum of integrated ion abundance of the quantitation ions for unlabeled PCDDs/PCDFs (Table 6).
Ars = Sum of integrated ion abundance of the quantitation ions of the appropriate recovery standard (Table 6).
Ais = Sum of integrated ion abundance of the quantitation ion of the appropriate internal standard.
Qn = quantity of the unlabeled PCDD/PCDF analyte injected (pg).
Qis = quantity of the appropriate internal standard injected (pg).
Qrs = quantity of the appropriate recovery standard injected (pg).

The RRFn and RRFis are dimensionless quantities; therefore, the units used to express the Qn, Qis, and Qrs must be the same.

8.9.2.1 Calculate the mean RRF and percent relative standard deviations (%RSDs) of the five RRFs (HRCC1 to HRCC5) for each unlabeled PCDD/PCDF and labeled PCDD/PCDF and internal standards present in all five concentration calibration solutions:

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8.9.2.2 The RRFs calculated for the unlabeled PCDDs/PCDFs are relative to the appropriate labeled standard, and the RRFs for the labeled PCDD/PCDFs are relative to the internal standards.

8.9.2.3 The relative response factors to be used for the determination of the concentration of total isomers in a homologous series (Table 9) are calculated as follows:

- For congeners that belong to a homologous series containing only one isomer (e.g., OCDD and OCDF) or only one 2,3,7,8-substituted isomer (Table 8; TCDD,PeCDD, HpCDD, and TCDF), the mean RRF used will be the same as the mean RRF determined in Sec. 8.9.2.1
- For congeners that belong to a homologous series containing more than one 2,3,7,8-substituted isomer (Table 8), the mean RRF used for those homologous series will be the mean RRFs calculated for all individual 2,3,7,8-substituted congeners.

$$\%RSD = \frac{\text{Standard Deviation}}{\text{Mean RRF}} \times 100$$

8.9.2.4 The %RSD is calculated for the unlabeled and labeled analytes only. The %RSD (HRCC1-HRCC5) for the unlabeled PCDDs/PCDFs and the labeled PCDD/PCDFs must not exceed 20% & 30% respectively.

8.10 Continuing Calibration -The continuing calibration consists of two parts: analysis of the homologue window defining mix containing isomer specific TCDDs to verify that the appropriate descriptor switching times are used and % Valley criteria is met; and analysis of the HRCC3 solution to demonstrate DB-5 GC column resolution and for use as a quantitation standard. At the beginning of a 12-hour analytical period, a window defining mix is analyzed and if all criteria are met, the HRCC3 solution is analyzed.

8.10.1 Continuing Calibration Procedure

8.10.1.1 Inject a 2µL aliquot of the continuing calibration solution (HRCC3) into the GC/MS. The identical GC/MS conditions used for the analysis of the initial calibration solutions <u>must</u> be used for the continuing calibration solution.

8.10.1.2 Calculate the relative response factors for the 17 unlabeled target analytes relative to their appropriate labeled standards (RRFn) and the response factors for the 9 internal PCDD/PCDFs relative to the appropriate recovery standard (RRFis), according to the following formulas:

$$RRFn = \frac{An \times Qis}{Ais \times Qn} \qquad RRFis = \frac{Ais \times Qrs}{Ars \times Qis}$$

An, Ais, Qis, Qn Ars, and Qrs are defined in Sec.8.9.2.

The RRFn and RRFis are dimensionless quantities; therefore, the units used to express the Qn, Qis, and Qrs must be the same.

8.10.1.3 The Continuing Calibration Check must meet the following criteria:

- The measured RRFs for the unlabeled standards obtained during the routine calibration runs must be within ± 20 percent of the mean values established during the initial calibration.
- The measured RRFs for the labeled standards obtained during the routine calibration runs must be within ± 30 percent of the mean values established during the initial calibration.
- The ion abundance ratios (Table 7) must be within the allowed control limits.

8.10.1.4 Analyze the Continuing Calibration Check Solution at the end of the 12-hour sequence. It also must meet the criteria specified in Sec. 8.10.1.3.

8.10.1.5 If any one of the criteria in Secs. 8.10.1.3(a), (b)and (c) is not satisfied, repeat one more time. If these criteria are still not satisfied, the entire routine calibration process must be reviewed. It is realized that it may not always be possible to achieve all RF criteria. For example, it has occurred that the RF criteria for $_{13}C_{12}$-HpCDD and $_{13}C_{12}$-OCDD were not met, however, the RF values for the corresponding unlabeled compounds were routinely within the criteria established in the method. In these cases, 24 of the 26 RF parameters have met the QC criteria, and the data quality for the unlabeled HpCDD and OCDD values were not compromised as a result of the calibration event. In these situations, the analyst must assess the effect on overall data quality as required for the data quality objectives and decide on appropriate action. Corrective action would be in order, for example, if the compounds for which the RF criteria were not met included both the unlabeled and the corresponding internal standard compounds. If the ion abundance ration criterion is not satisfied, refer to Section 8.9.1.3.

8.11 GC/MS Analysis – Remove the extract of the sample or blank from storage. Gently swirl the solvent on the lower portion of the vial to ensure complete dissolution of the PCDDs/PCDFs. Add 20µL of the recovery standard solution (Table 4).

8.11.1 Analyze the extract by GC/MS, and monitor all of the ions listed in Table 6. The same MS parameters used to analyze the calibration solutions shall be used for the sample extracts.

8.12 Identification Criteria - For a gas chromatographic peak to be identified as a PCDD or PCDF, it must meet all of the following criteria.

8.12.1 Relative Retention Times - In order to make a positive identification of the 2,3,7,8-substituted isomers for which an isotopically labeled internal or recovery standard is present in the sample extract, the absolute retention time at the maximum peak height of the analyte must be within -1 to +3 seconds of the retention time of the corresponding isotopically labeled standard.

8.12.1.1 In order to make a positive identification of the 2,3,7,8-substituted isomers for which an isotopically labeled standard is <u>not</u> present in the sample extract, the relative retention time (RRT) of the analyte must be within 0.005 RRT units of the RRT established by the continuing calibration. Identification of OCDF is based on its retention time relative to 13C12-OCDD as determined from the daily continuing calibration results. The RRT is calculated as follows:

$$RRT = \frac{\text{Retention time of analyte}}{\text{Retention time of corresponding labeled compound}}$$

8.12.1.2 For non-2,3,7,8-substituted compounds (tetra through octa) the retention time must be within the corresponding homologous retention time windows established by the column performance check solution.

8.12.1.3 All of the specified ions listed in Table 6 for each PCDD/PCDF homologue and labeled standards must be present in the SICP. The ion current response for the quantitation and confirmation ions for the analytes and labeled standards must maximize simultaneously (± 2 seconds).

8.12.2 Signal-to-Noise Ratio - The integrated ion current for each analyte ion listed in Table 6 must be at least 2.5 times background noise and must not have saturated the detector.

8.12.3 The relative ion abundance criteria listed in Table 7 for native analytes and labeled standards must be met using areas to calculate ratios.

8.12.4 Polychlorinated diphenyl ether interferences - The identification of a GC peak as a PCDF cannot be made if no signal having S/N > 2.5 is detected, at the same retention time (+ or - 2 seconds), in the corresponding polychlorinated diphenyl ether (PCDPE) channel (see Table 6).

9.0 CALCULATIONS

9.1 Concentration – For GC peaks that have met all the identification criteria, calculate the concentration of the individual PCDD or PCDF isomers using the formulae:

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9.1.1 All matrices other than water:

$$Cn\ (ng/kg) = \frac{Qis \times An}{W \times Ais \times \overline{RRFn}}$$

WATER

$$Cn\ (pg/L) = \frac{Qis \times An}{V \times Ais \times \overline{RRFn}}$$

Where:

Cn = Concentration of unlabeled PCDD/PCDF congeners (or group of coeluting isomers within an homologous series)

An= Sum of integrated ion abundance of the quantitation ion for the unlabeled PCDDs/PCDFs (Table 6)

Ais= Sum of integrated ion abundance of the quantitation ion of the appropriate internal standard (Table 6)

W = Weight (g) of sample extracted.

V = Volume (L) of sample extracted.

Qis = Quantity (pg) of the appropriate labeled standard added to the sample prior to extraction.

RRFn = Average response factor from initial calibration.

9.1.2 The concentrations of the specific isomers are calculated as follows:

9.1.2.1 For homologues that contain only one isomer (OCDD and OCDF) or only one 2,3,7,8-substituted isomer (TCDD, PeCDD, HpCDD, and TCDF), the average RRF of the 2,3,7,8-substituted isomer from the initial calibration will be used to quantitate both the 2,3,7,8-substituted isomers and the non-2,3,7,8 isomers.

9.1.2.2 For homologues that contain more than one 2,3,7,8-substituted isomer, the average RRF used to calculate the concentration of each 2,3,7,8-substituted isomer will be the average RRF determined for that isomer during the initial calibration.

9.1.2.3 For homologues that contain one or more non-2,3,7,8 isomers, the average RRF used to calculate the concentration of these isomers will be the average of the RRFs determined during the initial calibration for the 2,3,7,8-substituted isomers in that homologue.

9.1.2.4 In addition to the concentrations of specific isomers, the total homologue concentrations are reported on Form I. Calculate the total concentration of each homologue of PCDDs/PCDFs as follows:

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- Total concentration = sum of the concentrations of every positively identified 2,3,7,8-isomer and the non-2,3,7,8 isomers which meet ion ratio identification criteria and are within the retention time window. Therefore, the total should also include the 2,3,7,8-substituted congeners.

9.1.2.5 If the original sample, prior to any dilutions, has a percent recovery of the labeled standard of less than 40% or greater than 135%, then re-extraction and reanalysis is required.

9.1.2.6 If the concentration of any PCDD/PCDF in the sample has exceeded the calibration range, or the detector has been saturated, a dilution shall be performed.

9.1.2.7 "Estimated Maximum Possible Concentration" (EMPC) calculated according to the same expression, except that "An" should represent the sum of the area under the smaller peak and of the other peak area calculated using the theoretical chlorine isotope ratio.

9.2 Calculate the percent recovery for each labeled standard in the sample extract, Ris, using the equation:

$$Ris = \frac{Ais \times Qrs}{Ars \times \overline{RRFis} \times Qis} \times 100\%$$

The $^{13}C_{12}$-1,2,3,4-TCDD is used to quantitate the tetra and penta labeled standards, and $^{13}C_{12}$-1,2,3,7,8,9-HxCDD is used to quantitate the HxCDD, HpCDF and OCDD labeled standards (see Table 2).

NOTE: For human adipose tissue, adjust the percent recoveries by adding 1 percent to the calculated value to compensate for the 1 percent of the extract diverted for the lipid determination.

9.3 If the concentration in the final extract of any of the fifteen 2,3,7,8-substituted PCDD/PCDF compounds (Table 3) exceeds the upper method calibration limits (MCL) listed in Table 1, the linear range of response versus concentration may have been exceeded, and a second analysis of the sample (using a one tenth aliquot) should be undertaken. The volumes of the internal and recovery standard solutions should remain the same as described for the sample preparation. For the other congeners (including OCDD), however, report the measured concentration and indicate that the value exceeds the MCL.

9.3.1 If a smaller sample size would not be representative of the entire sample, one of the following options is recommended:

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9.3.1.1 Re-extract an additional aliquot of sufficient size to insure that it is representative of the entire sample. Spike it with a higher concentration of internal standard. Prior to GC/MS analysis, dilute the sample so that it has a concentration of internal standard equivalent to that present in the calibration standard. Then analyze the diluted extract.

9.3.1.2 Re-extract an additional aliquot of sufficient size to insure that it is representative of the entire sample. Spike it with a higher concentration of internal standard. Immediately following extraction, transfer the sample to a volumetric flask and dilute to known volume. Remove an appropriate aliquot and proceed with cleanup and analysis.

9.3.1.3 Use the original analysis data to quantitate the internal standard recoveries. Respike the original extract with 100 times the usual quantity of internal standards. Dilute the re-spiked sample by a factor of 100. Reanalyze the diluted sample using the internal standard recoveries calculated from the initial analysis to correct the results for losses during isolation and cleanup.

9.3.2 Sample Specific Estimated Detection Limit – The sample specific estimated detection limit (EDL) is the concentration of a given analyte required to produce a signal with a peak height of at least 2.5 times the background signal level. An EDL is calculated for each 2,3,7,8-substituted congener that is not identified, regardless of whether or not other non-2,3,7,8-substituted isomers are present.

9.3.2.1 A sample giving a response for both quantitation ions (Tables 6 and 9) that is less than 2.5 times the background level.

- Use the expression for EDL (specific 2,3,7,8-substituted PCDD/PCDF) below to calculate an EDL for each absent 2,3,7,8-substituted PCDD/PCDF (i.e., S/N < 2.5). The background level is determined by measuring the range of the noise (peak to peak) for the two quantitation ions (Table 6) of a particular 2,3,7,8-substituted isomer within an homologous series, in the region of the SICP trace corresponding to the elution of the internal standard (if the congener possesses an internal standard) or in the region of the SICP where the congener is expected to elute by comparison with the routing calibration data (for those congeners that do not have a 13C-labeled standard), multiplying that noise height by 2.5, and relating the product to an estimated concentration that would produce that peak height

$$EDL = \frac{2.5 \times Hn \times Qis}{His \times W \times RRFn}$$

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Where:

EDL = estimated detection limit for homologous 2,3,7,8-substituted PCDDs/PCDFs.

Hn = sum of the height of the noise level for each quantitation ion (Table 6) for the unlabeled PCDDs/PCDFs, measured as shown in Figure 3.

W, RRFn, and Qis are defined in Sec. 9.1.1.

9.3.3 The 2,3,7,8-TCDD toxicity equivalents (TE) of PCDDs and PCDFs present in the sample are calculated according to the method recommended by the Chlorinated Dioxins Workgroup (CDWG) of the EPA and the Center for Disease Control (CDC). This method assigns a 2,3,7,8-TCDD toxicity equivalency factor (TEF) to each of the fifteen 2,3,7,8-susbtituted PCDDs and PCDFs (Table 3) and to OCDD and OCDF, as shown in Table 10. The 2,3,7,8-TCDD equivalent of the PCDDs and PCDFs present in the sample is calculated by summing the TEF times their concentration for each of the compounds or groups of compounds listed in Table 10.

9.3.3.1 Two GC Column TEF Determination – The concentration of 2,3,7,8-TCDD is calculated from the analysis of the sample extract on the 60 m DB-5 fused silica capillary column. The chromatographic separation between the 2,3,7,8-TCDD and its close eluters (1,2,3,7/1,2,3,8-TCDD and 1,2,3,9-TCDD) must be equal or less than 25 percent valley.

9.3.3.2 The concentration of the 2,3,7,8-TCDF is obtained from the analysis of the sample extract on the SP-2331 GC column. The chromatographic separation between the 2,3,7,8-TCDF and its close eluters (2,3,4,7-TCDF and 1,2,3,9-TCDF) must be equal or less than 25 percent valley.

NOTE: The confirmation and quantitation of 2,3,7,8-TCDF may be accomplished on the SP-2331 GC column.

10.0 QUALITY ASSURANCE/QUALITY CONTROL

10.1 System performance criteria – It must be documented that all applicable system performance criteria (specified in Secs. 8.7.2, 8.8.1 and 8.8.2) were met before analysis of any sample is performed. Figure 3 provides a typical 12 hour analysis sequence, whereby the response factors and mass spectrometer resolving power checks must be performed at the beginning and the end of each 12 hour period of operation. A GC column performance check is only required at the beginning of each 12-hour period during which samples are analyzed. An HRGC/HRMS method blank run is required between a calibration run and the first sample run. The same method blank extract may thus be analyzed more than once if the number of samples within a batch requires more than 12 hours of analyses.

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10.1.1 GC column performance – Inject 2 µL of the column performance check solution (Sec. 7.6) and acquire selected ion monitoring (SIM) data as described in Sec. 8.7.2 within a total cycle time of ≤ 1 second (Sec. 8.7.3)

10.1.2 The chromatographic separation between 2,3,7,8-TCDD and the peaks representing any other unlabeled TCDD isomers must be resolved with a valley of ≤ 25 percent (Figure 4).

Where: Valley percent = (x/y) (100)
x = measured as in Figure 4 from the 2,3,7,8-closeset TCDD eluting isomer
y = the peak height of 2,3,7,8-TCDD.

The GC column performance check solution also contains the known first and last PCDD/PCDF eluters under the conditions specified in this protocol. Their retention times are used to determine the eight homologue retention time windows that are used for qualitative (Sec. 8.12.1) and quantitative purposes. All peaks should be labeled and identified on the chromatograms. Furthermore, all first eluters of a homologous series should be labeled with the letter F, and all last eluters of a homologous series should be labeled with the letter L.

10.1.3 The retention times for the switching of SIM ions characteristic of one homologous series to the next higher homologous series must be indicated in the SICP.

10.2 Mass Spectrometer Performance – The mass spectrometer must be operated in the electron ionization mode. A static resolving power of at least 10,000 (10 percent valley definition) must be demonstrated at appropriate masses before any analysis is performed (Sec. 8.7.2). Static resolving power checks must be performed at the beginning and at the end of each 12-hour period of operation. Corrective action must be implemented whenever the resolving power does not meet the requirement.

10.2.1 Chromatography time for PCDDs and PCDFs exceeds the long-term mass stability of the mass spectrometer. Because the instrument is operated in the high-resolution mode, mass drifts of 5 ppm in mass can have serious adverse effects on instrument performance. Therefore, a mass drift correction is mandatory. It is recommended to select a lock-mass ion from the reference compound (PFK) used for tuning the mass spectrometer. The selection of the lock-mass ion is dependent on the masses of the ions monitored within each descriptor. Although Table 6 offers suggestions for the lock-mass ions, an acceptable lock-mass ion at any mass between the lightest and heaviest ion in each descriptor can be used to monitor and correct mass drifts. The level of PFK metered into the ion chamber during HRGC/HRMS analyses should be adjusted so that the amplitude of the most intense selected lock-mass ion signal does not exceed 10 percent of the full scale deflection for a given set of detector parameters.

10.2.2 Documentation of the instrument resolving power is accomplished by recording the peak profile of the high-mass reference signal (m/z 380.9760) obtained during the above peak matching experiment by using the low-mass PFK ion (m/z

302.9824) as a reference. The minimum resolving power of 10,000 must be demonstrated on the high-mass ion while it is transmitted at a lower accelerating voltage than the low-mass reference ion, which is transmitted at full sensitivity. The format of the peak profile representation must allow manual determination of the resolution, i.e., the horizontal axis must be a calibrated mass scale (amu or ppm per division). The result of the peak width measurement (performed at 5 percent of the maximum, which corresponds to the 10 percent valley definition) must appear on the hard copy and cannot exceed 100 ppm at m/z 380.9760 (or 0.038 amu at that particular mass).

10.3 Quality Control Samples

10.3.1 Performance Check Solutions – At the beginning of each 12-hour period during which samples are to be analyzed, an aliquot of the GC column performance check solution and of the HRCC-3 shall be analyzed to demonstrate adequate GC resolution and sensitivity, response factor reproducibility, and mass range calibration, and to establish the PCDD/PCDF retention time windows. A mass resolution check shall also be performed to demonstrate adequate mass resolution. If the required criteria are not met, remedial action must be taken before any samples are analyzed.

10.3.2 To validate positive sample data, the continuing calibration (HRCC-3) and the mass resolution check must be performed also at the end of each 12-hour period during which samples are analyzed. An HRGC/HRMS method blank run must be recorded following a calibration run and before the first sample run.

10.3.2.1 If only one 12-hour period (or less) of operation is performed, the performance check solution must be analyzed only once at the beginning of that period for data validation. However, the mass resolution and continuing calibration checks must be performed at the beginning as well as at the end of the period of operation.

10.3.2.2 If consecutive 12-hour periods of operation are performed, analysis of the GC performance check solution must be performed at the beginning of each 12-hour period. The mass resolution and continuing calibration checks from the previous period can be used for the beginning of the next period.

10.3.3 Results of at least one analysis of the GC column performance check solution and of two mass resolution and continuing calibration checks must be reported with the sample data collected during a 12-hour period.

10.3.4 Deviations from criteria specified for the GC performance check or for the mass resolution check invalidate all positive sample data collected between analyses of the performance check solution, and the extracts from those positive samples shall be reanalyzed.

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10.3.5 If the routine calibration run fails at the beginning of a 12-hour shift, the instructions in Sec. 8.10.1.5 must be followed. If the continuing calibration check performed at the end of a 12-hour period fails by no more than 25 percent RPD for the 17 unlabeled compounds and 35 percent RPD for the 9 labeled reference compounds, use the mean average of RRFs from the two daily routine calibration runs to compute the analyte concentrations, instead of the average of RRFs obtained from the initial calibration. A new initial calibration average of new RRFs is required immediately following the analysis of the samples, whenever the RPD from the end-of-shift routine calibration exceeds 25 percent or 35 percent, respectively. Failure to perform a new initial calibration immediately following the analysis of the samples will automatically require reanalysis of all positive sample extracts analyzed before the failed end-of-shift continuing calibration check.

10.3.6 Field Blanks – Each batch of samples usually contains a field blank sample of uncontaminated soil, sediment or water that is to be fortified before analysis. In addition to this field blank, a batch of samples may include a rinsate, which is a portion of the solvent that was used to rinse sampling equipment. The rinsate is analyzed to assure that the samples were not contaminated by the sampling equipment.

10.3.6.1 Fortified Field Blank – Weigh a 10 g portion or use 1 L (for aqueous samples) of the specified field blank sample and add 1mL of the internal standard solution (Table 4). Extract by using the procedures beginning in Secs. 8.4.5 or 8.4.6, as applicable. Add 20 µL of the recovery standard solution (Sec. 8.6.3.6) and analyze a 2 µL aliquot of the concentrated extract. Calculate the concentration of 2,3,7,8-substituted PCDDs/PCDFs and the percent recovery of the internal standards. Extract and analyze a new simulated fortified field blank whenever new lots of solvents or reagents are used for sample extraction or for column chromatographic procedures.

10.3.6.2 Rinsate Sample – The rinsate sample must be fortified like a regular sample. Take a 100 mL portion of the sampling equipment rinse solvent, filter (if necessary), and add 1mL of the internal standard solution (Table 4). Using a KD apparatus (or a vacuum concentrator), concentrate to approximately 5 mL. Transfer the 5 mL concentrate from the KD concentrator tube in 1 mL portions to a 1 mL minivial, reducing the volume in the minivial as necessary with a gentle stream of dry nitrogen. Rinse the KD concentrator tube with two 0.5 mL portions of hexane and transfer the rinses to the 1 mL minivial. Blow down with dry nitrogen as necessary. Just before analysis, add 20 µL recovery standard solution (Table 4) and reduce the volume to its final volume. No column chromatography is required. Analyze an aliquot following the same procedures used to analyze samples. Report percent recovery of the internal standard and the presence of any PCDD/PCDF compounds in µg/L of rinsate solvent.

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10.3.7 Duplicate Analyses – In each batch of samples, a sample may be specified for duplicate analysis. If so, analyze a second 10 g soil or sediment sample portion or 1 L water sample (or an appropriate amount of the type of matrix under consideration). The results of the laboratory duplicates (percent recovery and concentrations of 2,3,7,8-substituted PCDD/PCDF compounds) should agree within 25 percent relative difference.

10.3.8 Matrix Spike and Matrix Spike Duplicate – Add an appropriate volume of the matrix spike fortification solution (Sec. 7.9) and of the sample fortification solution (Sec. 7.7), adjusting the fortification level as specified in Table 1 under IS Spiking Levels, to a specified sample. Analyze the MS and MSD samples as described in Sec. 8.11. The results obtained from the MS and MSD samples (concentrations of 2,3,7,8-substituted PCDDs/PCDFs) should agree within 20 percent relative difference.

10.4 Percent Recovery of the Internal Standards – For each sample, method blank and rinsate, calculate the percent recovery. The percent recovery should be between 40 percent and 135 percent for all 2,3,7,8-substituted internal standards.

10.4.1 A low or high percent recovery for a blank does not require discarding the analytical data but it may indicate a potential problem with future analytical data.

10.5 Identification Criteria – If any one of the identification criteria appearing in Sec. 8.11 is not met for an homologous series, it is reported that the sample does not contain unlabeled 2,3,7,8-substituted PCDD/PCDF isomers for that homologous series at the calculated detection limit (Sec. 9.3.2).

10.5.1 If the first initial identification criteria (Secs. 8.12.1 through 8.12.1.3 are met, but the criteria appearing in Sec. 8.12.3 are not met, that sample is presumed to contain interfering contaminants. This must be noted on the analytical report form and the sample should be rerun or the extract reanalyzed.

10.6 Unused portions of samples and sample extracts must be preserved for six months after sample receipt to allow further analyses.

11.0 WASTE DISPOSAL. All samples are disposed of in accordance with the Laboratory Waste Disposal SOP.

12.0 METHOD PERFORMANCE. Method performance data are currently not available.

13.0 REFERENCES

13.1 "Method 8290: The Analysis of Polychlorinated Dibenzodioxins (PCDDs) and Polychlorinated Dibenzofurans (PCDFs) by High-Resolution Gas Chromatography/High-Resolution Mass Spectrometry (HRGC/HRMS)", Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, EPA SOW SW-846 Update II, September 1994.

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13.2 "Method 1613: Tetra- Through Octa- Chlorinated Dioxins and Furans by Isotope Dilution HRGC/HRMS", US EPA, Revision A, April 1990.

13.3 "Method 1613: Tetra- Through Octa-Chlorinated Dioxins and Furans by Isotope Dilution HRGC/HRMS", US EPA, Revision B, October 1994.

14.0 DEFINITIONS

14.1 Analyte:.....A CDD or CDF tested for by this method, listed in Table 3.

14.2 Calibration standard:.....A solution prepared from a secondary standard and/or stock solutions and used to calibrate the response of the instrument with respect to analyte concentration.

14.3 Continuing calibration standard:....The mid-point calibration standard (HRCC-3) that is used to verify calibration. See Table 5.

14.4 CDD:.....Chlorinated Dibenzo-p-Dioxin. The isomers and congeners of tetra-through octa-chlorodibenzo-p-dioxin.

14.5 CDF: .....Chlorinated Dibenzofuran. The isomers and congeners of tetra-through octa-chlorodibenzofuran.

14.6 Estimated Detection Limit (EDL): The concentration of an analyte required to produce a signal with peak height of at least 2.5 times the background signal level. The EDL is calculated for each 2,3,7,8-substituted isomer for which the response of the quantitation and confirmation ions is less than 2.5 times the background level.

14.7 Estimated Maximum Possible Concentration (EMPC): The concentration of a given analyte that would produce a signal with a given peak area. The EMPC is calculated for 2,3,7,8-substituted isomers for which the quantitation and/or the confirmation ion(s) has signal-to-noise in excess of 2.5 but does not meet identification criteria.

14.8 Field blank:.....An aliquot of reagent water or other reference matrix that is placed in a sample container in the laboratory or the field, and treated as a sample in all respects, including exposure to sampling site conditions, storage, preservation, and all analytical procedures. The purpose of the field blank is to determine if the field or sample transporting procedures and environments have contaminated the sample.

14.9 Homologue:.....A member or members of a particular homologous series that has the same molecular weight but not necessarily the same structural arrangement.

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14.10 Homologous Series:.....A series of organic compounds in which each seccessive member has one more atom or group of atoms than the preceding member.

14.11 HRCC-1, HRCC-2, HRCC-3,:..... See Calibration standards and Table 5.
HRCC-4, HRCC-5

14.12 Initial Calibration: .....Analysis of analytical standards for a series of different specified concentrations. The initial calibration is used to define the linearity and dynamic range of the response of the mass spectrometer to the target compounds.

14.13 Internal Standards:.....Isotopically labeled standards used to determine exact retention times of unlabeled analytes.

14.14 Isomer: .....Chemical compounds that contain the same number of atoms of the same elements, but differ in structural arrangement and properties.

14.15 Matrix: .....The predominant material that comprises the sample to be analyzed. Matrix is not synonymous with phase.

14.16 Method blank: .....An analytical control consisting of all reagents, internal standards and recovery standards that is carried through the entire analytical procedure. The method blank is used to define the level of laboratory background contamination.

14.17 Recovery: .....A determination of the acuracy of the analytical procedure made by comparing measured values for a fortified (spiked) sample against the known spike values.

14.18 Recovery Standards:.....13C12-1,2,3,4-TCDD and 13C12-1,2,3,7,8,9-HxCDD are added to every blank, quality control sample, and sample extract aliquot just prior to analysis and are present in all solutions except the internal standard solutions. Recovery standards are used to measure the recovery of the internal standards.

14.19 Relative Response Factor (RRF) – The ration of the response of the mass spectrometer to a know amount of an analyte relative to that of a known amount of an internal standard as measured in the initial and continuing calibrations. The RRF is used to determine instrument performance and is used in the quantitation calculations.

14.20 Resolution: .....The separation between peaks on a chromatogram, calculated by dividing the height of the valley between the peak height of the smaller peak being resolved, multiplied by 100.

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14.21 Selected Ion Monitoring (SIM) – A mass spectrometric technique whereby ions with predetermined mass/charge ratios (m/z) are monitored.

14.22 Signal-To-Noise Ratio (S/N) – the ratio of analyte signal to random background signal.

15.0 ATTACHMENTS

15.1 Table 1 – Table 12

15.2 Figure 1 – Figure 4

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Table 1
Types of Matrices, Sample Sizes and 2,3,7,8-TCDD-Based Method Calibration Limits (Parts Per Trillion)

	Water	Soil Sediment Paper Pulp(b)	Fly Ash	Fish Tissue(c)	Human Adipose Tissue	Sludges, Fuel Oil	Still-Bottom
Lower MCL(a)	0.01	1.0	1.0	1.0	1.0	5.0	10
Upper MCL(a)	2	200	200	200	200	1000	2000
Weight (g)	1000	10	10	20	10	2	1
IS Spiking Levels (ppt)	1	100	100	100	100	500	1000
Final Extr. Vol. (uL)(d)	10-50	10-50	50	10-50	10-50	50	50

(a) For other congeners multiply the values by 1 for TCDF/PeCDD/PeCDF, by 2.5 for HxCDD/HxCDF/HpCDD/HpCDF, and by 5 for OCDD/OCDF.
(b) Sample dewatered according to Sec. 6.5.
(c) One half of the extract from the 20 g sample is used for determination of lipid content (Sec. 7.2.2).
(d) See Sec. 7.8.1, Note.

NOTE: Chemical reactor residues are treated as still bottoms if their appearances so suggest.

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Table 2
Reference Compounds for Quantitation of Native And Labeled PCDDs and PCDFs

Compound Number	Type	Name	Reference Compound
1	NAT	2,3,7,8-TCDD	18
2	NAT	1,2,3,7,8-PeCDD	19
3	NAT	1,2,3,4,7,8-HxCDD	20
4	NAT	1,2,3,6,7,8-HxCDD	20
5	NAT	1,2,3,7,8,9-HxCDD	20
6	NAT	1,2,3,4,6,7,8-HpCDD	21
7	NAT	OCDD	22
8	NAT	2,3,7,8-TCDF	23
9	NAT	1,2,3,7,8-PeCDF	24
10	NAT	2,3,4,7,8-PeCDF	24
11	NAT	1,2,3,4,7,8-HxCDF	25
12	NAT	1,2,3,6,7,8-HxCDF	25
13	NAT	1,2,3,7,8,9-HxCDF	25
14	NAT	2,3,4,6,7,8-HxCDF	25
15	NAT	1,2,3,4,6,7,8-HpCDF	26
16	NAT	1,2,3,4,7,8,9-HpCDF	26
17	NAT	OCDF	22
18	IS	^{13}C-2,3,7,8-TCDD	27
19	IS	^{13}C-1,2,3,7,8-PeCDD	27
20	IS	^{13}C-1,2,3,6,7,8-HxCDD	28
21	IS	^{13}C-1,2,3,4,6,7,8-HpCDD	28
22	IS	^{13}C-OCDD	28
23	IS	^{13}C-2,3,7,8-TCDF	27
24	IS	^{13}C-1,2,3,7,8-PeCDF	27
25	IS	^{13}C-1,2,3,4,7,8-HxCDF	28
26	IS	^{13}C-1,2,3,4,6,7,8-HpCDF	28
27	RS	^{13}C-1,2,3,4-TCDD	
28	RS	^{13}C-1,2,3,7,8,9-HxCDD	
29	C/UP	^{37}Cl-1,2,3,7,8-TCDD	27

NAT = Native Compounds
IS = Internal Standard (Labeled Compounds)
RS = Recovery Standard
C/UP = Cleanup Standard

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Table 3
The Fifteen 2,3,7,8-Substituted PCDD and PCDF Congeners

PCDD	PCDF
2,3,7,8-TCDD(*)	2,3,7,8-TCDF(*)
1,2,3,7,8-PeCDD(*)	1,2,3,7,8-PeCDF(*)
1,2,3,6,7,8-HxCDD(*)	2,3,4,7,8-PeCDF
1,2,3,4,7,8-HxCDD	1,2,3,6,7,8-HxCDF
1,2,3,7,8,9-HxCDD(+)	1,2,3,7,8,9-HxCDF
1,2,3,4,6,7,8-HpCDD(*)	1,2,3,4,7,8-HxCDF(*)
	2,3,4,6,7,8-HxCDF
	1,2,3,4,6,7,8-HpCDF(*)

(*) The 13C-labeled analogue is used as an internal standard.
(+) The 13C-labeled analogue is used as a recovery standard.

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Table 4
Composition of the Sample Fortification and Recovery Standard Solutions

	Sample Fortification Solution Concentration(1) (pg/µL)	Recovery Standard Solution Concentration (2) (pg/µL)
	Solvent: Nonane	Solvent: Nonane
C12-2,3,7,8-TCDD	1.0	—
C12-2,3,7,8-TCDF	1.0	—
C12-1,2,3,4-TCDD	—	50
C12-1,2,3,7,8-PeCDD	1.0	—
C12-1,2,3,7,8-PeCDF	1.0	—
C12-1,2,3,6,7,8-HxCDD	2.5	—
C12-1,2,3,4,7,8-HxCDF	2.5	—
C12-1,2,3,7,8,9-HxCDD	—	50
C12-1,2,3,4,6,7,8-HpCDD	2.5	—
C12-1,2,3,4,6,7,8-HpCDF	2.5	—
C12-OCDD	5.0	—

These solutions should be made fresh every day because of the possibility of absorptive losses to glassware.

(1) Add 1 mL of this solution to sample before extraction
(2) Add 20 uL of this solution to dry extract.

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Table 5
Concentrations of Solutions Containing Labeled and Unlabeled PCDDs and PCDFs Calibration and Verification Solutions

Compound	HRCC5 (ng/mL)	HRCC4 (ng/mL)	Ver [a] HRCC3 (ng/mL)	HRCC2 (ng/mL)	HRCC1 (ng/mL)
Native CDDs and CDFs					
2,3,7,8-TCDD	200	50	10	2.5	1
2,3,7,8-TCDF	200	50	10	2.5	1
1,2,3,7,8-PeCDD	500	125	25	6.25	2.5
1,2,3,7,8-PeCDF	500	125	25	6.25	2.5
2,3,4,7,8-PeCDF	500	125	25	6.25	2.5
1,2,3,4,7,8-HxCDD	500	125	25	6.25	2.5
1,2,3,6,7,8-HxCDD	500	125	25	6.25	2.5
1,2,3,7,8,9-HxCDD	500	125	25	6.25	2.5
1,2,3,4,7,8-HxCDF	500	125	25	6.25	2.5
1,2,3,6,7,8-HxCDF	500	125	25	6.25	2.5
1,2,3,7,8,9-HxCDF	500	125	25	6.25	2.5
2,3,4,6,7,8-HxCDF	500	125	25	6.25	2.5
1,2,3,4,6,7,8-HpCDD	500	125	25	6.25	2.5
1,2,3,4,6,7,8-HpCDF	500	125	25	6.25	2.5
1,2,3,4,7,8,9-HpCDF	500	125	25	6.25	2.5
OCDD	1000	250	50	12.5	5
OCDF	1000	250	50	12.5	5
Internal Standards					
$^{13}C_{12}$-2,3,7,8-TCDD	50	50	50	50	50
$^{13}C_{12}$-2,3,7,8-TCDF	50	50	50	50	50
$^{13}C_{12}$-1,2,3,7,8-PeCDD	50	50	50	50	50
$^{13}C_{12}$-1,2,3,7,8-PeCDF	50	50	50	50	50
$^{13}C_{12}$-1,2,3,6,7,8-HxCDD	125	125	125	125	125
$^{13}C_{12}$-1,2,3,4,7,8-HxCDF	125	125	125	125	125
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD	125	125	125	125	125
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF	125	125	125	125	125
$^{13}C_{12}$-OCDD	250	250	250	250	250
Recovery Standard					
$^{13}C_{12}$-1,2,3,4-TCDD (b)	50	50	50	50	50
$^{13}C_{12}$-1,2,3,7,8,9-HxCDD (c)	125	125	125	125	125

(a) Calibration verification (VER) solution.

(b) Used for recovery determinations of TCDD, TCDF, PeCDD and PeCDF internal standards.

(c) Used for recovery determinations of HxCDD, HxCDF, HpCDD, HpCDF and OCDD internal standards.

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Table 6
Descriptors, m/z Types, and Elemental Compositions of the CDDs and CDFs[1]

Descriptor Number	Accurate m/z[2]	m/z Type	Elemental Composition	Compound[3]	Primary m/z?
1	292.9825	LOCK	$C_7 F_{11}$	PFK	
	303.9016	M	$C_{12} H_4 {}^{35}Cl_4 O$	TCDF	YES
	305.8987	M+2	$C_{12} H_4 {}^{35}Cl_3$	TCDF	
	315.9419	M	${}^{13}C_{12} H_4 {}^{35}Cl_4 O$	TCDF[4]	YES
	317.9389	M+2	${}^{13}C_{12} H_4 {}^{35}Cl_3$	TCDF[4]	
	319.8965	M	$C_{12} H_4 {}^{35}Cl_4 O_2$	TCDD	YES
	321.8936	M+2	$C_{12} H_4 {}^{35}Cl {}^{37}Cl O_2$	TCDD	
	327.8847	M	$C_{12} H_4 {}^{37}Cl_4 O_2$	TCDD[5]	
	330.9792	QC	$C_7 F_{13}$	PFK	
	331.9368	M	${}^{13}C_{12} H_4 {}^{35}Cl_4 O_2$	TCDD[4]	YES
	333.9339	M+2	${}^{13}C_{12} H_4 {}^{35}Cl O_2$	TCDD[4]	
	375.8364	M+2	$C_{12} H_4 {}^{35}Cl_5 {}^{37}Cl O$	HxCDPE	
2	339.8597	M+2	$C_{12} H_3 {}^{35}Cl_4 {}^{37}Cl O$	PeCDF	YES
	341.8567	M+4	$C_{12} H_3 {}^{35}Cl_3 {}^{37}Cl_2 O$	PeCDF	
	351.9000	M+2	${}^{13}C_{12} H_3 {}^{35}C_{14} {}^{37}Cl O$	PeCDF[4]	YES
	353.8970	M+4	${}^{13}C_{12} H_3 {}^{35}Cl_3 {}^{37}Cl_2 O$	PeCDF[4]	
	354.9792	LOCK	$C_9 F_{13}$	PFK	
	355.8546	M+2	$C_{12} H_3 {}^{35}Cl_4 {}^{37}Cl O_2$	PeCDD	YES
	357.8516	M+4	$C_{12} H_3 {}^{35}Cl_3 {}^{37}Cl_2 O_2$	PeCDD	
	367.8949	M+2	${}^{13}C_{12} H_3 {}^{35}Cl_4 {}^{37}Cl O_2$	PeCDD[4]	YES
	369.8919	M+4	${}^{13}C_{12} H_3 {}^{35}Cl_3 {}^{37}Cl_2 O_2$	PeCDD[4]	
	409.7974	M+2	$C_{12} H_3 {}^{35}Cl_6 {}^{37}Cl O$	HpCDPE	
3	373.8208	M+2	$C_{12} H_2 {}^{35}Cl_5 {}^{37}Cl O$	HxCDF	YES
	375.8178	M+4	$C_{12} H_2 {}^{35}Cl_4 {}^{37}Cl2 O$	HxCDF	
	383.8639	M	${}^{13}C_{12} H_2 {}^{35}Cl_6 O$	HxCDF[4]	YES
	385.8610	M+2	${}^{13}C_{12} H_2 {}^{35}Cl_5 {}^{37}Cl O$	HxCDF[4]	
	389.8157	M+2	$C_{12} H_2 {}^{35}Cl_5 {}^{37}Cl O_2$	HxCDD	YES
	391.8127	M+4	$C_{12} H_2 {}^{35}Cl_4 {}^{37}Cl_2 O_2$	HxCDD	
	392.9760	LOCK	$C_9 F_{15}$	PFK	
	401.8559	M+2	${}^{13}C_{12} H_2 {}^{35}Cl_5 {}^{37}Cl O_2$	HxCDD[4]	YES
	403.8529	M+4	${}^{13}C_{12} H_2 {}^{35}Cl_4 {}^{37}Cl_2 O_2$	HxCDD[4]	YES
	430.9729	QC	$C_9 F_{13}$	PFK	
	445.7555	M+4	$C_{12} H_2 {}^{35}Cl_6 {}^{37}Cl_2 O$	OCDPE	
4	407.7818	M+2	$C_{12} H {}^{35}Cl_6 {}^{37}Cl O$	HpCDF	YES
	409.7789	M+4	$C_{12} H {}^{35}Cl_7 O$	HpCDF	
	417.8253	M	${}^{13}C_{12} H {}^{35}Cl_7 O$	HpCDF[4]	YES
	419.8220	M+2	${}^{13}C_{12} H {}^{35}Cl_6 {}^{37}Cl O$	HpCDF[4]	
	423.7766	M+2	$C_{12} H {}^{35}Cl_6 {}^{37}Cl O_2$	HpCDD	YES
	425.7737	M+4	$C_{12} H {}^{35}Cl_5 {}^{37}Cl_2 O_2$	HpCDD	
	430.9729	LOCK	$C_9 F_{17}$	PFK	
	435.8169	M+2	${}^{13}C_{12} H {}^{35}Cl_6 {}^{37}Cl O_2$	HpCDD[4]	YES
	437.8140	M+4	${}^{13}C_{12} H {}^{35}Cl_5 {}^{37}Cl_2 O_2$	HpCDD[4]	
	479.7165	M+4	$C_{12} H {}^{35}Cl_7 {}^{37}Cl_2 O$	NCDPE	

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Table 6 (Continued)
Descriptors, m/z Types, and Elemental Compositions of the CDDs and CDFs(1)

Descriptor Number	Accurate m/z(2)	m/z Type	Elemental Composition	Compound(3)	Primary m/z?
5	441.7428	M+2	$C_{12}\ ^{35}Cl_7\ ^{37}Cl\ O$	OCDF	YES
	442.9728	LOCK	$C_{10}\ F_{17}$	PFK	
	443.7399	M+4	$C_{12}\ ^{35}C_{16}\ ^{37}Cl_2\ O$	OCDF	
	457.7377	M+2	$C_{12}\ ^{35}C_{17}\ ^{37}Cl\ O_2$	OCDD	YES
	459.7348	M+4	$C_{12}\ ^{35}C_{16}\ ^{37}Cl_2\ O_2$	OCDD	
	469.7779	M+2	$^{13}C_{12}\ ^{35}C_{17}\ ^{37}Cl\ O_2$	OCDD(4)	YES
	471.7750	M+4	$^{13}C_{12}\ ^{35}C_{16}\ ^{37}Cl_2\ O_2$	OCDD(4)	
	513.6775	M+4	$C_{12}\ ^{35}C_{18}\ ^{37}Cl_2\ O$	DCDPE	

(1) From Reference 5

(2) Nuclidic masses used:

H = 1.007825 C = 12.00000 ^{13}C = 13.003355 F = 18.9984
O = 15.994915 ^{35}Cl = 34.968853 ^{37}Cl = 36.965903

(3) Compound abbreviations:

Chlorinated dibenzo-p-dioxins:
TCDD = Tetrachlorodibenzo-p-dioxin
PeCDD = Pentachlorodibenzo-p-dioxin
HxCDD = Hexachlorodibenzo-p-dioxin
HpCDD = Heptachlorodibenzo-p-dioxin
OCDD = Octachlorodibenzo-p-dioxin

Chlorinated dibenzofurans
TCDF = Tetrachlorodibenzofuran
PeCDF = Pentachlorodibenzofuran
HxCDF = Hexachlorodibenzofuran
HpCDF = Heptachlorodibenzofuran

Chlorinated diphenyl ethers
HxCDPE = Hexachlorodiphenyl ether
HpCDPE = Heptachlorodiphenyl ether
OCDPE = Octachlorodiphenyl ether
NCDPE = Nonachlorodiphenyl ether
DCDPE = Dechachlorodiphenyl ether

Lock mass and QC compound
PFK = Perfluorokerosene

(4) Labeled compound

(5) There is only one m/z for $^{37}Cl_4$-2,3,7,8-TCDD (cleanup standard).

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Table 7
Theoretical Ion Abundance Ratios and Control Limits

No. of Chlorine Atoms	m/z's Forming Ratio	Theoretical Ratio	Control Limits (1)	
			Lower	Upper
4(2)	M/M+2	0.77	0.65	0.89
5	M+2/M+4	1.55	1.32	1.78
6	M+2/M+4	1.24	1.05	1.43
6(3)	M/M+2	0.51	0.43	0.59
7	M+2/M+4	1.05	0.88	1.20
7(4)	M/M+2	0.44	0.37	0.51
8	M+2/M+4	0.89	0.76	1.02

(1) Represent ±15% windows around the theoretical ion abundance ratios.

(2) Does not apply to $^{37}Cl_4$-2,3,7,8-TCDD (cleanup standard).

(3) Used for ^{13}C-HxCDF only.

(4) Used for ^{13}C-HpCDF only.

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Table 8
Isomers of Chlorinated Dioxins and Furans as a Function of the Number of Chlorine Atoms

Number of Chlorine Atoms	Number of Dioxin Isomers	Number of 2,3,7,8 Isomers	Number of Furan Isomers	Number of 2,3,7,8 Isomers
1	2	---	4	---
2	10	---	16	---
3	14	---	28	---
4	22	1	38	1
5	14	1	28	2
6	10	3	16	4
7	2	1	4	2
8	1	1	1	1
Total	75	7	135	10

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Table 9
Relative Response Factor [RF (Number)] Attributions

Number	Specific Congener Name
1	2,3,7,8-TCDD (and total TCDDs)
2	2,3,7,8-TCDF (and total TCDFs)
3	1,2,3,7,8-PeCDD (and total PeCDDs)
4	1,2,3,7,8-PeCDF
5	2,3,4,7,8-PeCDF
6	1,2,3,4,7,8-HxCDD
7	1,2,3,6,7,8-HxCDD
8	1,2,3,7,8,9-HxCDD
9	1,2,3,4,7,8-HxCDF
10	1,2,3,6,7,8-HxCDF
11	1,2,3,7,8,9-HxCDF
12	2,3,4,6,7,8-HxCDF
13	1,2,3,4,6,7,8-HpCDD (and total HpCDDs)
14	1,2,3,4,6,7,8-HpCDF
15	1,2,3,4,7,8,9-HpCDF
16	OCDD
17	OCDF
18	(13)C12-2,3,7,8-TCDD
19	(13)C12-2,3,7,8-TCDF
20	(13)C12-1,2,3,7,8-PeCDD
21	(13)C12-1,2,3,7,8-PeCDF
22	(13)C12-1,2,3,6,7,8-HxCDD
23	(13)C12-1,2,3,4,7,8-HxCDF
24	(13)C12-1,2,3,4,6,7,8-HpCDD
25	(13)C12-1,2,3,4,6,7,8-HpCDF
26	(13)C12-OCDD
27	Total PeCDFs
28	Total HxCDFs
29	Total HxCDDs
30	Total HpCDFs

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Table 10
2,3,7,8-TCDD Toxicity Equivalency Factors (TEFS) for the Polychlorinated Dibenzodioxins and Dibenzofurans

Number	Compound(s)	TEF(a)
1	2,3,7,8-TCDD	1.00
2	1,2,3,7,8-PeCDD	0.50
3	1,2,3,6,7,8-HxCDD	0.10
4	1,2,3,7,8,9-HxCDD	0.10
5	1,2,3,4,7,8-HxCDD	0.10
6	1,2,3,4,6,7,8-HpCDD	0.01
7	1,2,3,4,6,7,8,9-OCDD	0.001
8	2,3,7,8-TCDF	0.1
9	1,2,3,7,8-PeCDF	0.05
10	2,3,4,7,8-PeCDF	0.5
11	1,2,3,6,7,8-HxCDF	0.1
12	1,2,3,7,8,9-HxCDF	0.1
13	1,2,3,4,7,8-HxCDF	0.1
14	2,3,4,6,7,8-HxCDF	0.1
15	1,2,3,4,6,7,8-HpCDF	0.01
16	1,2,3,4,7,8,9-HpCDF	0.01
17	1,2,3,4,6,7,8,9-OCDF	0.001

(a) Taken from "Interim Procedures for Estimating Risks Associated with Exposures to Mixtures of Chlorinated Dibenzo-p-Dioxin and -Dibenzofurans (CDDs and CDFs) and 1989 Update", (EPA/625/3-89/016, March 1989).

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Table 11
Analyte Relative Retention Time Reference Attributions

Analyte	Analyte RRT Reference
2,3,7,8-TCDD	^{13}C-2,3,7,8-TCDD
2,3,7,8-TCDF	^{13}C-2,3,7,8-TCDF
1,2,3,7,8-PeCDD	^{13}C-1,2,3,7,8-PeCDD
1,2,3,7,8-PeCDF	^{13}C-1,2,3,7,8-PeCDF
2,3,4,7,8-PeCDF	^{13}C-1,2,3,7,8-PeCDF
1,2,3,4,7,8-HxCDD	^{13}C-1,2,3,6,7,8-HxCDD
1,2,3,6,7,8-HxCDD	^{13}C-1,2,3,6,7,8-HxCDD
1,2,3,7,8,9-HxCDD	^{13}C-1,2,3,6,7,8-HxCDD
1,2,3,4,7,8-HxCDF	^{13}C-1,2,3,4,7,8-HxCDF
1,2,3,6,7,8-HxCDF	^{13}C-1,2,3,4,7,8-HxCDF
1,2,3,7,8,9-HxCDF	^{13}C-1,2,3,4,7,8-HxCDF
2,3,4,6,7,8-HxCDF	^{13}C-1,2,3,4,7,8-HxCDF
1,2,3,4,6,7,8-HpCDD	^{13}C-1,2,3,4,6,7,8-HpCDD
1,2,3,4,6,7,8-HpCDF	^{13}C-1,2,3,4,6,7,8-HpCDF
1,2,3,4,7,8,9-HpCDF	^{13}C-1,2,3,4,6,7,8-HpCDF
OCDD	^{13}C-OCDD
OCDF	^{13}C-OCDD

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Table 12
PCDD and PCDF Congeners Present in the GC Performance Evaluation Solution And Used for Defining the Homologous GC Retention Time Windows on a 60M DB-5 Column

No. of Chlorine Atoms	PCDD Positional Isomer		PCDF Positional Isomer	
	First Eluter	Last Eluter	First Eluter	Last Eluter
4(a)	1,3,6,8	1,2,8,9	1,3,6,8	1,2,8,9
5	1,2,4,6,8/ 1,2,4,7,9	1,2,3,8,9	1,3,4,6,8	1,2,3,8,9
6	1,2,4,6,7,9/ 1,2,4,6,8,9	1,2,3,4,6,7	1,2,3,4,6,8	1,2,3,4,8,9
7	1,2,3,4,6,7,9	1,2,3,4,6,7,8	1,2,3,4,6,7,8	1,2,3,4,7,8,9
8		1,2,3,4,6,7,8,9		1,2,3,4,6,7,8,9

(a) In addition to these two TCDD isomers, the 1,2,3,4-,1,2,3,7-, 1,2,3,8-, 2,3,7,8-,(13)C12-2,3,7,8-, and 1,2,3,9-TCDD isomers must also be present as a check of column resolution.

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Table 13
Matrix Spike Fortification Solution

ANALYTE	SPIKE ADDED (PG)
2378-TCDD	200
12378-PeCDD	500
123478-HxCDD	500
123678-HxCDD	500
123789-HxCDD	500
1234678-HpCDD	500
0CDD	1000
2378-TCDF	200
12378-PeCDF	500
23478-PeCDF	500
123478-HxCDF	500
123678-HxCDF	500
123789-HxCDF	500
234678-HxCDF	500
1234678-HpCDF	500
1234789-HpCDF	500
OCDF	1000

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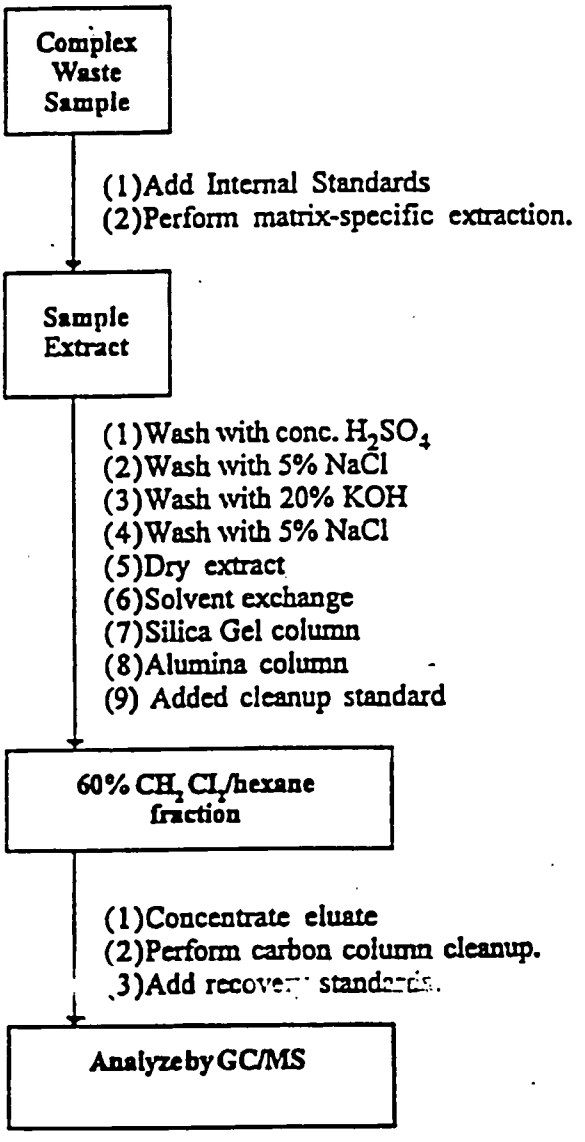


Figure 1
Flow Chart for Sample Extraction and Cleanup as Used for the Analysis of PCDDs and PCDFs in Complex Waste Samples

C2-191

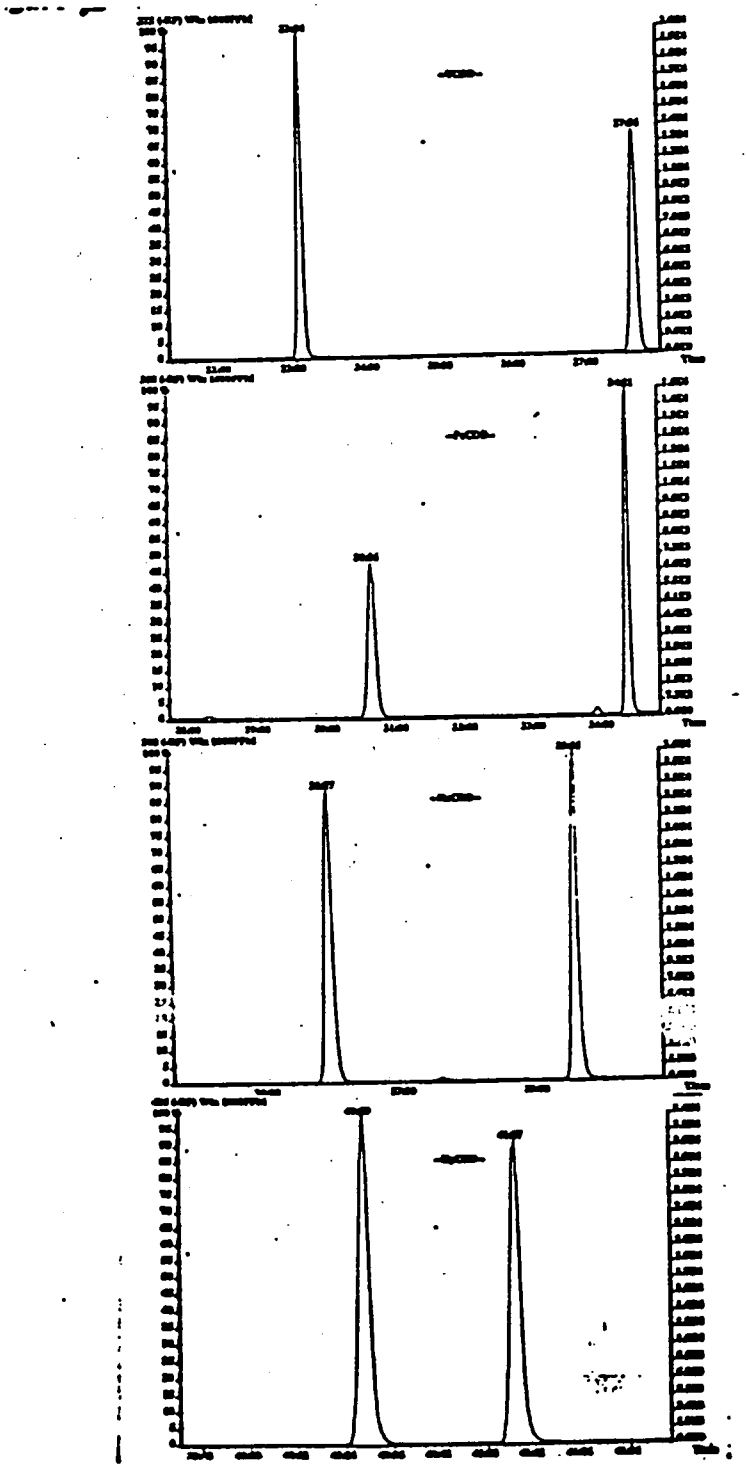


Figure 2
PCDD Isomers in the Window Defining Mix

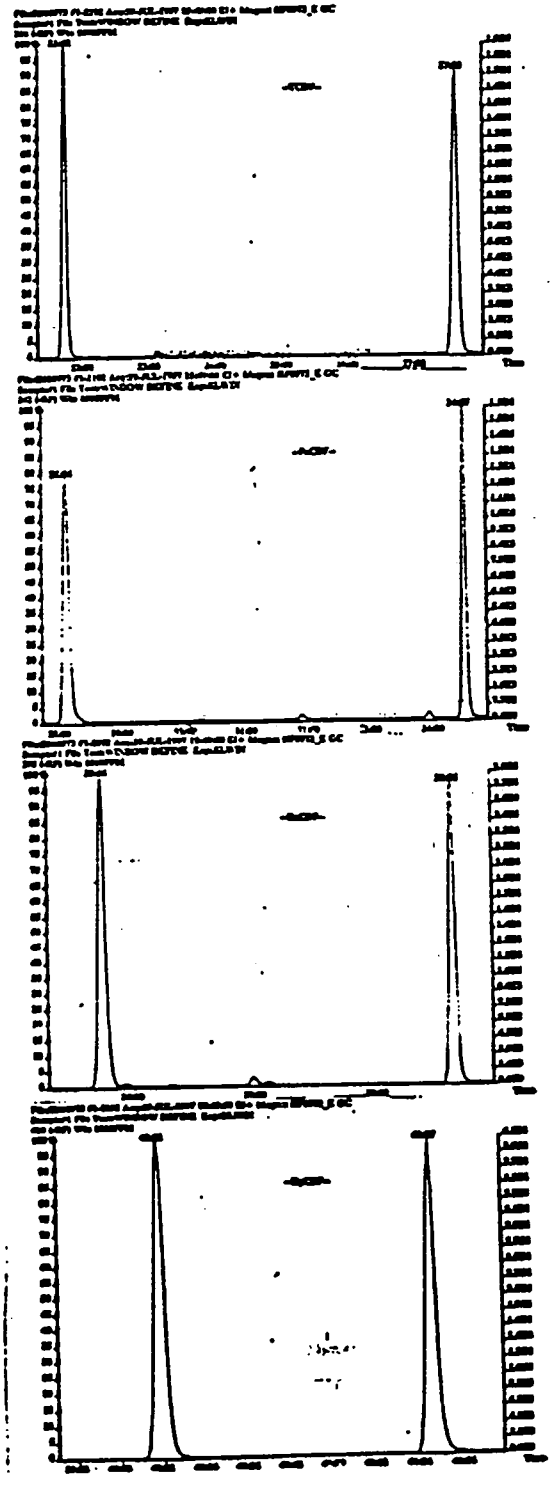


Figure 2 (Continued)
PCDF Isomers in the Window Defining Mix

Page 3 of 6

SAIS/SWL OK, INC.
Noise/Signal Height Summary

CLIENT SAMPLE ID.
LCS

Run 43 Filename [illegible] Scan: 1 Inj: 1 Acquired: 29-JUL-97 10:15:00

Processed: 31-Jul-97 21:43:47 Comments: LCS72LMD

	Typ	Name	Noise/Signal 1	Noise/Signal 2
1	Unk	2,3,7,8-TCDF	2.000e+01	1.600e+01
2	Unk	1,2,3,7,8-PeCDF	2.000e+02	1.120e+02
3	Unk	2,3,4,7,8-PeCDF	2.000e+02	1.120e+02
4	Unk	1,2,3,4,7,8-HxCDF	1.000e+02	2.000e+01
5	Unk	1,2,3,6,7,8-HxCDF	1.000e+02	2.000e+01
6	Unk	2,3,4,6,7,8-HxCDF	1.000e+02	2.000e+01
7	Unk	1,2,3,7,8,9-HxCDF	1.000e+02	2.000e+01
8	Unk	1,2,3,4,6,7,8-HpCDF	1.000e+02	1.120e+02
9	Unk	1,2,3,4,7,8,9-HpCDF	1.000e+02	1.120e+02
10	Unk	OCDF	1.000e+02	1.120e+02
11	Unk	2,3,7,8-TCDD	1.200e+01	1.200e+01
12	Unk	1,2,3,7,8-PeCDD	1.300e+02	1.040e+02
13	Unk	1,2,3,4,7,8-HxCDD	1.120e+02	1.000e+02
14	Unk	1,2,3,6,7,8-HxCDD	1.120e+02	1.000e+02
15	Unk	1,2,3,7,8,9-HxCDD	1.000e+02	1.000e+02
16	Unk	1,2,3,4,6,7,8-HpCDD	1.000e+02	1.000e+02
17	Unk	OCDD	1.000e+02	1.000e+02
18	IS/RT	13C-2,3,7,8-TCDF	1.498e+04	1.953e+04
19	IS	13C-1,2,3,4,6,7,8-HpCDF	1.326e+04	1.777e+04
20	IS	13C-2,3,7,8-TCDD	1.241e+04	1.699e+04
21	IS	13C-1,2,3,6,7,8-HxCDD	4.331e+04	3.673e+04
22	IS	13C-OCDD	8.142e+04	9.360e+04

S/N = X/Y

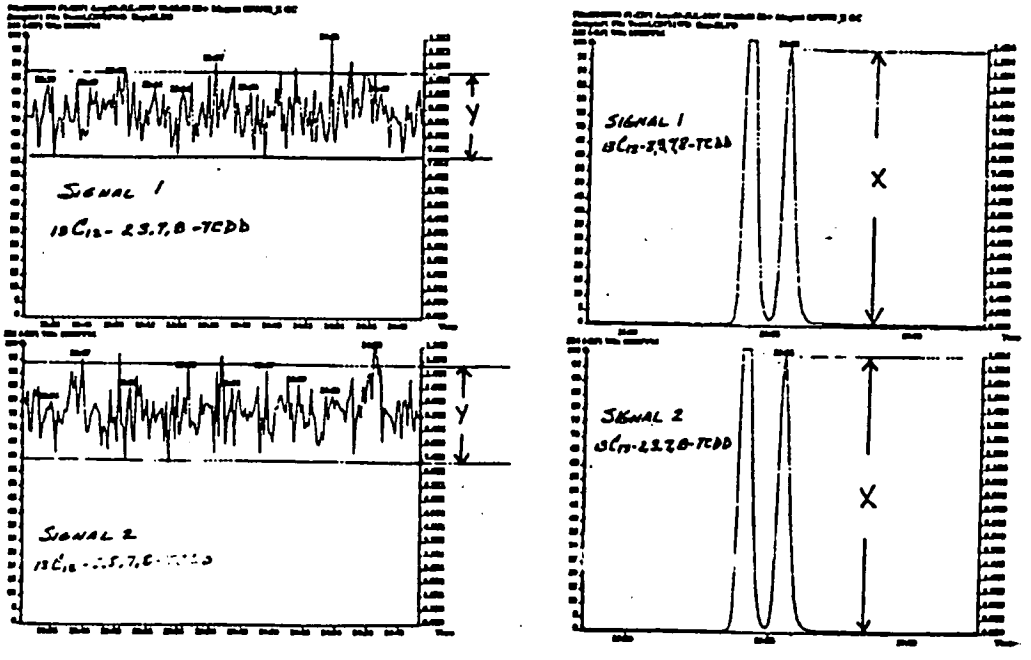
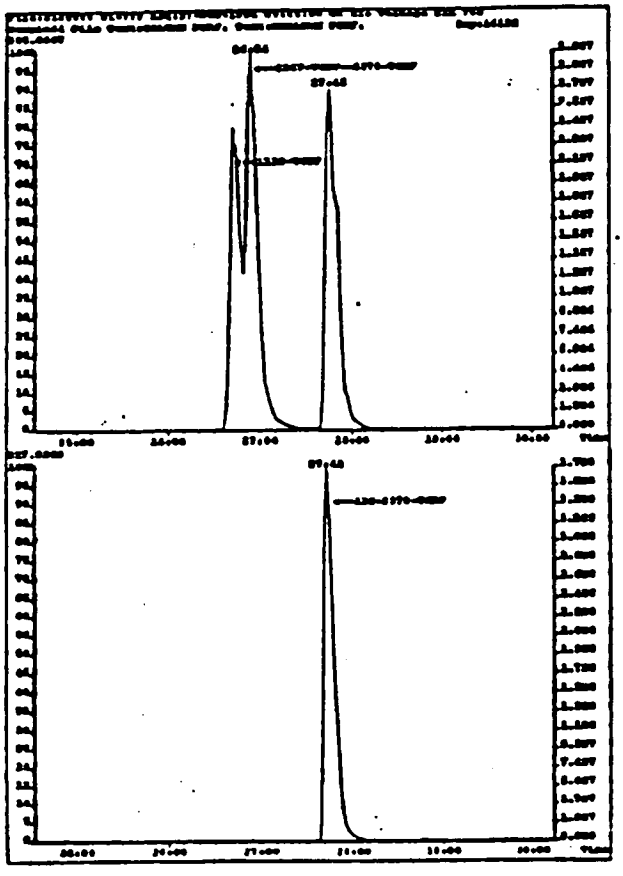


Figure 3
Signal to Noise Measurement
S/N Ratio Determined by Data System

C2-194

SP-2331 Column:
2,3,7,8-TCDF and
other closely eluting isomers.



DB-5:
2,3,7,8-TCDD and
other closely eluting isomers.

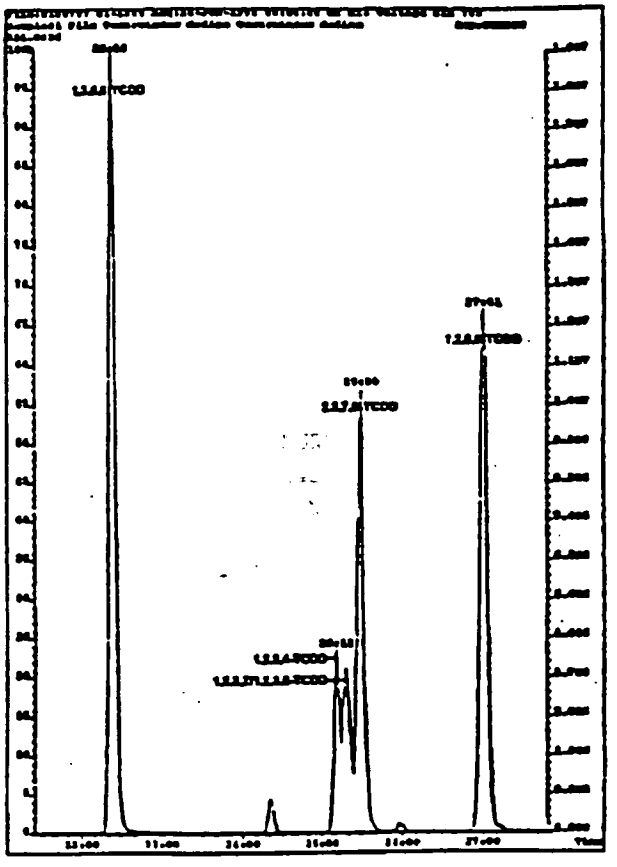


Figure 4
Window Define and Column
Performance Resolution Check

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Request for New or Revised Procedure, SWL-GA-101

SWL SOP Rev. # 1.0 - 05/14/97; Form ID: GA101-DOCREQ-F: Location/File ID: F:\SOP\GA\101-ATT5

REVISED (includes Forms/Logs) Submitted By: SKG Date: 09/30/97

Document Title: Analysis of Polychlorinated Dibenzo-p-dioxins and Polychlorinated Dibenzofurans By HRGC/HRMS (METHOD 8290)

Document No.: SWL-0.D-205 Rev. No. 4.0 — 09130197

Reference: Page ____ of ____ Section: ___.___; Subpart: _____

Attachment No. _____ — Title: _____

Brief Description (need for revision): _____

Approved: _____ Dated: _____ Denied: ☐ PM/SS Initials: _____
Program Manager/Section Supervisor (see attached explanation)

NEW (Procedural Document) Submitted By: _____ Date: _____

Justification for New Procedure: _____

Approved: _____ Dated: _____ Denied: ☐ PM/SS Initials: _____
Program Manager/Section Supervisor (see attached explanation)

Author: _____ Assigned By: _____ Date: _____

Review/Approval by Manager/Supervisor

Draft No. ___ Author's Initials: ____ Date: ____
Reviewed: ☐ Revise: ☐ Approved: ☐
PM/SS Initials ____ Date ____ (revise as marked)

Draft No. ___ Author's Initials: ____ Date: ____
Reviewed: ☐ Revise: ☐ Approved: ☐
PM/SS Initials ____ Date ____ (revise as marked)

Draft No. ___ Author's Initials: ____ Date: ____
Reviewed: ☐ Revise: ☐ Approved: ☐
PM/SS Initials ____ Date ____ (revise as marked)

Final Draft # ___ Routed to QA Dept. : Date _____ Initials ____

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Reviewed: ☐ Revise: ☐ Approved: ☐
QA/QC Initials ____ Date ____ (revise as marked)

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Reviewed: ☐ Revise: ☐ Approved: ☐
QA/QC Initials ____ Date ____ (revise as marked)

Draft No. ___ Author's Initials: ____ Date: ____
Reviewed: ☐ Revise: ☐ Approved: ☐
QA/QC Initials ____ Date ____ (revise as marked)

QA-Formal Document Completed: _____ Initials: ____
Date Rec'd by Document Control: _____ Initials: ____

C2-196

SOUTHWEST LABORATORY OF OKLAHOMA, INC.

TM

Standard Operating Procedure

Analysis of Semivolatile Organics by SW846-8270C

Document No.: SWL-OS-500

Rev No. / Date: Rev. 4.2 — 01/19/98

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APPROVALS	DATES
Harry M. Borg Procedure Prepared By	1-20-98 Date
Harry M. Borg Program Manager or Section Supervisor	1-20-98 Date
[illegible] Laboratory Safety Officer	1/21/98 Date
Chuck Hones Laboratory QA/QC Officer	1-20-98 Date

(Effective Date is 5 calendar days after the last signature above - QA/QC Officer)

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ANALYSIS OF SEMIVOLATILE ORGANICS BY SW846-8270C

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1.0 SCOPE AND APPLICATION

1.1 Method 8270C (GC/MS full scan mode) is used to determine the concentration of various organic compounds in extracts prepared from all types of solid waste matrices, soils, oils, and ground/waste waters.

This method is used to quantitate organic samples that are soluble in methylene chloride and are capable of being eluted without derivatization as sharp peaks from a gas chromatographic fused-silica column (Restek XTI-5 or equivalent.)

1.2 Practical Quantitation Limits (PQL) for water analytes under this method have a typical range of 10 ug/L to 50 ug/L, and for solids 330 ug/Kg to 1600 ug/Kg. These limits are highly matrix dependent and PQLs are proportionately higher for sample extracts that require cleanup or dilutions to avoid saturation of the detector. A list of the normal PQLs and compounds performed by this method is given in Table 10.

2.0 METHOD SUMMARY

2.1 Method — This SOP (SWL-OS-500) is based on SW846-8270C, Revision 3, 1997, and has been written into a procedure for Southwest Laboratory of Oklahoma, Inc. This SOP is used for analyses of the following SWLO Test Codes: MS500, MS510, MS530, MS540, MS550, MS599, MS505, MS507.

2.2 Procedure — A measured volume of aqueous liquid or weight of solid sample is extracted with methylene chloride. For water samples the pH is adjusted if needed to greater than 11 for the initial extraction process; and then again at less than 2. The methylene chloride extract is dried and concentrated to a volume of 1.0 mL. For soil samples, the sample is dried with sodium sulfate and then sonicated with a 50-50 acetone-methylene chloride solvent and concentrated to a final extract of 1 mL. Chromatographic conditions are described which permit the separation and measurement of the parameters in the extract. Qualitative identification is performed using the retention time and the relative abundance of one to three characteristic ions. Quantitative analysis is performed by 5-point calibration and continuing calibration checks.

2.3 Extraction QC — With each batch of 20 samples or less, a blank, a laboratory control spike and a matrix/matrix spike duplicate (if sufficient sample is sent) must be prepared and analyzed.

3.0 HEALTH AND SAFETY

3.1 All samples and standards received by the laboratory should be considered hazardous; thus extreme care must be taken when handling. Care should be taken to ensure exposure by contact or inhalation is limited. All samples are to be handled in accordance with the laboratory's Safety SOP, and Hazardous Waste SOP.

3.1.1 Safety glasses and gloves (Laytex Powder Free) and lab coats should be utilized when prepping samples for analysis.

3.1.2 Fume Hoods, and/or respirators with organic vapor cartridges should be used for extremely loaded samples.

3.2 MSDS sheets are kept in the bench area for quick reference for all standards used in each department.

3.3 Carbon Traps are to be on all GC/MS instruments on the vent outlets to filter solvent and sample vapors being discharged.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING AND STORAGE

4.1 Containers

4.1.1 All samples are received from the extractions department in 1, 5, 10mL Borosilicate glass vials with black phenolic caps and Teflon lined septa.

4.1.2 All samples received by the extraction department are grouped in episodes and kept in sample vial file boxes.

4.1.3 An aliquot of each sample is transferred to a 1.8mL glass crimp top vial with a 250uL insert and sealed with an aluminum/Teflon/rubber septa cap for analysis.

4.2 Handling and Storage

4.2.1 The extraction personnel delivering the samples with extraction logs (Attachment 4) log all samples and QC in the Extract Receipt Log Attachment (see Attachment 5).

4.2.2 Once received from extractions the samples and QC are then logged in and checked off the extract receipt log by the GC/MS sample custodian.

4.2.3 All samples and QC are then placed in vial files in numerical order by episode and sample number, and stored in a refrigeration unit that is monitored daily with a NIST calibrated mercury thermometer and kept between 1°C to 4.4°C.

4.2.4 All standards used for analysis are received, handled and stored in accordance to the Laboratory's Standards SOP and Chemical Hygiene Plan.

5.0 **INTERFERENCE AND POTENTIAL PROBLEMS** — Interference and Potential Problems may be caused from the following:

5.1 Phthalate contamination from improper use of or type of gloves and other plastics.

5.2 Matrix contamination from high levels of non-target compounds found in sample.

5.3 Power spikes or surges from the electrical supply to the instrument

5.4 Dirty syringe on autosampler. Syringe should be checked daily for signs of wear.

5.5 Dirty, contaminated, or empty wash vials on autosampler.

5.5.1 Wash vials should be rinsed and refilled daily with clean solvent.

5.5.2 Wash solvent waste vials should be emptied daily

5.6 Insufficient disk space on data system

5.7 Improper capping of sample vials may cause evaporation of sample.

5.8 Failure of refrigeration units.

6.0 EQUIPMENT/APPARATUS

6.1 The GC/Mass Spectrometer (HP 5970, 71, 72, or 73, or equivalent, Quadropole MS) is capable of scanning from 35 to 500 amu every 1 second or less, utilizing 70 volts (nominal) electron energy in the electron impact ionization mode and producing a mass spectrum which meets all required criteria when 50ng of decafluorotriphenylphosphine (DFTPP) is injected through the GC inlet.

6.2 HP 5890 and HP 5890 Series II gas chromatographs in conjunction with the above mentioned GC/Mass Spectrometer.

- The recommended GC operating conditions to be used are as follows:

Initial Column Temperature Hold	—	40°C for 1 minute
Column Temperature Program	—	18°C/min. to 100°C
		15°C/min. to 325°C
		Hold 2.5 min.
		40°C/min. to 350°C
Injector Temperature	—	280°C
Transfer Line Temperature	—	280°C
Source Temperature	—	According to manufacturer's spec's.

NOTE: These conditions will vary depending on column and instrument.

6.3 HP7673 autosamplers with 100 vial capacity used for sample introduction into the HP5890/HP5970, 71, 72, 73 system.

6.4 Column — 30m x 0.25mm ID (or 0.32mm) bonded-phase silicone coated fused silica capillary column (Restek XTI-5 or equivalent.) A film thickness of 0.25 microns is used. A film thickness of 1.0 microns is recommended if a large loading capacity is needed.

6.5 Personal Computers equipped with ChemStation Software (HPChem) for the aquisition of sample data.

6.6 Computers equipped with Target Version 3.0 software for the processing and review of acquired data.

6.7 HP LaserJet 3SIII/4S1 printers for data hardcopies.

7.0 REAGENTS

7.1 Methylene Chloride — HPLC or Pesticide grade

7.1.1 Samples brought to final volume in Methylene Chloride for semi-volatile analysis.

7.1.2 Used as dilution solvent when dilutions are required on a sample.

7.1.3 Main solvent used in calibration standards and internal standard.

7.2 Initial Calibration Standards (See Table 11) — EPA traceable standards. All standards stored in original containers under temperature requirements as dictated by manufacture, (ambient or refrigerate). Once opened all standards are placed in amber borosilicate glass vials sealed with Teflon lined caps, and stored at -10°C to -20°C. All standards prepared in accordance with the Laboratory's Standards Preparation SOP.

NOTE: Equivalent EPA traceable standard by other manufacturers may be substituted.

7.2.1 S.V. Calibration Mix #1 (Restek)

7.2.2 S.V. Calibration Mix #2 (Restek)

7.2.3 S.V. Calibration Mix #3 (Restek)

7.2.4 S.V. Calibration Mix #4 (Restek)

7.2.5 S.V. Calibration Mix #5 (Restek)

7.2.6 S.V. Calibration Mix #7 (Restek)

7.2.7 Acid Surrogate Mix (Restek)

7.2.8 Base/Neutral Surrogate Mix (Restek)

7.2.9 Benzidines Mix (NSI)

7.2.10 Benzoic Acid (NSI)

7.2.11 Pyridine Stock (Chem. Service)

7.2.12 2,3,4,6-Tetrachlorophenol Stock (Chem. Service)

7.2.13 1,2,4,5-Tetrachlorobenzine Stock (Chem. Service)

7.2.14 Aniline Stock (Chem. Service)

7.2.15 Azobenzene Stock (Chem. Service)

7.2.16 1-Methyl Naphthalene Stock (Chem. Service)

7.2.17 (DFTPP) Decafluorotriphenylphosphine (protocol)

NOTE: Individual compound stock standards prepared from NEAT. (Table 12)

7.3 Internal Standard Mix — S.V. CLP Internal Standard Mix (Absolute) (See Table 11).

7.3.1 Consisting of 1,4-Dichlorobenzene-d_4, Naphthalene-d_8, Acenaphthene-d_{10}, Phenanthrene-d_{10}, Chrysene-d_{12}, Perylene-d_{12}.

7.3.2 Stored at -10°C to -20°C in amber glass vials with Teflon lined caps.

7.3.3 Internal standard diluted to a concentration of 1000ug/ml in methylene chloride is added to all samples and at 40uL per 1ml of sample extract to give a concentration of 40ng/ul for each 1ml sample.

7.4 Ultra high purity helium used as a carrier gas to all instruments

8.0 PROCEDURE

8.1 Daily procedure

8.1.1 Check gas cylinders in the beginning of the day to ensure proper pressure and adequate abundance of gas

8.1.1.1 Change gas cylinder if amount falls below 500psi.

- Check each instrument assigned to cylinder to be changed and make sure all of the GC ovens are at ambient temp (40-50°C), and no instruments are in the middle of analysis.
- Place full cylinder next to empty one in as close proximity to where it will stay as possible.
- Make sure empty cylinder being removed has the valve completely shut off.
- With large crescent wrench unscrew regulator from cylinder as quickly as possible and immediately place on full cylinder, tighten and turn valve on. Open valve as far as it will go and back off ¼ turn to prevent valve sticking. This procedure should be completed as quickly as absolutely possible to minimize ambient air entering the gas lines and to avoid total loss of gas pressure to the instruments.
- Check fittings on regulator with snoop (soapy water) for air tightness. If there is a leak at fitting the snoop will bubble, if this occurs, check fitting and tighten or replace as necessary.

8.1.1.2 Log in cylinder pressure (PSI) on gas monitoring sheet.

8.1.2 Check refrigerator/freezer unit where samples and standards are stored.

8.1.2.1 Using an NIST Calibrated Mercury Thermometer check that the

temperatures are being held at 1°C to 4.4°C for the refrigerator and -10°C to -20°C for the freezer.

8.1.2.2 Log temperature for each in the Zone Calibration Check Refrigeration Log Book to the nearest 0.5°C.

8.1.3 Set daily working standards to be used for the day out of freezer to warm to room temperature before analysis.

8.1.4 Rinse and refill solvent vials used for sample preparation and dilutions. Empty waste vials. These vials are kept at sample prep tables.

8.2 Analytical Procedure

8.2.1 Check instrumentation for any obvious problems (i.e., broken syringe, power failure).

8.2.2 If samples analyzed the night before were dirty, set injection port and GC oven to cool to do daily maintenance. Turn off injection port. Set oven to 30°C.

8.2.3 Briefly review the previous day's analyses by processing the samples in accordance with the Target Processing GC/MS SOP, check for any major system problems (i.e., broken column, air leak, blown filament, etc.) and check to confirm that the previous sequence has been completed. If problems exist, inform supervisor and consult for corrective action. If you are operating two instruments, continue operating the functional instrument.

8.2.4 Perform daily maintenance on instrument injection port (Attachment 10) and column. Care must be taken to not touch any parts with bare hands unless absolutely unavoidable. Use tweezers for placement of new parts.

8.2.4.1 Before opening the closed injection port system make note of carrier gas pressure on the HP5890GC/HP5970 MSD and the electronic pressure controller (EPC) on HP5890/HP5971/HP5972 MSD. On the HP5971/72 make note of pressure at a given temp (i.e., 50°C → 7.7 pressure) and then set pressure to zero.

8.2.4.2 Change septa. Remove septum retainer nut and change out septa using tweezers. Save old septa to plug disconnected column; replace septum retainer nut finger tight. Do not over tighten, over tightening puts too much pressure on septum and allows syringe needle to push out chunks of the septum clogging the injection port liner and causing a leak.

8.2.4.3 Loosen insert weldment assembly and remove injection port liner and o-ring.

8.2.4.4 Disconnect column from injection port. Remove column nut and old ferrule from column. Use old septum to plug off column, to avoid excessive air being pulled through the system.

8.2.4.5 Remove insulator cup and reducing nut from injection port. Discard used seal and washer from inside reducing nut. Replace with cleaned or new seal and washer. Use tweezers to handle seals and washers. Do no touch with hands. Rinse new seal and washer with methylene chloride before placing back in reducing nut. Double check to make sure washer is on the bottom of the reducing nut with the seal, tip down, on top of washer. (Tip on seal should be down towards small end of reducing nut). Replace reducing nut, tightening completely, and replace insulating cup. Insulation inside insulating cup should be intact and relatively snug around reducing nut.

8.2.4.6 Install new injection port liner into top of injection port, using tweezers or forceps. Put a new o-ring around top of injection port liner. This can easily be done using two tweezers and pulling o-ring around liner. Make sure o-ring is as flat as possible against the injection port shell weldment. Tighten insert weldment assembly back down onto shell weldment. Make sure insert weldment is properly aligned with the tab in the back down in the large hole on shell weldment and straight over the threads. Tighten completely.

8.2.4.7 Re-install column to injection port. Place column nut back on column and put tapered .4mm ID Graphite/Vespel Ferrule on column, with wide end of Ferrule fitting into column nut. Clip column. If dirty samples were analyzed, 8-10 inches should be clipped off column. If clean samples were analyzed, clip 4-8 inches off. Extreme care must be taken while clipping a column to ensure a flat, even cut on end of column. Set column 4-5 mm above top of Ferrule and mark position under column nut with white out. Screw column nut back up into reducing nut on injection port, being careful to reset column according to white out mark to ensure proper column height within injection port liner.

8.2.4.8 Gently tighten detector interface nut on detector end of column to prevent air leak at this point. Do not over-tighten.

8.2.4.9 Once all maintenance is completed and returned to a closed system, check carrier gas pressure on dial on HP5890/HP5970 (to make sure it has returned to the same pressure as before daily maintenance), to ensure no major obvious air leaks are occurring, and reset EPC pressure on HP5890/HP5971/72 (i.e., 50°C oven temp = 7.7 pressure). If EPC pressure does not stabilize at set point within a few seconds there is probably a leak at some point where maintenance was just completed.

8.2.4.10 Turn injection port temperature back on. Turn oven temperature to 150°C and let bake for 10 to 15 minutes. This will facilitate ridding the instrument of any air/water within the system from the column being open.

8.2.5 Prepare an aliquot of DFTPP performance check solution (Table 13) and continuing calibration standard (SSTD050, Table11) while instrument is baking out. Also during this time check disk space on the chem station and the target system to ensure adequate disk space to acquire data for a twelve hour time period for all instruments on the data system.

8.2.5.1 In a 1.8mL clear glass crimp top vial with a 250uL glass insert add approximately 30uL of 50ng DFTPP solution. 30uL should be sufficient for several injections if needed. Do the same with the current continuing calibration solution. Label vials with solution name, solution lab ID number, (i.e., DFTPP 1-208-12; SSTD050 1-213-12).

8.2.5.2 Check disk space on chem station as follows:

- Double click Program Manager icon
- Double click Main icon in Program Manager dialog box
- Double click File Manager icon in main dialogue box
- Single click on C: drive
- Double click HPchem folder
- Double click on instrument number for one of the instruments on data system (i.e., 1=instrument A 2=instrument S)
- Single click on data folder branched under instrument number. Read in lower left corner of dialogue box the number of KB free (i.e., C: 181,344 KB free, 322,760KB total)

8.2.5.3 Check disk space on target system as follows:

- Single click on hpterm icon on controller box on bottom of target screen
- Type df –t in lower case letters in hpterm dialogue box at the $ prompt.
- Take note of number of blocks remaining for whichever disk your data system is on (i.e., /disk, /disks)
- Data on Target system should be copied to data tapes at least every 10 days never delete data off of target system until it is confirmed to be on tape and review and data package/forms are complete.

8.2.6 Inject 1uL of the 50ng DFTPP solution.

8.2.6.1 Fill in daily information in Run Logbook. (See Run LogBook SOP).

8.2.6.2 Fill in information for DFTPP run.

- Analysts initials
- Client sample ID (DFTPP)
- Lab ID (1-208-12)
- File ID (a 9865)
- Volume Injected (1uL)

8.2.7 While DFTPP is running, begin reviewing previous day's analysis that was previously processed in Section 8.2.3. (See Data Review SOP SWL-OS-103 Rev.3). Check each sample analyzed for the following:

8.2.7.1 Internal standards (istd.rp)

8.2.7.2 Surrogate/Spike recoveries (spike.rp) (See Table 2 for surrogate and QC-Blue-Book for up to data spike recoveries.)

8.2.7.3 Linearity and saturated peaks (target.rp)

8.2.7.4 All samples within 12 hour clock

8.2.7.5 Major analytical problems with samples (i.e., run terminated early; air leaks).

Note: Each of the above items may be checked by clicking on data file number, section to be checked and then open view (i.e., chem/j i/J970625A.D/JJV425.d/ISTD.RP) (Attachment 11)

8.2.7.6 Remedial Action If Criteria Are Not Met

- Waters: If peaks are saturated, the sample must be diluted to get the highest-level target compounds within linear range and reanalyzed.
- Soils: If peaks are saturated, the sample must be diluted to get the highest-level target compounds within linear range. If a dilution greater than 30 fold is required, then sample may need to be re-extracted as a medium level extract and reanalyzed. Consult supervisor for instructions. (IS intensities should be at least 10% or greater in the sample for correct dilution.)
- If any target analyte has a concentration above 1000ng on column, then following sample must be checked for carry-over. (See Data Review SOP).
- If sample is analyzed at medium level, laboratory control spike and blank analyses must also be performed for that level.

- If surrogate recovery for semivolatiles in any sample is less than 10%, the sample must be re-extracted and reanalyzed. If two or more acid surrogates or two or more base/neutral surrogates are outside of QC limits in any sample, the sample must be re-extracted and re-analyzed. If one acid surrogate and/or one base surrogate is outside of QC limits, and the recovery is greater than 10%. The data must be flagged and noted in clients narrative.

  NOTE: Clients QAP or QAPP must be followed, which in some cases does not allow for any surrogates outside QC limits without re-extracting.

  No surrogates can be out of QC limits for extraction blanks. If so, all samples extracted with blanks that have surrogates out must be re-extracted. Both sets of data will be reported if the re-extracts are not completed within the required holding times (consult the section supervisor).

- Extraction Blank Contamination: Extraction Blanks cannot have target analytes of interest above the PQL. The only exceptions could be the common laboratory contaminates of phthalate, which could not be detected above 5 times the PQL. Anytime phthalate is detected above the PQL the blank must be reanalyzed and verified by supervisor as usable.

  NOTE: Clients QAP or QAPP must be followed, which in some cases does not allow for any analytes to be above reporting levels.

  When any other target analytes in blank are above PQL, all associated samples containing these analytes must be re-extracted. See supervisor for directions on any contaminated blanks.

  NOTE: All Blank contamination must be reported.

- All samples that are re-extracted must be logged-in on the "Re-extract Tracking Sheet" (Attachment 1), and turned into the Supervisor.

- If re-extraction is required for a sample, fill out a Non-Conformance Report (GA-105-NCR-F Attachment 3). Include a copy of the extraction sheet and a copy of spike report or tabular report documenting why sample is being re-extracted. Non-Conformance Report must be signed by Organic Program Manager/Supervisor/Group Leader prior to being submitted to extractions department.

8.2.8 Note the following in the Run Log Book (Attachment 6) for each run analyzed:

8.2.8.1 Run time for each run made

8.2.8.2 Internal standards pass/fail (ex. 2L=2int.stds.low)

8.2.8.3 Surrogate recoveries pass/fail (ex. 2H=2 surrogates high)

8.2.8.4 Compounds within linear range

8.2.8.5 Any comments pertinent to each particular sample (i.e., rerun, dilution needed, re-extract needed, out of clock).

8.2.9 When the DFTPP run is complete, check to see if the tuning criteria has been met (see Table 1), and that all peaks in DFTPP solution are accounted for and less than 20% 4,4-DDT breakdown is present (see Formula 9.6), or poor recovery of pentachlorophenol, or tailing of benzidine (Attachment 7, 8, and 9). NOTE: Benzidine and pentachlorophenol should be present at their normal responses, and no peak tailing should be visible. If so, start the standard analysis, and print the DFTPP report. Note in the Run Log Comment section that DFTPP passed. If DFTPP does not pass, re-shoot DFTPP or tune the mass spec first, then shoot DFTPP. If the tune does not pass after 3 attempts, consult your supervisor for instruction.

8.2.10 Inject 1uL of the continuing calibration, or if an initial calibration is being performed, set up a sequence for standards in the calibration. Start the sequence running the continuing calibration and prep and load the remaining standards.

- Fill in Run Log Book with standard information

8.2.11 If the standard is a continuing calibration, not an initial calibration running, set out 3-5 samples (either samples requiring re-analysis, or samples being initially run) to warm to room temperature. Add internal standards to samples if necessary once all have warmed. Prepare an aliquot of each sample for injection.

NOTE: All vials for sample analysis must be labeled with entire lab id number (i.e., 29859.02, BL0618WA-29859).

8.2.11.1 Internal standards solution is added to each sample at 40ng/uL on column. Add 40uL of the 1000ng/uL solution to each 1.0mL extract of sample to achieve the appropriate 40ng/uL concentrations.

8.2.12 When the continuing calibration has finished, check to see if the acceptance criteria has been met (Table 8 & 9) and that the data system has correctly identified all isomeric compounds (Tables 5 & 6). If so, load the 3-5 samples, and start the sequence. If the standard does not pass, and if no major problems exist (e.g., very low RFs for SPCCs <0.05, % Deviation> 20 for CCC etc.), rerun the continuing calibration. If the second calibration does not pass, set up an initial calibration sequence. All standards should be reviewed immediately after analysis to make sure it passes before samples are analyzed. Submit for secondary review after checking standard.

- Fill in Run Log Book with sample data for all samples set up in short sequence.

8.2.13 While the short sequence of samples (or initial calibration) is being analyzed, continue reviewing previously processed data.

8.2.14 Print each sample as it is reviewed. Each sample is to be assembled in the following order

8.2.14.1 Sample Report (CLP.RP) (Form 1 type report)

8.2.14.2 Surrogate/Spike Report (SPIKE.RP)

8.2.14.3 Internal Standard Report (ISTD.RP)

8.2.14.4 Total Ion Chromatogram

8.2.14.5 Target Quant. Report (TARGET.RP)

8.2.14.6 Manual Integration

8.2.14.7 Target Compound Spectra

8.2.14.8 TIC Quant. Report (As required) (TICQUANT.RP)

8.2.14.9 TIC Spectra (As required)

8.2.15 Print a SAMPLE.X.MAP for each episode ran during the 12-hour clock.

8.2.15.1 Highlight runs for each episode as follows:

- DFTPP — yellow
- Calibration — orange
- Samples — green

8.2.15.2 Write the following information on the upper right corner of SAMPLE.X.MAP:

- Client Name
- Episode Number
- Test Code
- Matrix
- Instrument ID

8.2.15.3 Double check all samples are within 12-hour clock on SAMPLE.X.MAP.

8.2.15.4 If there is a problem with any runs, note in right margin what it is (ex. 2 internal stds low, 2 surrogates low, rerun @2x, re-extracting).

8.2.15.5 Check that QC Group Ref. # is the same for all: DFTPP, calibrations, samples.

8.2.15.6 Check that all samples match the calibration number for the initial cal. ref. #

8.2.15.7 Use SAMPLE.X.MAP to double check client Ids, and Lab Ids.

8.2.15.8 Check lab Prep Batch for each episode.

8.2.15.9 Double check that correct method and sublist was used.

8.2.16 Upon completion of data review, determine the remaining samples to be analyzed. (Re-analysis or dilutions from reviewed data or new samples.)

8.2.16.1 Set samples out to warm to room temperature.

8.2.16.2 When samples have warmed to room temperature, add internal standards, if not previously added, or do dilutions as necessary.

8.2.17 Add additional samples to be analyzed to short sequence already running.

8.2.18 After entire sequence has been typed in, pause the sequence between runs and save the sequence on the chem station. Print a hard copy of the sequence to check for correctness and proper vial placement. Restart sequence where it was paused. Keep hard copy of sequence with Run LogBook.

8.2.19 Fill in the information for the additional samples in the run log book.

8.2.20 Complete Run Log Book for data previously processed by lining through and initial and date any unused portions of the page and sign book.

8.2.21 For any corrections or errors made cross out error with a single line and initial and date.

8.2.22 After each sample has been printed and put together as in Section 8.2.14 (but entire episode is not complete), keep each sample for an episode together with the SAMPLE.X.MAP for that clock.

8.2.22.1 Attach a note to each group of data stating why it is not complete and what is remaining on it (ex. Rerun 29864.02 @ 10X, 29864.01 sent for re-extract, 29864.06 - .12 running now, etc.)

8.2.22.2 Keep sample extraction sheet with data.

8.2.23 Completed episodes must be assembled and turned in for secondary review in the following order:

8.2.23.1 Extraction Sheet (Attachment 4).

8.2.23.2 Copies of Run Log Book pages pertaining to the episode for data needing CLP like forms (for each page with QC, samples, calibration, initial calibrations).

8.2.23.3 Data Review Sheet (see Attachment 2)

8.2.23.4 Back-up Blank Sheet (If needed) (See Attachment 12 and Back-up Blank SOP)

8.2.23.5 SAMPLE.X.MAPS for each day samples were analyzed.

8.2.23.6 Sample Data

- SBLK
- LCS/LCSD
- Samples

8.2.23.7 Re-extracts (if needed) arranged in same order as original data.

8.2.23.8 Extra Data

8.2.24 As time permits, data should be worked up as it is running. Data ran overnight or during another shift should be processed and reviewed the following shift as stated beginning in Section 8.2.3.

9.0 CALCULATIONS

9.1 Water

$$\text{Concentration (ug/L)} = \frac{(Ax)\ (Is)\ (Vt)}{(Ais)\ (RF)\ (Vo)\ (Vi)}$$

Where:

Ax	=	Area of the characteristic ion for the compound to be measured.
Ais	=	Area of the characteristic ion for the internal standard.
Is	=	Amount of internal standard injected in nanograms (ng).
Vo	=	Volume of water extracted in mililiters (mL).
Vi	=	Volume of extract injected (uL).
Vt	=	Volume of total extract (uL).
RF	=	Average Response Factor

9.2 Sediment/Soil

$$\text{Concentration (ug/Kg)} = \frac{(Ax)\ (Is)\ (Vt)}{(Ais)\ (RF)\ (Ws)\ (Vi)\ (D)}$$

(Dry weight basis)

Where:

Ax, Is, Ais = Same as given for water, above.
Vt = Final volume of low level total extract (uL).
Vi = Volume of extract injected (uL).
D = $\frac{100 - \%\ moisture}{100}$
Ws = Weight of sample extracted (grams).
RF = Average Response Factor

9.3 An estimated concentration for Non-TCL components tentatively identified shall be quantified by the internal standard method. For quantification, the nearest internal standard shall be used.

9.4 Internal Standard added to all samples

$$\frac{1000ng/uL\ Internal\ Standard\ Solution\ x\ 40uL}{1000uL\ Sample} = 40ng/uL\ per\ sample$$

9.5 Common Sample Dilutions

- 2x = 500uL sample + 500uL CH_2Cl_2 + 20uL Int.Std.
- 5x = 200uL sample + 800uL CH_2Cl_2 + 32uL Int.Std.
- 10x = 100uL sample + 900uL CH_2Cl_2 + 36uL Int.Std.
- 20x = 50uL sample + 950uL CH_2Cl_2 + 38uL Int.Std.
- 50x = 20uL sample + 980uL CH_2Cl_2 + 40uL Int.Std.
- 100x = 10uL sample + 990uL CH_2Cl_2 + 40uL Int.Std.

9.6 % Breakdown for DDT

$$\%\ BD = \frac{DDE_{(PA/PH)} + DDD_{(PA/PH)}}{DDT_{(PA/PH)} + DDE_{(PA/PH)} + DDD_{(PA/PH)}} \times 100$$

BD = Breakdown
(PA/PH) = Peak area or peak height of analyte.
NOTE = % breakdown of DDT in DFTPP must be less than 20%.

9.7 See Table 14 for remaining calculations, for linearity, Quadrate, surrogate recovery, matrix spike and lab spike recovery, and percent RSDs.

10.0 QUALITY ASSURANCE/QUALITY CONTROL

10.1 Extraction Blanks and Laboratory Control Spikes are extracted at a minimum of 5% of samples being extracted.

10.2 Any sample or QC sample with a surrogate less than 10% must be re-extracted and re-analyzed. If two or more acid surrogates or two or more base/neutral surrogates are outside QC limits in any sample, the sample must be re-extracted and re-analyzed. If one acid and/or one base surrogate is outside of QC limits and the recovery is greater than 10%, the data must be flagged and noted in clients narrative. NOTE: Clients QAP or QAPP must be followed, which in some cases does not allow for any surrogates outside QC limits without re-extracting.

10.3 No surrogates can be outside QC limits for extraction blanks. If the extraction blank has surrogates outside QC limits, all samples extracted with that blank must be re-extracted.

10.4 Extraction blanks cannot have target analytes above the PQL. The only exceptions could be the common laboratory contaminate of phthalate, which could not be detected above 5 times the PQL. Anytime phthalate is detected above the PQL the blank must be re-analyzed and verified by supervisor as "OK" to use. NOTE: Clients QAP on QAPP must be followed, which in some cases does not allow for any analytes to be above reporting levels. When target analytes in the extraction blank and laboratory control spikes (other than spike compounds) are above the PQL all associated samples containing these analytes must be re-extracted. Consult supervisor for instructions on any contaminated QC.

10.5 Laboratory Control Spike

10.5.1 The Laboratory Control Spike (LCS and LCSD) are internal laboratory QC samples designed to show the capability of our laboratory to be in compliance with the protocol.

10.5.2 An acceptable LCS and LCSD should meet the internal standard criteria stated in Data Review SOP and the surrogate recoveries in Table 2. The percent recovery for each of the compounds in the LCS and LCSD should be within recovery limits shown in the QC Method Reference "Blue Book". Limits are derived using a control chart program as per the Control Chart SOP (SWL-GA-112).

10.5.3 If the compounds are outside QC limits, corrective action is needed immediately, beginning with rerunning the LCS to verify that it was injected correctly.

10.6 Matrix Spikes — MS/MSDs will be analyzed at a rate of 1 MS/MSD per batch of 20 or fewer samples. A minimum of the 11 compounds will be used; however all compounds can be spiked.

10.7 Each analytical instrument is calibrated daily with a DFTPP tuning standard (see Table 1) and a calibration standard with all compounds of interest.

10.7.1 Tune

10.7.1.1 Before analysis of samples can begin, the instrument must meet all calibration criteria. The first step in the calibration of a Semi-volatile GC/MS system is to demonstrate the ionization and fragmentation of standard mass spectral tuning compounds. This, as well as a sensitivity check, is accomplished with the use of

Decafluorotriphenylphosphine (DFTPP), an EPA specified compound. This standard is run daily to validate the GC/MS system and to begin the 12-hour clock. DFTPP is analyzed by direct injection of a 50ng aliquot into the instrument.

10.7.1.2 When the analysis is complete, the tune is checked against standard criteria (See Table 1 for Tune Criteria). SW8270B tune criteria for DFTPP must be met before any other calibration standards or sample analysis is performed.

10.7.1.3 If the tune fails to meet the criteria, DFTPP is rerun. If it fails again, the instrument must be re-tuned through "Auto Tune" or "Manual Tune", then DFTPP should be run again. If all criteria are met, the calibration sequence is run, but if it fails again, a supervisor is contacted because instrument maintenance such as source cleaning may be required.

10.7.2 Initial Calibration

10.7.2.1 Once DFTPP has met its tune criteria, calibration may begin. Initial calibration of the GC/MS is established and validated by the analysis of EPA traceable standards at five concentration levels. Standard levels are 10, 25, 50, 80, and 100ng/uL, but other levels may be used. These standards are made up in methylene chloride before analysis by GC/MS. The internal standard calibration procedure is used. In addition to surrogate recovery compounds, standards and sample extracts are spiked with internal standards that span the retention time range of the analytes of interest.

10.7.2.2 The Response Factors (RF) for the analytes in the standard are calculated and stored in a Target Method File. Using the RFs from the initial calibration, the percent relative standard deviation (%RSD) for all compounds is calculated utilizing the formula shown in Table 13. The criteria the initial calibration must meet to be acceptable is found in Table 8 and 9 for CCC's and SPCC's.

10.7.2.3 If the five-point does not meet criteria each standard is checked for the need of a possible rerun, in case the standards had a bad injection. If re-analyzing a standard will not solve the problem, a supervisor is contacted.

10.7.2.4 If the initial calibration does meet the criteria stated in Table 8 and 9, and there is time remaining in the 12-hour time period, samples may be run.

- If the % RSD of any compound is 15% or less, then the relative response factor is assumed to be constant over the calibration range, and the average relative response factor may be used for Quantitation.

- If the % RSD of any compound is greater than 15%. The compounds should be checked to verify that all integrations were performed properly, that each calibration level appears to be at the correct concentration levels and that the system is operating properly (i.e. normal response factors and retention times). If all of this is in order, then a calibration curve must be constructed of area ratios (A/Ais) versus concentration using first or higher order regression fit of the five calibration points (i.e., linear regression or quadratic).

  The use of calibration curves is a recommended alternative to average response factor calibration and a useful diagnostic of standard preparation accuracy and absorption activity in the chromatographic system.

10.7.3 Calibration Verification

10.7.3.1 A calibration verification of the GC/MS uses the same EPA traceable standards, but at only one level: 50ng/uL. The analysis is performed in the same manner as stated before in the initial calibration. The calibration verification checks the same compounds as the initial calibration, and the criteria the calibration verification must meet to be acceptable are also found in Table 8.and 9.

All other compounds should have a %Drift less than 30%.

10.7.3.2 The calibration verification is performed after the DFTPP and must meet all criteria before any other analysis continues. If the CCC/SPCC criteria are not met, the standard must be rerun, as it is possible, the standard was not prepared correctly or that there was a bad injection. However, if the second standard still doesn't meet CCC/SPCC criteria, and initial calibration must be run or corrective action performed.

10.7.3.3 If the calibration verification does meet the criteria stated in Tables 8 and 9, samples may be run. Samples are quantitated based on the criteria stated above in Section 10.7.2.4.

11.0 WASTE DISPOSAL

11.1 All waste solvent is collected in a designated area and disposed of by a hazardous waste department designee, in accordance with the Hazardous Waste SOP.

11.2 All sample vials are collected in designated boxes at each instrument, and periodically picked up and disposed of by hazardous waste designee and in accordance with SWLO Hazardous Waste SOP.

12.0 METHOD PERFORMANCE

12.1 Surrogate Recovery Limits (Table 2)

12.2 Matrix Spike Recovery Limits (Table 3)

12.3 Laboratory Control Spike Limits (QC Blue Book)

12.4 DFTPP – Tuning Criteria (Table 1)

12.5 Continuing Calibration SPCC/CCC Criteria (Tables 8 & 9)

12.5.1 SPCC – Semivolatile System Performance Check Compounds

12.5.2 CCC – Semivolatile Calibration Check Compounds

12.6 MDL run annually and must be below reporting limits (PQL's). All MDLs stored with section supervisor and QC Red Book.

13.0 REFERENCES

13.1 SW846-8270C, Revision 3, December 1997

13.2 Hewlett Packard, chemical analysis columns and supplies catalog 1996-97.

13.3 Semi-Volatile GC/MS Data Processing SOP, Rev. 1.0.

13.4 Semi-Volatile Data Review SOP, SWL-OS-103, Rev. 3.0.

13.5 Semi-Volatile Run Log Book SOP, SWL-OS-102, Rev. 2.0.

13.6 SWL Hazardous Waste SOP.

13.7 SWL Safety SOP.

13.8 SWL Chemical Hygiene Plan.

13.9 Guide to Semivolatile GC/MS Standards Log, SWL-OS-309.

14.0 DEFINITIONS

14.1 Terminology

14.1.1 Standard Operating Procedure (SOP): Written narratives that describe step-by-step instructions and procedures specific to the laboratory.

14.1.2 SAMPLE.X.MAP: ..... Summary of sample information displaying pertinent information about individual analytical batches. Printed from the Target System. Summary contains at a minimum Data File, Dilution Information, Analysis Method and Batch,

Lab Prep Batch, and Initial Calibration Reference Number.

14.1.3 Batch: ..... Daily analytical sequence consisting of the daily tune, calibration, method blank, QC spikes, and samples analyzed within a 12-hour time clock.

14.1.4 Extra Data: ..... Data classified as non-reportable, but stored by the laboratory with the pertinent episode information for reference purposes.

14.1.5 Extraction Sheet: ..... Information generated in Extraction. Sheet is specific to each set of samples and contains, at a minimum, the Client, Client ID, SWLO ID, pH, %Moisture, and Extraction Information.

14.1.6 Lab Prep Batch: ..... Identification assigned when the Method Blank is analyzed and used by all samples corresponding to that Method Blank.

14.1.7 Standard Tabular Report: ..... Type of data package report generated by the Reporting Department from information.

14.1.8 Sequence: ..... Same as Batch

14.1.9 Test Code: ..... Code assigned to each sample to determine which department sample is for and what method sample is to be analyzed by.

14.2 Acronyms

14.2.1 RT..... Retention Times

14.2.2 MS/MSD..... Matrix Spike/Matrix Spike Duplicate

14.2.3 TIC ..... Tentatively Identified Compound

14.2.4 MSDS ..... Material Safety Data Sheets

14.2.5 GC ..... Gas Chromatogram

14.2.6 GC/MS..... Gas Chromatography/Mass Spectroscopy

14.2.7 HP..... Hewlett Packard

14.2.8 NIST..... National Institute of Standards and Technology

14.2.9 DFTPP ..... Decafluorotriphenylphosphine

14.2.10 PQL ..... Practical Quantitation Limit

14.2.11 IS ..... Internal Standards

14.2.12 CCC ..... Calibration Check Compound

14.2.13 SPCC ..... System Performance Check Compound

14.2.14 LCS/LCSD.... Laboratory Control Spike/Laboratory Control Spike Duplicate

15.0 **TABLES** — See following pages.

16.0 **ATTACHMENTS**

16.1 ATTACHMENT 1: Re-Extract Tracking Sheet

16.2 ATTACHMENT 2: Data Review Sheet

16.3 ATTACHMENT 3: Non-Conformance Report

16.4 ATTACHMENT 4: Sample Analysis/Extraction Log

16.5 ATTACHMENT 5: Extract Receipt Log

16.6 ATTACHMENT 6: Semivolatiles GC/MS Run Log

16.7 ATTACHMENT 7: DFTPP (Page 1 of 3)

16.8 ATTACHMENT 8: DFTPP (Page 2 of 3)

16.9 ATTACHMENT 9: DFTPP (Page 3 of 3)

16.10 ATTACHMENT 10: Split/Splitless (Multimode) Inlet

16.11 ATTACHMENT 11: Target File Window

16.12 ATTACHMENT 12: Back-up Blank Usage Form

Table 1
DFTPP Key Ions and Abundance Criteria

Mass	Ion Abundance Criteria
51	30% to 60% of mass 198
68	Less than 2% of mass 69
70	Less than 2% of mass 69
127	40% to 60% of mass 198
197	Less than 1% of mass 198
198	Base peak. 100% relative abundance
199	5% to 9% of mass 198
275	10% to 30% of mass 198
365	Greater than 1% of mass 198
441	Present but less than mass 443
442	Greater than 40% of mass 198
443	17% to 23% of mass 442

Table 2
Surrogate Spike Recovery Limits

Fraction	Surrogate Compound	Water	Low/Medium Soil
BN	Nitrobenzene-d5	35 - 114	23 - 120
BN	2-Fluorobiphenyl	43 - 116	30 - 115
BN	p-Terphenyl-d14	33 - 141	18 - 137
Acid	Phenol-d5	10 - 94	24 - 113
Acid	2-Fluorophenol	21 - 100	25 - 121
Acid	2,4,6-Tribromophenol	10 - 123	19 - 122

* Method Limits

Table 3
Matrix Spike Recovery Limits

Fraction	Matrix Spike Compound	Water	RPD	Sediment/Soil	RPD
BN	1,2,4-Trichlorobenzene	39 - 98	28	38 - 107	23
BN	Acenaphthene	46 - 118	31	31 - 137	19
BN	2,4-Dinitrotoluene	24 - 96	38	28 - 89	47
BN	Pyrene	26 - 127	31	35 - 142	36
BN	N-Nitroso-Di-n-Propylamine	41 - 116	38	41 - 126	38
BN	1,4-Dichlorobenzene	36 - 97	28	28 - 104	27
Acid	Pentachlorophenol	9 - 103	50	17 - 109	47
Acid	Phenol	12 - 89	42	26 - 90	35
Acid	2-Chlorophenol	27 - 123	40	25 - 102	50
Acid	4-Chloro-3-Methylphenol	23 - 97	42	26 - 103	33
Acid	4-Nitrophenol	10 - 80	50	11 - 114	50

* Recovery Limits from EPA/CLP are advisory.

Table 4
Characteristic Ions for Pesticide/PCBs

Parameter	Primary Ion	Secondary Ion(s)
BHC	183	181, 109
Beta-BHC	181	183, 109
Delta-BHC	183	181, 109
Gamma-BHC (Lindane)	183	181, 109
Heptachlor	100	272, 274
Aldrin	66	263, 220
Heptachlor Epoxide	353	355, 351
Endosulfan I	195	339, 341
Dieldrin	79	263, 279
4,4'-DDE	246	248, 176
Endrin	263	82, 81
Endosulfan II	337	339, 341
4,4'-DDD	235	237, 165
Methoxychlor	227	228
Chlordane (alpha and/or gamma)	373	375, 377
Toxaphene	159	231, 233
Aroclor-1016	222	260, 292
Aroclor-1221	190	222, 260
Aroclor-1232	190	222, 260
Aroclor-1242	222	256, 292
Aroclor-1248	292	362, 326
Aroclor-1254	292	362, 326
Aroclor-1260	360	362, 394
Endrin Ketone	317	67, 319

Table 5
Characteristic Ions for Semivolatile TCL Compounds

Parameter	Primary Ion	Secondary Ion(s)	Parameter	Primary Ion	Secondary Ion(s)
Phenol	94	65,66	2,4-Dinitrophenol	184	63,154
bis(2-Chloroethyl) Ether	93	63,95	4-Nitrophenol	109	139,65
2-Chlorophenol	128	64,130	Dibenzofuran	168	139
1,3-dichlorobenzene	146	148,113	2,4-Dinitrotoluene	165	63,182
1,4-Dichlorobenzene	146	148,113	2,6-Dinitrotoluene	165	89,121
Benzyl Alcohol	108	79,77	Diethylphthalate	149	177,150
1,2-Dichlorobenzene	146	148,113	4-Chlorophenyl-phenylether	204	206,141
2-Methylphenol	108	107	Fluorene	166	165,167
bis(2-chloroisopropyl) Ether	45	77,79	4-Nitroaniline	138	92,108
4-Methylphenol	108	107	4,6-Dinitro-2-Methylphenol	198	182,77
N-Nitroso-Di-Propylamine	70	42,101,130	N-Nitrosodiphenylamine	169	168,167
Hexachloroethane	117	201,199	4-Bromophenyl-phenylether	248	250,141
Nitrobenzene	77	123,65	Hexachlorobenzene	284	142,249
Isophorone	82	95,138	Pentachlorophenol	266	264,268
2-Nitrophenol	139	65,109	Phenanthrene	178	179,176
2,4-Dimethylphenol	107	121,122	Anthracene	178	179,176
Benzoic Acid	122	105,77	Di-N-Butylphthalate	149	150,104
bis(-2-Chloroethoxy) Methane	93	95,123	Fluoranthene	202	101,100
2,4-Dichlorophenol	162	164,98	Pyrene	202	101,100
1,2,4-Trichlorobenzene	180	182,145	Butylbenzylphthalate	149	167,279
Naphthalene	128	129,127	3,3-Dichlorobenzidine	252	253,125
4-Chloroaniline	127	129	Benzo (a) Anthracene	228	229,226
Hexachlorobutadiene	237	235,272	bis(2-Ethylhexyl)Pthalate	149	167,279
2,4,6-Trichlorophenol	196	198,200	Chrysene	228	226,229
2,4,5-Trichlorophenol	196	198,200	Di-N-Octyl Phthalate	149	———
2-Chloronaphthalene	162	164,127	Benzo (b) Fluoranthene	252	253,125
2-Nitroaniline	65	92,138	Benzo (k) Fluoranthene	252	253,125
Dimethyl Phthalate	63	194,164	Benzo (a) Pyrene	252	253,125
Acenaphthylene	152	151,153	Indeno (1,2,3-cd) Pyrene	276	138,227
3-Nitroaniline	138	108,92	Dibenz (a,h) Anthracene	78	139,279
Acenaphthene	153	152,154	Benzo (g,h,i) Perviene	76	138,277

Table 6
Characteristic Ions for Surrogate and Internal Standards for Semivolatile Organic Compounds

Surrogate Standards			Internal Standards		
Parameter	Primary Ion	Secondary Ion(s)	Parameter	Primary Ion	Secondary Ion(s)
Phenol-d5	99	42,71	1,4-Dichlorobenzene-d4	152	115
2-Fluorophenol	112	64	Naphthalene-d8	136	68
2,4,6-Tribromophenol	330	332,141	Acenaphthene-d10	164	162,160
d-5 Nitrobenzene	82	128,54	Phenanthrene-d10	188	94,80
2-Fluorobiphenyl	172	171	Chrysene-d12	240	120,236
Terphenyl	244	122,212	Perylene-d12	264	260,265

Table 7
Semivolatile Internal Standards with Corresponding TCL Analytes Assigned for Quantitation

1,4-Dichlorobenzene-d4	Napthalene-d8	Acenaphthene-d10
Phenol	Nitrobenzene	Hexachlorocyclopentadiene
bis (2-Chloroethyl) ether	Isophorone	2,4,6-Trichlorophenol
2-Chlorophenol	2-Nitrophenol	2,4,5-Trichlorophenol
1,3-Dichlorobenzene	2,4-Dimethylphenol	2-Chloronaphthalene
1,4-Dichlorobenzene	Benzoic acid	2-Nitroaniline
Benzyl Alcohol	bis (2-Chloroethoxy) methane	Dimethyl Phthalate
1,2-Dichlorobenzene	2,4-Dichlorophenol	Acenaphthylene
2-Methylphenol	1,2,4-Trichlorobenzene	2,4-Dinitrophenol
bis (2-Chloroisopropyl) ether	Naphthalene	4-Nitrophenol
4-Methylphenol	4-Chloroaniline	Dibenzofuran
N-nitroso-Di-n-propylamine	Hexachlorobutadiene	2,4-Dinitrotoluene
Hexachloroethane	4-Chloro-3-Methylphenol	2,6-Dinitrotoluene
2-Fluorophenol (surr)	2-Methylnaphthalene	Diethyl Phthalate
Phenol-d6 (surr)	Nitrobenzene-d5 (surr)	4-Chlorophenyl phenyl ether
		Fluorene
		4-Nitroaniline
		2-Fluorobiphenyl (surr)
		2,4,6-Tribromo Phenol (surr)
Phenanthrene-d10	**Chrysene-d12**	**Perylene-d12**
4,6-Dinitro-2-methylphenol	Pyrene	Di-n-octyl Phthalate
N-nitrosodiphenylamine	Butylbenzyl	Benzo (b) fluoranthene
1,2-Diphenylhydrazine	3,3'-Dichlorobenzidine	Benzo (k) fluoranthene
4-Bromophenyl Phenyl Ether	Benzo (a) anthracene	Benzo (a) pyrene
Hexachlorobenzene	bis(2-ethylhexyl) Phthalate	Indeno (1,2,3-cd) pyrene
Pentachlorophenol	Chrysene	Dibez (a,h) anthracene
Anthracene	Terphenyl-d14 (surr)	Benzo (g,h,i) pyrene
Di-n-butyl Phthalate		
Fluoranthene		

Table 8
Semivolatile Calibration Check Compounds (CCC)

Acenaphthene	Fluoranthene	2-Nitrophenol
1,4-Dichlorobenzene	Benzo(A)Pyrene	Phenol
Hexachlorobutadiene	4-Chloro-3-Methylphenol	Pentachlorophenol
N-Nitroso-Di-N-Phenylamine	2,4-Dichlorophenol	2,4,6-Trichlorophenol
Di-N-Octylphthalate		

Initial calibration 10, 25, 50, 80, and 100ng.

The maximum % RSD for Initial Calibration is 30% for CCC compounds.

Maximum percent difference (or drift) in the continuing calibration is 20% for CCC compounds.

Table 9
Semivolatile System Performance Check Compounds (SPCC)

N-Nitroso-Di-n-Propylamine
Hexachlorocyclopentadiene
2,4-Dinitrophenol
4-Nitrophenol

The Minimum Relative Response Factor/Initial Calibration Average: 0.05, Continuous Calibration: 0.05. All other compounds must meet a minimum response factor of 0.01.

Table 10
Target Compound List (TCL) and Contract Required Quantitation Limits (CRQL) and Practical Quantitation Limits (PQL) (continue)

Semivolatiles	CAS Number	Quantitation Limits Water Low ug/L	* Soil/ Sediment ug/Kg
1 Phenol	108-95-2	10	330
2 bis(2-Chloroethyl)ether	111-44-4	10	330
3 2-Chlorophenol	95-57-8	10	330
4 1-3-Dichlorobenzene	541-73-1	10	330
5 1,4-Dichlorobenzene	106-46-7	10	330
6 Benzyl alcohol	100-51-6	10	330
7 1,2-dichlorobenzene	95-50-1	10	330
8 2-Methylphenol	95-48-7	10	330
9 bis(2-Chloroisopropyl)ether	108-60-1	10	330
10 4-Methylphenol	106-44-5	10	330
11 N-Nitroso-di-n-dipropylamine	621-64-67	10	330
12 Hexachloroethane	67-72-1	10	330
13 Nitrobenzene	98-95-3	10	330
14 Isophorone	78-59-1	10	330
15 2-Nitrophenol	88-75-5	10	330
16 2,4-Dimethylphenol	105-67-9	10	330
17 Benzoic acid	65-85-0	50	1600
18 bis(2-Chloroethoxy)methane	111-91-1	10	330
19 2,4-Dichlorophenol	120-83-2	10	330
20 1,2,4-Trichlorobenzene	120-82-1	10	330
21 Naphthalene	91-20-3	10	330

Medium soil/sediment Contract Required Detection Limits (CRQL) for semivolatile TCL compounds are 60 times the individual low soil/sediment CRQL.

Table 10 (continued)
Target Compound List (TCL) and Contract Required Quantitation Limits (CRQL) and Practical Quantitation Limits (PQL)

Semivolatiles	CAS Number	Quantitation Limits Water Low ug/L	* Soil/ Sediment ug/Kg
22 4-Chloroaniline	106-47-8	10	330
23 Hexachlorobutadaiene	77-47-4	10	330
24 4-Chloro-3-methylphenol (para-chloro-meta-cresol)	59-50-7	10	330
25 2-Methylnaphthalene	91-57-6	10	330
26 Hexachlorocyclopentadiene	77-47-4	10	330
27 2,4,6-Trichlorophenol	88-06-2	10	330
28 2,4,5-Trichlorophenol	95-95-4	50	1600
29 2-Chloronaphthalene	91-58-7	10	330
30 2-Nitroaniline	88-74-4	50	1600
31 Dimethylphthalate	131-11-3	10	330
32 Acenaphthylene	208-96-8	10	330
33 2,6-Dinitrotoluene	606-20-2	10	330
34 3-Nitroaniline	99-09-2	50	1600
35 Acenaphthene	83-32-9	10	330
36 2,4-Dinitrophenol	51-28-5	50	1600
37 4-Nitrophenol	100-02-7	50	1600
38 Dibenzofuran	132-64-9	10	330
39 2,4-dinitrotoluene	121-14-2	10	330
40 1,2-Diethylphthalate	84-66-2	10	330
41 4-Chlorophenyl-phenyl ether	7005-72-3	10	330
42 Fluorene	86-73-7	10	330
43 4-Nitroaniline	100-01-6	50	1600
44 4,6-Dinitro-2-methylphenol	534-52-1	50	1600
45 N-nitrosodiphenylamine	86-30-6	10	330
46 4-Bromophenyl-phenyl ether	101-55-3	10	330
47 Hexachlorobenzene	118-74-1	10	330
48 Pentachlorophenol	87-86-5	50	1600
49 Phenanthrene	85-01-8	10	330
50 Anthracene	120-12-7	10	330
51 Di-n-butylphthalate	84-74-2	10	330
52 Fluoranthene	206-44-0	10	330
53 Pyrene	129-00-0	10	330
54 Butylbenzylphthalate	85-68-7	10	330
55 3,3'-Dichlorobenzidine	91-94-1	20	660
56 Benzo(a)anthracene	56-55-3	10	330
57 Chrysene	218-01-9	10	330
58 bis(2-Ethylhexyl)phthalate	117-81-7	10	330
59 Di-n-octylphthalate	117-84-0	10	330
60 Benzo(b)fluoranthene	205-99-2	10	330
61 Benzo(k)fluoranthene	207-08-9	10	330
62 Benzo(a) pyrene	50-32-8	10	330
63 Indeno(1,2,3-cd)pyrene	193-39-5	10	330
64 Dibenz(a,h)anthracene	53-70-3	10	330
65 Benzo(g,h,i)perylene	191-24-2	10	330

* Medium Soil/Sediment Contract Required Quantitation Limits (CRQL) for semivolatile TCL Compounds are 60 times the individual Low/Soil/Sediment CRQL.

~ Specific quantitation limits are highly matrix dependent. The quantitation limits listed herein are provided for guidance and may not always be achievable.

~ Quantitation limits listed for soil/sediment are based on wet weight. The quantitation limits calculated by the laboratory for soil/sediment, calculated on dry weight basis as required by the contract, will be higher.

* Reporting Limits are from Method.

Table 11
Standards Preparation

Compound	Conc. (µg/mL)	Aliquot (mL)	Total Vol. (mL)	Conc. (µg/mL)	Volume used/µL
Absolute Internal Std. Mix	4000	10	40	1000	—
STOCK STANDARD (Restek)			12		
Methylene Chloride		880			
SV Calibration Mix #1	2000	1200		200	—
SV Calibration Mix #2	2000	1200		200	—
SV Calibration Mix #3	2000	1200		200	—
SV Calibration Mix #4	2000	1200		200	—
SV Calibration Mix #5	2000	1200		200	—
SV Calibration Mix #7	2000	1200		200	—
Benzidines Mn (nsi)	2000	1200		200	—
Benzoic Acid (nsi)	5000	480		200	—
Acid Surr. Mix	7500	320		200	—
B-N Surr. Mix	5000	480		200	—
Pyridine	10000	240		200	—
Aniline	10000	240		200	—
1,2,4,5-Tetrachlorobenzene	10000	240		200	—
2,3,4,6-Tetrachlorophenol	10000	240		200	—
1-methylnaphthalene	10000	240		200	—
Azo Benzene	10000	240		200	—
Stock Std.	200	2000		50	—
IS mix (SSTD050)	1000	320	8	40	—
Stock Std.	200	1200		80	—
IS mix (SSTD080)	1000	120	3	40	—
Stock Std.	200	375		25	—
IS mix (SSTD025)	1000	120	3	40	—
Stock Std.	200	1500		100	—
IS mix (SSTD100)	1000	120	3	40	—
Stock Std.	200	150		10	—
IS mix (SSTD010)	1000	120	3	40	—

These standards are verified with EPA standards regularly. Every time a standard is made, it is recorded in the current standards logbook, and follows protocol for expirations as provided in SOP SWL-OS-309, "Guide To Semivolatile GC/MS Standard Log".

Table 12
Individual Compound Stock Standards Prep

Compound	Concentration	Aliquot	Total Volume	Final Concentration
Pyridine	NEAT	0.1g	10mL	10,000ug/mL
Aniline	NEAT	0.1g	10mL	10,000ug/mL
1,2,4,5-Tetrachlorobenzene	NEAT	0.1g	10mL	10,000ug/mL
2,3,4,6-Tetrachlorophenol	NEAT	0.1g	10mL	10,000ug/mL
1-Methylnaphthalene	NEAT	0.1g	10mL	10,000ug/mL
Azobenzene	NEAT	0.1g	10mL	10,000ug/mL

Table 13
DFTPP Solution Prep

Compound	Concentration ug/mL	Aliquot mL	Total Volume mL	Concentration ug/mL
DFTPP Solution (Protocol)	2500	0.20	10	50
Phenanthrene-d10	2000	0.25		50
Methylene Chloride		9.55		

* DFTPP Solution from Protocol contains 4,4-DDT, Benzidine and Pentachlorophenol along with DFTPP, each at 2500ug/mL.

Table 14
Calculations

Response Factor and Concentration Calculation

$$RF = \frac{(A_x)\ (C_{is})}{(A_{is})\ (C_x)} \quad \text{and} \quad conc_{RF} = \frac{(A_x)\ (C_{is})}{(A_{is})\ (\overline{RF})} \quad \text{or} \quad conc_{LR} = \frac{(A_x) - b}{M} \quad \text{or} \quad conc_{quad} = a + bx + cx^2$$

Where:

A_x = Area of characteristic ion for the measured compound.
A_{is} = Area of the characteristic ion for the specified internal standard
C_{is} = Concentration of the internal standard (ng/µL)
C_{RF} = Concentration of the measured compound (ng/µL) using the average response factor
$\overline{RF}$ = Average of Five Initial RF's for a compound
C_{LR} = Concentration of the measured compound (ng/µL) using Linear Regression
C_{quad} = Concentration of the measured compound (ng/µL) using the Quadratic Equation

---

Surrogate Recovery: % Surrogate Recovery = $\frac{Qd}{Qa} \times 100\%$

Where:

Qd = Quantity determined by analysis
Qa = Quantity added to sample

Matrix Spike recovery and RPD:

Matrix Spike Recovery = $\frac{SSR - SR}{SA} \times 100$ Matrix Spike RPD = $\frac{|MSR - MSDR|}{(½)(MSR + MSDR)} \times 100$

Where:

SSR = Spike Sample Result
SR = Sample Result
SA = Spike Added

Laboratory Control Spike (LCS) Recovery: LCS Spike Recovery = $\frac{SSR - SR}{SA} \times 100$

Where:

SSR = Spiked Sample Result
SR = Sample Result
SA = Spike Added

Calibration Calculations:

$$\%RSD = \frac{SD}{\overline{RF}} \times 100\%$$

Where:

SD = Standard Deviation
$\overline{RF}$ = Average of Five Initial RF's for a compound
RSD = Relative Standard Deviation

$$\%Drift = \frac{(C_1 - C_c)}{C_1} \times 100$$

Where:

C_1 = Calibration Check Compound Standard Concentration
C_c = Measured concentration using selected quantitation method

Form I

RE-EXTRACT TRACKING SHEET

	CLIENT	EPISODE/ LAB ID #	H20	SOIL	INST.	MS CODE	DATE RCV'D	INITIAL EXT. DATE	RE-EXT. SUB. DATE	REASON FOR RE-EXTRACT			RE-EXT. DATE	RE-EXTRACT RESULTS			W/HOLD TIME Y/N
										ACID	B/N	QC		ACID	B/N	QC	
1																	
2																	
3																	
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Attachment 1
Re-extract Tracking Sheet

DATA REVIEW SHEET
SWOK/AATS

ANALYST SECTION

EPISODE_____ INSTRUMENT____
MS CODE_____ ANALYST_____

_____ All usable analysis with in 12 hour clock

_____ Correct method updated with passing continuing calibration or initial calibration

_____ Correct sublist used for requested compound list

_____ Misc. info line correct (MSCODE*TCLP BLK*INST ID*CLIENT ID*SWLO #*DILUTION RUN STRING*)

_____ Sample info, client info, concentration parameters entered correctly

_____ SBLK pass QC criteria (no contamination greater than CRQL, all surrogates and internal standards pass)

_____ Internal standard areas and retention times checked (Internal standard areas compared to correct SSTD050)

_____ Surrogate recoveries checked (One acid and one base/neutral surrogate allowed out, however, no surrogates can have a responce of less than 10%)

_____ Compound linearity checked (ng on column amount must be less than the highest concentration in the initial calibration)

_____ All isomeric compounds checked for separation

_____ Target compounds checked for proper integration

_____ TICs included for applicable samples (integration parameters ser correctly in method)

_____ All applicable target reports included (i.e., clp.rp, spike.rp, istd.rp, total ion chromatogram, target.rp, spectra, etc.)

_____ SWLO LCS_RPT or MS_RPT generated for control spikes and matrix spikes (std tab report only)

_____ QC ref #, batch #, initial cal #, checked on sample.x.map sheet

_____ If any re-extracts were performed, are remedial action forms attached? Yes____No____

Analyst comments_____

DATA REVIEW SECTION

_____ False positives deleted from system _____ Quant reports replaced _____ TICs edited

_____ Holding times met for extraction and analysis? Yes _____ No _____

_____ Any dilutions or reanalysis checked against original sample

_____ Do the MS/MSD agree with each other and the original sample? Yes _____ No _____

_____ Do the LCS/LCSD pass? Yes _____ No _____

Initial Data Review by _____ Date____/_____ 1997 Final Review/Verification by_____Date ____/____ 1997

Data downloaded to DDB by _____ Date ____/____ 1997

Reviewer Comments _____

Attachment 2
Data Review Sheet

NON-CONFORMANCE REPORT QA DEPT.

SWL-GA-105 GA-105-NCR-F SWL Rev. No.:_____

Department:

8.2.1.2 DESCRIPTION OF NON-CONFORMANCE

Initiated By: Date:

Section Supervisor: Date:

Date of Occurrence:

Description of Event:

System and/or Episode of Samples Affected (Client/Test code/Matrix if applicable):

8.2.1.3 CAUSE

Description:

SIGNATURE:

Section Supervisor: Date:

8.2.1.3 ACTION TO BE TAKEN

Re-preparation/ Re-extraction Required (Yes/ No)? Date Needed:

Within hold time: (Yes/No)? Sample Receipt Date (if applicable):

Description (with dates):

SIGNATURE:

Completion Date: Section Supervisor:

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

Attachment 3
Non-Conformance Report

SAMPLE EXTRACTION LOG

SWOK • AATS

Southwest Laboratory of Oklahoma, Inc.
American Analytical & Technical Services, Inc.

☐ RUSH ☐ REEXTRACT ☐ TCLP ☐ DIOXIN

CLIENT: _____ DATE RECEIVED: _____

CASE: _____ EXTRACT DATE: _____

METHOD	GV	PEST • PCB	OTHER
CONT. EX.			
SEP-F			
SONC.			

SAMPLE ID (EPISODE #)	CLIENT ID	MATRIX • LEVEL	SAMPLE pH	% MOISTURE	DRY WEIGHT	AMOUNT EXTRACTED	EXTRACTED VOLUME	GPC VOLUME	GPC ELUTE VOL	FLORISIL VOL CONC	FLORISIL FIN VOL CONC	VOL SV • P	FIN PROCESS VOLUME	SCREEN Y/N	MS SV • P	SUPR SV • P	BASE pH CONFIRMATION	ACID pH CONFIRMATION	COMMENTS
				%	g	g • mL	mL	mL	mL	mL	mL	mL	mL		µL	µL			

INITIALS	
WEIGHING:	EXTRACTION:
CLEANUP:	KD:
BLOWDOWN:	
SURROG. SPIKER:	VERIFIER:
MATRIX SPIKER:	VERIFIER:
SUBMITTED BY:	REVIEWED BY:

ID NUMBERS
FLORISIL CARTRIDGE #:
GPC COLUMN #:
SURROGATES ID CONC #:
MATRIX SPIKE SOL ID/CONC #(S):
MISC:

COMMENTS

[D0013-0393-01]

54201

Attachment 4
Sample Analysis/Extraction Log

Typist LLR

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EXTRACT RECEIPT LOG

SEMIVOLATILE GC/MS LABORATORY

DATE	TIME	SWOK / AATS SAMPLE ID	CLIENT ID	EXTRACTION CHEMIST INITIALS	GC/MS SAMPLE CUST. INITIALS	COMMENTS	NO. OF SAMPLES

SOUTHWEST LABORATORY OF OKLAHOMA, INC. • AMERICAN ANALYTICAL & TECHNICAL SERVICES, INC.
1700 W. ALBANY • BROKEN ARROW, OKLAHOMA 74012 • OFFICE (918) 251-2858 • FAX (918) 251-2599

[SGM005-0193-02]

Attachment 5
Extract Receipt Log

SEMIVOLATILES GC/MS RUN LOG

SWOK/AATS 1700 W. ALBANY BROKEN ARROW, OK 74012

SEMIVOLATILES GC/MS DEPT

Ref: SWL-OS-102 Rev. 2.1

Book ID # OS-102-RUN-O2

ANALYTICAL SOP REFERENCE: SWL-OS-_____REV.____

INT. STD. #:_____ ANALYSIS METHODS:_____ DATE:_____

EPISODE:_____ TARGET METHODS: _____ INST:_____EMV:_____

MSCODE: TARGET BATCH: /chem/ ___ .I/ TUNE FILE ID:

TIME	INIT	CLIENT SAMPLE ID	LAB ID	FILE ID	SAMPLE AMOUNT			DIL FACT	IS OK	SURR OK	LIN-EAR OK	ARCHIVE TAPE	COMMENTS
					WT	VOL	INJ						

F:\common\organics\bnarun, F=mickey\sys

Analyst Signature _____

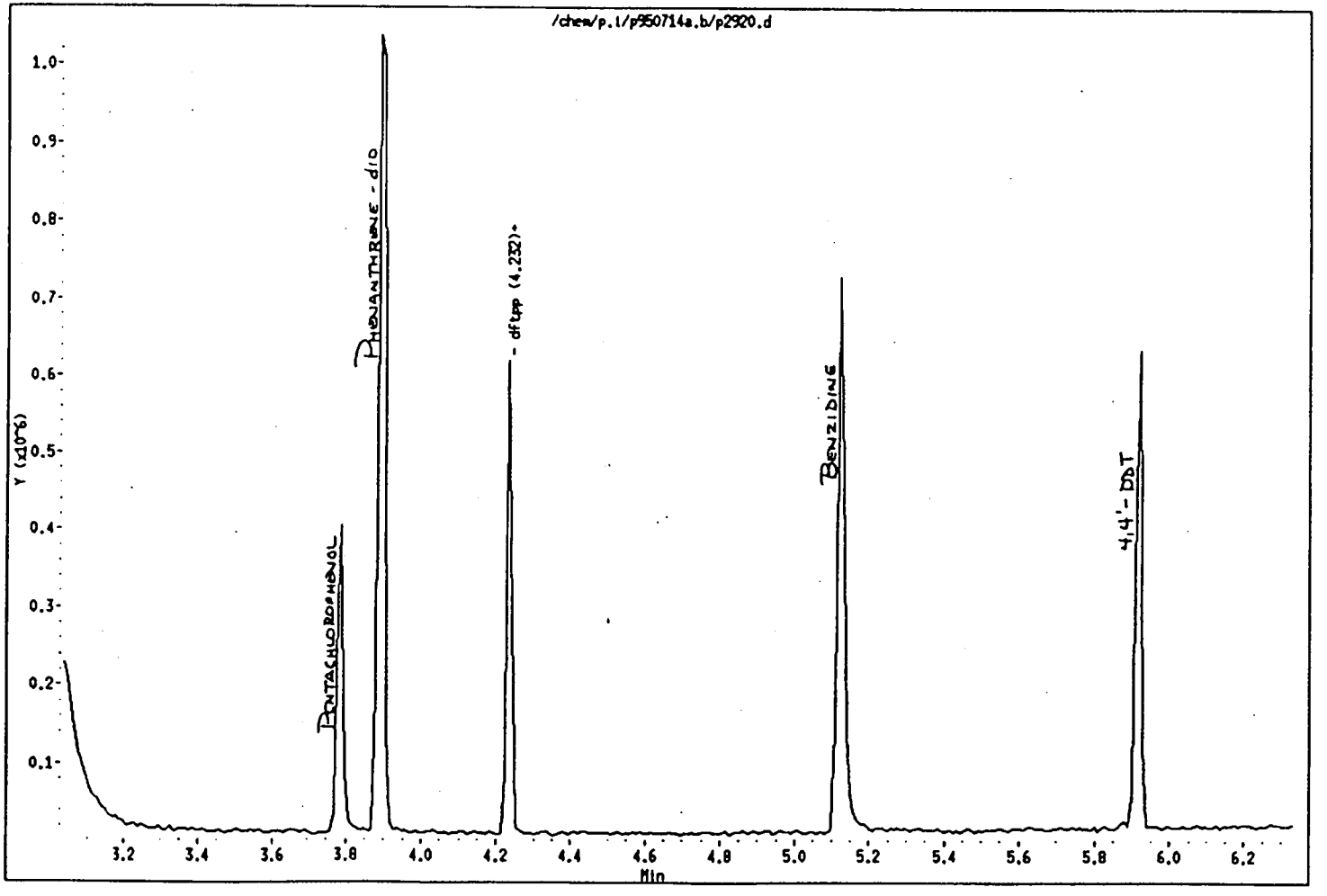
Reviewd by:_____

Attachment 6
Semivolatiles GC/MS Run Log

Data File: /chem/p.1/p950714a.b/p2920.d
Date : 14-JUL-95 14:25
Instrument : p.1
Sample ID : DFTPP
Column phase : XTI-5
Volume Injected (uL) : 2.0

Page 1

Column diameter : 0.25



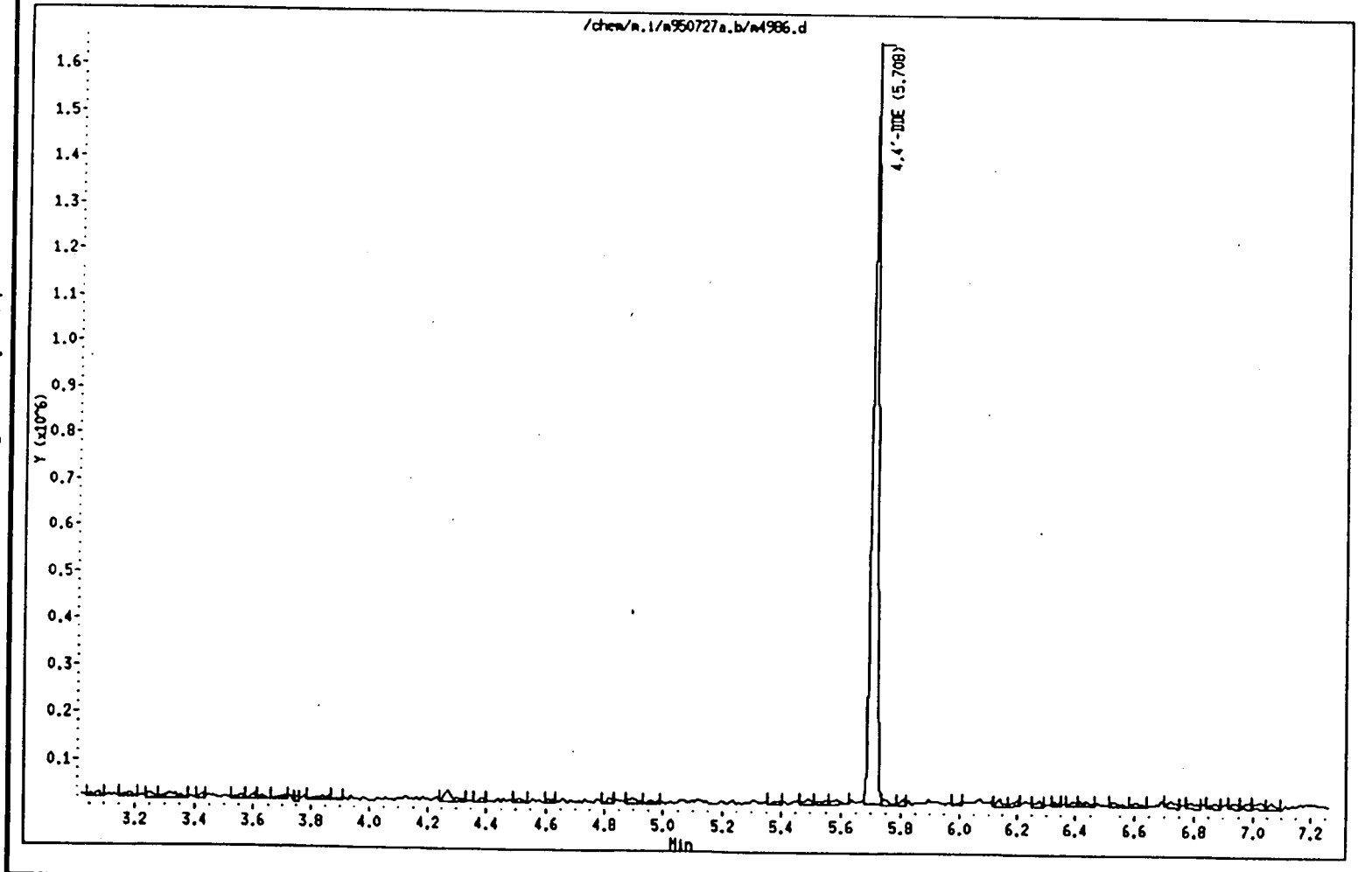
Attachment 7
DFTPP (Page 1 of 3)

CR-237

Data File: /chem/m.1/m950727a.b/m4986.d
Date : 27-JUL-1995 16:37
Instrument : m.1
Sample ID : 4,4'-DDE
Column phase : XTI-5
Volume Injected (uL) : 2.0

Page 1

Column diameter : 0.25



Attachment 8
DFTPP (Page 2 of 3)

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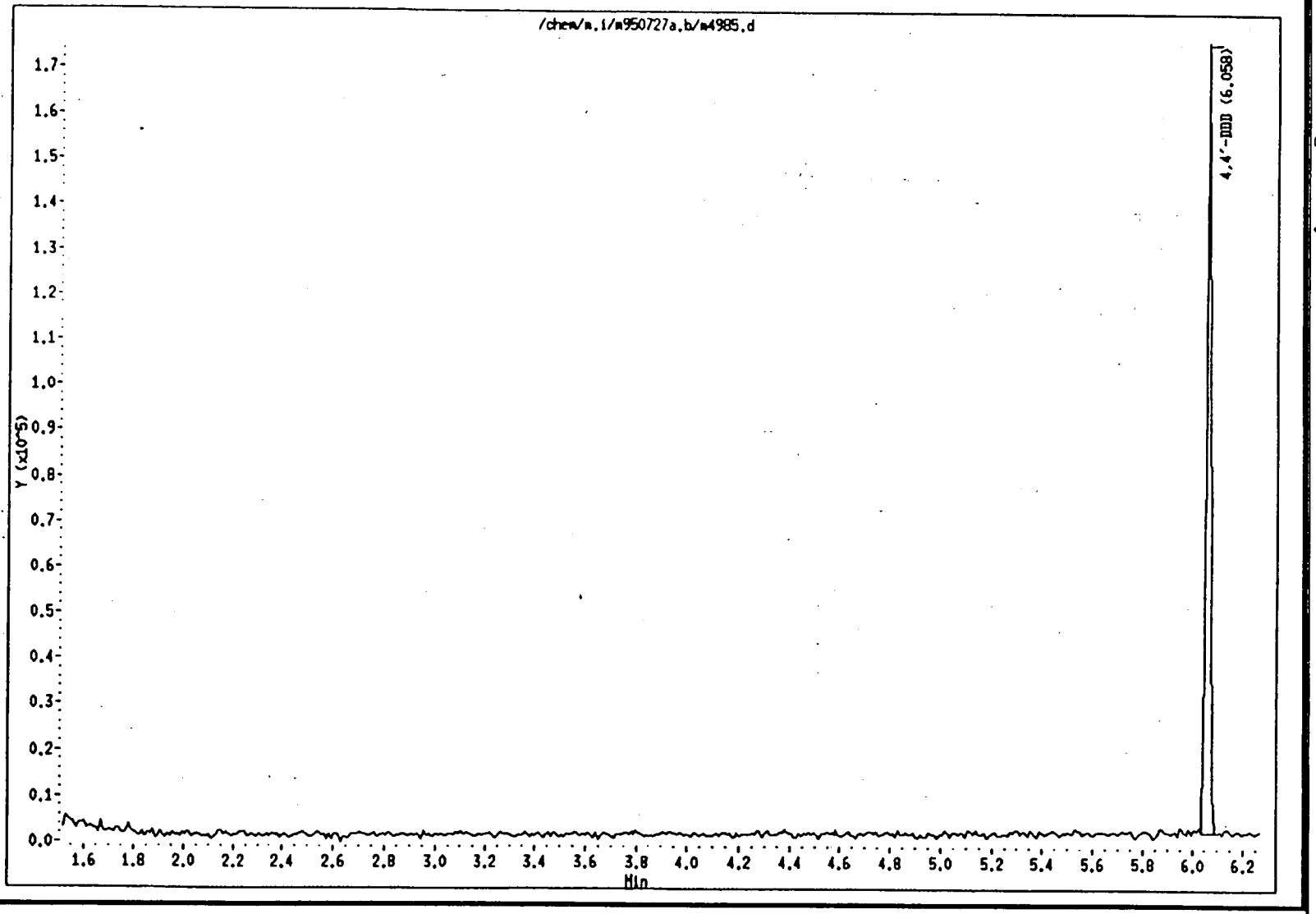
Data File: /chem/m.i/m950727a.b/m4985.d
Date : 27-JUL-1995 16:27
Instrument : m.i
Sample ID : 4,4'-DDD
Column phase : XTI-5
Volume Injected (uL) : 2.0

Page 1

Column diameter : 0.25



Attachment 9
DFTPP (Page 3 of 3)

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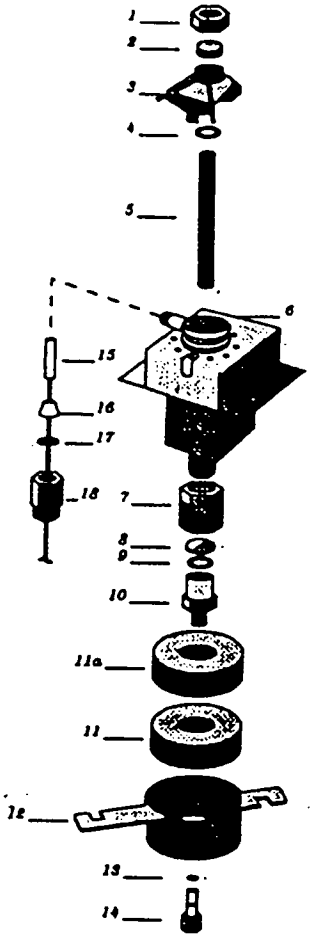
1	Septum nut	
	Slot (strainer nut for headspace)	
	or Merlin Microseal (see page 51)	
2	Septa (see page 50)	
3	Split/splitless insert weldment with tubing	19251-60577
4	Liner seals: (also refer to liner table on page 42)	
	a. Viton O-ring for high temperature (max. 350 °C) (12/pk)	5180-4182
	b. Graphite O-rings (temperature above 350 °C)	
	For split liner (12/pk)	5180-4184
	For splitless liner (12/pk)	5180-4173
5	See liner table on page 42	
6	Shell weldment (refer to instrument manuals)	
7	Retaining nut	19251-20620
8	Gold-plated seal	19740-20885
	or stainless steel seal	19740-20880
9	Washer (12/pk)	5061-5869
10	Reducing nut (1 ea)	19740-20800
11	Lower insulation	19243-00060
11a	Insulation	19243-00065
12	Lower insulation cover	19243-00070
13	Column seals/ferrules—page 44	
14	Column nut (2/pk)	5181-8830
15	Splitter tube	19251-60525
16	Front ferrule, ¼-in. brass (10/pk)	5180-4109
17	Back ferrule, ¼-in. brass (10/pk)	5180-4115
18	Tubing nut, ¼-in. brass (10/pk)	5180-4103



Split/Splitless (Multimode) Inlet

Attachment 10
Split/Splitless (Multimode) Inlet

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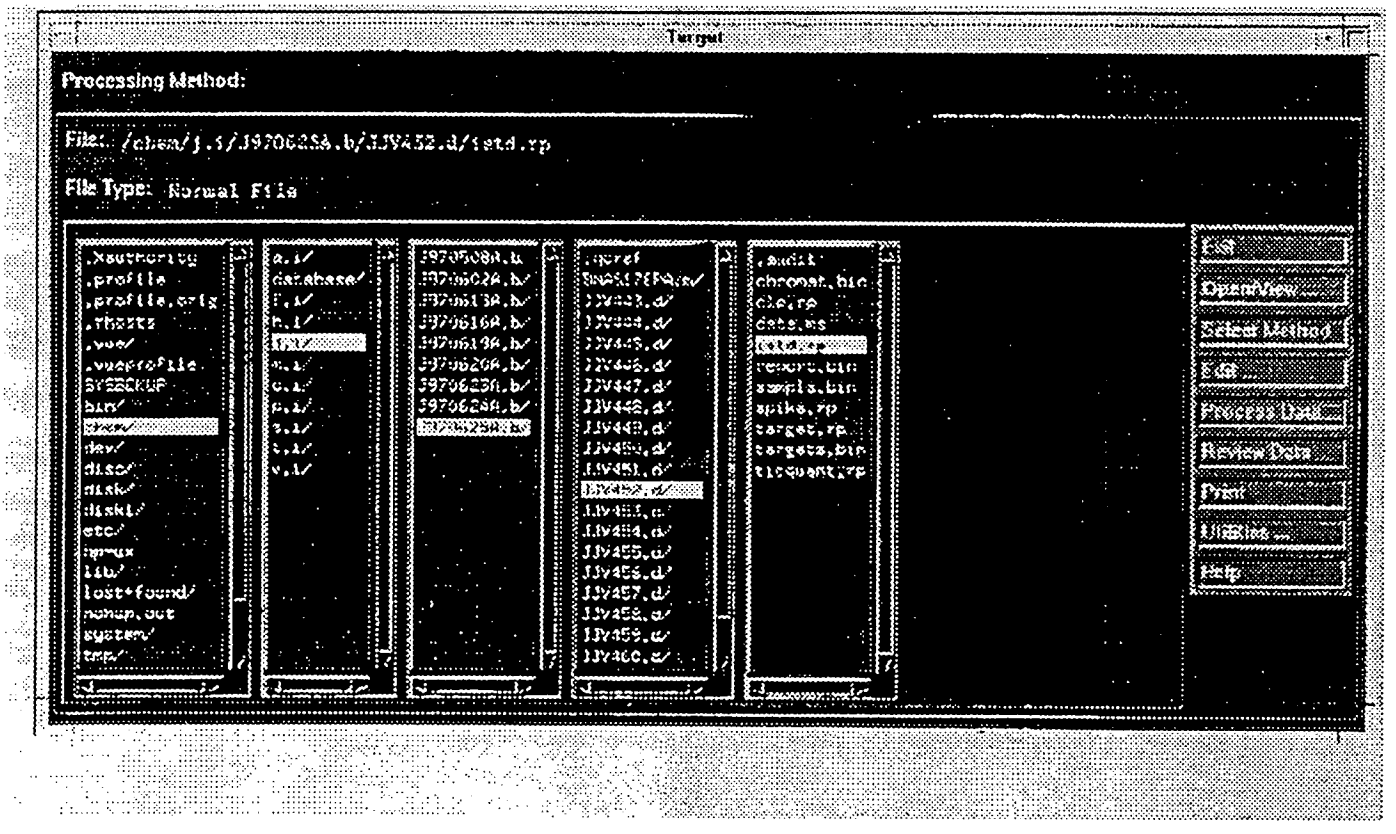

Attachment 11
Target File Window

FORM 1

BACK-UP BLANK USAGE FORM

When a back-up blank is used this form must be completed. The signed original must be retained with the original data and a copy given to the department supervisor.

Primary Blank ID: BL_____ Date Analyzed:_____

Primary Blank Data file ID:_____ Analyst:_____

Supervisor:_____

Back-up Blank ID: BB_____ Date Analyzed:_____

Back-up Blank Data file ID:_____ Pass or Fail (circle one)

Matrix: Water Soil Other_____ Analyses Code: MS_____

GC_____

Reasons for Back-up Blank Usage: (Check all that apply)

____Surrogates Failed: ____ Acid Surrogates ____ Base/Neutral Surrogates

____Internal Standards Failed: ____Out High ____Out Low

____Contamination (Target Analyte):____Above reporting limit ____Trace Amounts

____Extract Lost: ____Vial Broken ____Extract Spilled ____Other (explain)

_____

_____

Samples Affected:_____

_____

_____

If both blanks fail, note Remedial or Corrective (circle one) Action Taken:_____

_____

Attachment 12
Back-up Blank Usage Form

Typist LLR

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SOUTHWEST LABORATORY OF OKLAHOMA, INC.

Standard Operating Procedure

Analysis of Pesticides/PCBs by GC, SW-846, Method 8081

Document No.: SWL-OP-412

Rev No. / Date: Rev.1.0 — 09/17/97

OFFICIAL COPY RED STAMP INDICATES ORIGINAL

APPROVALS	DATES
Procedure Prepared By	Date 9/17/97
Organic Program Manager	Date 9-17-97
Laboratory Safety Officer	Date 9/17/97
Laboratory QA/QC Officer	Date 9/17/97

(Effective Date is 5 calendar days after the last signature above - QA/QC Officer)

Document Status

[ ] Controlled DC No. _____ Issued to: _____ Date: _____

[✓] Official Copy OC No.: 2028 Issued to: Alan Fosdick Date: 2/4/98 mm

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1.0 Scope and Application

1.1 This method is designed to determine the concentrations of various organochlorine Pesticides and polychlorinated biphenyls (PCBs) as Aroclors, in extracts from solid and liquid matrices. The method is based on EPA SW-846 methods 8000A and 8081 for analysis and methods 3510, 3520, 3550, 3610, 3620, 3640, and 3660 for the extractions and cleanups. The method can be used to determine analyte concentrations of pesticides in the ppt range and PCBs in the ppb range in water. Test codes encompassed by this method include but are not limited to GC801, GC601, GC899, and GC699.

1.2 A large number of compounds will give response in the electron capture detector (ECD) using this method. The following list of pesticides and PCBs are analyzed using this method:

ANALYTE:.	CAS #
Aldrin	309-00-2
Alpha-BHC	319-84-6
Beta-BHC	319-85-7
Delta-BHC	319-86-8
Gamma-BHC	58-89-9
alpha-Chlordane	5103-71-9
gamma-Chlordane	5103-74-2
4,4'-DDD	72-54-8
4,4'-DDE	72-55-9
4,4'-DDT	50-29-3
Dieldrin	60-57-1
Endosulfan I	959-98-8
Endosulfan II	33212-65-9
Endosulfan Sulfate	1031-07-8
Endrin	72-20-8
Endrin Aldehyde	7421-93-4
Endrin Ketone	53494-70-5
Heptachlor	76-44-8
Heptachlor Epoxide	1024-57-3
4,4'-Methoxychlor	72-43-5
Toxaphene	8001-35-2
Aroclor-1016	12674-11-2
Aroclor -1221	104-28-2
Aroclor -1232	11141-16-5
Aroclor -1242	53469-21-9
Aroclor -1248	12672-29-6
Aroclor -1254	11097-69-1

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Aroclor -1260 11096-82-5

1.3 This method utilizes wide bore capillary (0.53 mm ID) or narrow bore capillary (0.32 mm ID) columns for both the quantitation and confirmation analyses.
1.4 Table 1 lists the Quantitation Limits (QL) and approximate retention times.

2.0 Method Summary

2.1 Continuous liquid-liquid or separatory funnel extraction procedures are employed for the extraction of aqueous samples. Sonication extraction is required for soil/sediment samples. Gel Permeation (GPC) and florisil or alumina columns may be used for cleanup of extracts.
2.2 Two columns of different stationary phases are used for quantitation and confirmation of the analytes.
2.3 Columns are calibrated using the external calibration procedure with 5 concentrations of the analytes covering the working linear range of the ECD detectors beginning from the level of quantitation or below. Calibration criteria specified in method 8000A is followed (unless otherwise specified).
2.4 Calibration verification standards are analyzed each 12 hour shift (not to exceed 20 samples) and before the start of an analysis sequence so that they bracket the samples.
2.5 An evaluation standard containing both Endrin and 4,4'-DDT is analyzed at the beginning of a calibration sequence and at the beginning of each distinct analysis sequence not to exceed a 12-hour shift. This evaluation standard monitors the breakdown of these components.
2.6 A method blank, laboratory control spike, and a matrix spike/matrix spike duplicate must be prepared for all batches of 20 samples or less.
2.7 All analytes tentatively identified within established retention windows on the quantitation column must be analyzed on the confirmation column (quantitation and confirmation are run simultaneously as a routine). The lower analytical result is reported if the analyte is identified on both columns and both columns can be used for quantitation. At least one column must meet quantitation criteria for the entire sequence. Samples must be bracketed by acceptable calibration verification standards on at least one column.

3.0 Health and Safety

3.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method (See the Standard Operating Procedure for Safety). A reference file of Material Safety Data Sheets (MSDS) is available to all personnel involved in the chemical analysis.
3.2 The following parameters covered by this method have been tentatively classified as known or suspected, human or mammalian carcinogens: 4,4'-DDT, 4.4'-DDD, the BHCs, and the PCBs. Primary standards of these toxic compounds must be prepared in a hood. A NIOSH/MESA

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approved toxic gas respirator must be worn when the analyst handles high concentrations of these toxic compounds.

3.3 Caution should be taken when working with all sample extracts and calibration standards. Samples are of unknown origin and should be considered hazardous. Many of the spiking solutions used, along with chlorinated solvents are considered carcinogens. Concentrated acids and bases, if not handled properly, can cause severe burns.

3.4 Gloves should be used for the manipulation of all sample extracts and calibration standards and preparation of all acid and base solutions. Wear personal protective equipment (PPE) and goggles or face shield when warranted. See the "Standard Operating Procedure for Safety" for detailed safety procedures.

4.0 Sample Preservation, Containers, Handling, and Storage

4.1 Sample Extraction

4.1.1 Aqueous samples must be extracted within 7 days of collection. Solid samples must be extracted within 14 days of collection.

4.1.2 Extracts must be stored under refrigeration at 4°C (±2°C) and analyzed within 40 days of extraction.

4.2 Sample Storage

4.2.1 The samples must be protected from light and refrigerated at 4°C (±2°C) from the time of receipt until 60 days after delivery of a complete reconciled sample data package to the client. After 60 days the samples may be disposed of in a manner that complies with all applicable regulations.

4.2.2 The sample must be stored in an atmosphere demonstrated to be free of all potential contaminants.

4.2.3 Samples, sample extracts, and standards must be stored separately.

4.3 Sample Extract Storage

4.3.1 Sample extracts must be protected from light and stored at 4°C (±2°C) until 365 days after delivery of a complete data package to the client.

4.3.2 Sample extracts must be stored in an atmosphere demonstrated to be free of all potential contaminants.

4.3.3 Samples, sample extracts, and standards must be stored separately.

4.4 All containers should be glass jars or bottles with Teflon lids. A minimum of one liter is required for aqueous samples. A minimum of 30 g is required for soil samples (125 mL glass jar).

5.0 Interferences and Potential Problems

5.1 Sources of interference in this method can be grouped into three broad categories: contaminated solvents, reagents or sample processing hardware; contaminated GC carrier gas, parts,

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column surfaces or detector surfaces; and the presence of coeluting compounds in the sample matrix to which the ECD will respond. Knowledge of good laboratory practices is assumed, including steps to be followed in routine testing and cleanup of solvents, reagents and sample processing hardware, and instrument maintenance. The discussion that follows focuses on sources of interference associated with the sample matrix and compound classes that represent common sources of interference, particularly phthalate esters, organosulfur compounds, lipids, and waxes. Interferences co-extracted from the samples will vary considerably from sample to sample. While general cleanup techniques are referenced or provided as part of this method, unique samples may require additional cleanup approaches to achieve desired degrees of discrimination and quantitation.

5.2 Interferences by phthalate esters introduced during sample preparation can pose a major problem in pesticide determinations. These materials may be removed prior to analysis using Gel Permeation Cleanup - pesticide option (Method 3640) or as Fraction III of the silica gel cleanup procedure (Method 3630). Common flexible plastics contain varying amounts of phthalate esters which are easily extracted or leached from such materials during laboratory operations. Cross-contamination of clean glassware routinely occurs when plastics are handled during extraction steps, especially when solvent-wetted surfaces are handled. Interferences from phthalate esters can best be minimized by avoiding contact with any plastic materials and checking all solvents and reagents for phthalate contamination. Exhaustive cleanup of solvents, reagents and glassware may be required to eliminate background phthalate ester contamination.

5.3 The presence of elemental sulfur will result in large peaks that interfere with the detection of later eluting organochlorine pesticides. Method 3660 is suggested for removal of sulfur.

5.4 Waxes, lipids, and other high molecular weight co-extractables can be removed by Gel-Permeation Cleanup (Method 3640).

5.5 Depending on column phase and instrument parameters, some pesticides may coelute. Care must be taken to ensure enough separation of analytes to allow for meaningful quantitation. The single component pesticides must be separated into two mixtures to reduce the chance of misidentification of co-eluting and close-eluting analytes.

6.0 Equipment/Apparatus

6.1 Gas chromatograph — Analytical system complete with gas chromatograph suitable for on-column, direct, and split-splitless injection and all required accessories including syringes, analytical columns, gases, electron capture detector, and recorder/integrator or data system.

6.1.1 HP 5890 Gas Chromatograph with ECD detector(s) and a 7673 autosampler. The autosampler must be capable of 1 µL or 2 µL injections. The carrier gas should be ultra pure helium or hydrogen, and the makeup gas (detector gas) should be ultra pure nitrogen or 5% methane/95% argon.

6.1.2 Fisons 8130 Gas Chromatograph with ECD detector(s) and a CTC A200S autosampler. The autosampler must be capable of 1 µL or 2 µL injection. The carrier gas should be ultra pure helium or hydrogen and the makeup gas (detector gas) should be 5% methane/95% argon, or ultra pure nitrogen.

6.1.3 Wide-bore columns:

6.1.3.1 Column 1 — 30 m x 0.53 mm ID fused silica capillary column chemically bonded with 35 to 50 percent phenyl methylpolysiloxane (DB-608, SPB-608, RTx-35, or equivalent), 0.5 um or 1.0 µm film thickness.

6.1.3.2 Column 2 — 30 m x 0.53 mm ID fused silica capillary column chemically bonded with 14 percent cyanopropylphenyl methylpolysiloxane (DB-1701, or equivalent), 1.0 µm film thickness.

6.1.3.3 Column 3 — 30 m x 0.53 mm ID fused silica capillary column chemically bonded with 5 percent phenyl methylpolysiloxane (DB-5, SPB-5, RTx-5, or equivalent), 1.5 µm film thickness.

6.1.3.4 Wide-bore columns should be installed with deactivated liners designed specifically for use with these columns.

6.1.4 Narrow-bore columns:

6.1.4.1 Column 4 — 30 m x 0.32 mm ID fused silica capillary column (RTX-50, DB-17, or equivalent), 0.25, 0.5, or 1.0 µm film thickness.

6.1.4.2 Column 5 — 30m x 0.32 mm ID fused silica capillary column (RTX-1701, DB1701, or equivalent), 0.25, 0.5, or 1.0 µm film thickness.

6.1.4.3 Column 6 — Dual capillary columns. Dual DB-17/1701 combination produced by J&W, 0.25 m film thickness.

6.1.4.4 All columns should be equipped with megabore (0.53 mm) deactivated guard columns.

6.1.5 GC injection ports are of critical concern, especially in the analysis of DDT and Endrin. Injectors that are contaminated, chemically active, or too hot can cause the degradation ("breakdown") of the analytes. Endrin and DDT breakdown to endrin aldehyde, endrin ketone, DDD, or DDE. When such breakdown is observed, replace inlet line, and if necessary clip one to three feet off guard column and remount it. A performance evaluation standard is analyzed at the beginning of each 12-hour period. See Table 5 for preparation.

7.0 Reagents

7.1 Reagent or pesticide grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 Organic-free reagent water: All references to water in this method refer to organic-free reagent water.

7.3 Solvents and reagents: As appropriate for Method 3510, 3520, 3540, 3541, 3550, 3630, 3640, or 3660: n-hexane, diethyl ether, methylene chloride, acetone, ethyl acetate, and isooctane (2,2,4-trimethylpentane). All solvents should be pesticide quality or equivalent, and each lot of solvent should be determined to be phthalate free.

7.4 All gases (i.e., carrier, auxiliary, etc.) should be at least high purity grade.

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8.0 Procedure

8.1 Stock Standard Solutions

8.1.1 Concentrated and certified standard mixtures can be obtained from several sources. For example, Restek and Supelco supply the pesticide and PCB mixtures. It is recommended that these be used for traceability purposes. Stock standard solutions can then be diluted directly from the concentrates.

8.1.2 The pesticides must be made in two solutions, Individual A (INDA) and Individual B (INDB). See Tables 2 and 3 for preparation concentration. The surrogate standards tetrachloro-m-xylene and decachlorobiphenyl are added to all standards, samples and blanks.

8.1.3 Stock standard solutions must be kept refrigerated at 4°C (±2°C) for no longer than 6 months.

8.2 Calibration Standards

8.2.1 Prepare solutions of the calibration standards for INDA and INDB and each Aroclor at 5 concentration levels beginning at the quantitation limit and spanning the linear working range of the detector. See Tables 2 and 3 for the solution concentrations.

8.2.2 Calibration standards must be remade at least every six months, or verified against new standards before use.

8.3 Spiking Solutions

8.3.1 Surrogate Solution

8.3.1.1 The surrogates, Tetrachloro-m-xylene and Decachlorobiphenyl, are added to all standards, samples, matrix spikes, and blanks. Prepare a surrogate spiking solution of 0.2 ug/mL of each of the two compounds in acetone or methanol. The solution should be checked frequently for stability. The solution must be replaced after six months, or sooner, if comparison with quality control check samples indicates a problem.

Caution:Analysts must allow all spiking solutions to equilibrate to room temperature before use.

8.3.1.2 Aroclor Matrix Spiking Solution

8.3.1.2.1 Prepare a solution in acetone or methanol that contains Aroclors 1016 and 1260 in the concentration specified below:

Analyte	Concentration (ug/mL)
Aroclor 1016	6.0
Aroclor 1260	12.0

8.3.1.3 Pesticide Matrix Spiking Solution

8.3.1.3.1 Prepare a spiking solution in acetone or methanol that contains the following pesticides in the concentrations specified:

ANALYTE:	Concentration (ug/mL)

C2-250

Aldrin	0.25
Alpha-BHC	0.25
Beta-BHC	0.25
Delta-BHC	0.25
Gamma-BHC	0.25
alpha-Chlordane	0.25
gamma-Chlordane	0.25
4,4'-DDD	0.25
4,4'-DDE	0.25
4,4'-DDT	0.25
Dieldrin	0.25
Endosulfan I	0.25
Endosulfan II	0.25
Endosulfan Sulfate	0.25
Endrin	0.25
Endrin Aldehyde	0.25
Endrin Ketone	0.25
Heptachlor	0.25
Heptachlor Epoxide	0.25
4,4'-Methoxychlor	1.0

8.3.1.3.2 The solution must be prepared every six months, or sooner if the solution has degraded or concentrated.

8.3.1.4 Florisil Cartridge Check Solution

8.3.1.4.1 Florisil cleanup is not performed as a routine, but clients may occasionally request its use. Before use, pesticide recoveries are verified through use of the following check solution. The Florisil Check Solution consists of the following compounds made in acetone:

Analyte	Concentration (ug/mL)
2,4,5-Trichlorophenol	0.10
Alpha-BHC	0.02
Gamma-BHC	0.02
4,4'-DDD	0.04
4,4'-DDT	0.04
Dieldrin	0.04
Endosulfan I	0.04
Endrin	0.04
Heptachlor	0.02
4,4'-Methoxychlor	0.2
TCX	0.02
DCB	0.04

8.3.1.4.2 The check solution should exhibit 80 - 120% recovery of the pesticides and less than 5% recovery of 2,4,5-TCP.

8.3.1.5 Store all standard solutions in amber glass bottles or vials with a Teflon-lined screw cap at 4°C (±2°C) and protect from light.

8.4 GC Analysis Procedures

8.4.1 Gas chromatography conditions. These conditions are only recommended starting conditions. Each instrument may vary and require changes to be optimized.

8.4.1.1 Columns: column phases may be varied as long as equivalency is established.

8.4.1.1.1 Column 1 and Column 2:

Carrier gas (He or H2)	5-7 mL/minute
Makeup gas (N2)	90 mL/min
Injector temperature	220°C
Detector temperature	320°C
Initial temperature	150°C, hold 0.5 minute
Temperature program	150°C to 275°C at 6°C/min.
Final temperature	275°C hold 18 minutes.

8.4.1.1.2 Column 3:

Carrier gas (He or H2)	6 mL/minute
Makeup gas (N2)	90 mL/min
Injector temperature	220°C
Detector temperature	300°C
Initial temperature	140°C, hold 2 minutes
Temperature program	140°C to 240°C at 10°C/min, hold 5 min. at 240°C, 240°C to 265°C at 5°C
Final temperature	265°C, hold 18 minutes.

8.4.1.1.3 Column 4, Column 5, and Column 6

Carrier gas (He or H2)	2 mL/min
Makeup gas	(N2 or 5% Me/95% Argon) 90 mL/min
Injector temperature	220°C
Detector temperature	300°C
Initial temperature	110°C, hold 0.5 minutes
Temperature program	110°C to 140°C at 20°C/min, hold 0 min. at 140°C, 140°C to 280°C at 11°C/min.
Final temperature	280°C, hold 6.18 minutes.

8.4.2 Calibration

8.4.2.1 Refer to Method 8000A for proper calibration techniques. Percent relative standard deviation must be ≤ 20 for all analytes over the five point calibration range to assume linearity through the origin (and use the mean calibration factor for quantitation). Linear fit calibration or non-linear (quadratic) calibration may be used if % RSD is > 20 as

long as the correlation coefficient or coefficient of determination (for quadratic) are 0.99 or greater.

8.4.2.2 Calculation of retention time windows does not follow method 8000A/8080A because retention time windows established by this method are 0.00 to 0.01 (to the 1/100th of a minute) for all compounds. This is due to highly stable chromatographic systems. The use of such retention time windows for "real-world" samples may result in numerous false negatives, therefore, the windows established in EPA's SOW OLM03.2 have been adopted (See Table 6). 72-hour retention time studies (Form 9Es) are maintained for each instrument and column per method 8000A to prove the stability of the instruments.

8.4.2.3 The procedure of external calibration is used.

8.4.2.4 SWLO's policy is to analyze a five point initial calibration for all pesticide analytes and a single-point low-level calibration for all multi-component analytes. The single-point, low-level multi-response initial calibration is intended for a qualitative determination of multi-response compounds only. The low-level standard is analyzed to ensure that the PQL can be detected by the instrumentation. If a multi-component analyte is identified in a sample, then a five-point calibration is analyzed for the appropriate analyte and the sample is re-analyzed to obtain a quantitative value for the sample. If no multi-response analytes are detected, then no five-point multi-response analyte calibrations are run.

8.4.2.5 Because several of the pesticides may coelute on any single column, two calibration mixtures are provided to minimize the problem. The concentrations provided should be detectable on a GC/ECD suitable for use with this method. Mixtures of Aroclors other than 1016/1260 and 1221/1254 are not recommended for use with this method. See Tables 2 and 3 for pesticide calibration standards. See Table 4 for Aroclor standards.

8.4.2.6 Because of the low concentration of pesticide standards injected on a GC/ECD, column adsorption may be a problem when the GC has not been used for a day. Therefore, the GC column should be primed or deactivated by injecting a PCB or pesticide standard mixture approximately 4 times more concentrated than the mid-concentration standard. Inject this standard mixture plus 10 to 15 solvent rinses prior to beginning initial or daily calibration, especially if the instrument has set for more than several hours.

Caution: Several analytes, including Aldrin, may be observed in the injection just following this system priming. Always run an acceptable blank prior to running any standards or samples.

8.4.3 Gas chromatographic analysis:

8.4.3.1 Follow Method 8000A for instruction on the analysis sequence, appropriate dilutions, and identification criteria. Analysis of a mid-concentration standard every 12-hour shift (not to exceed 20 samples) is required. Analysis of low and high calibration verification standards will be performed for those clients which request the most strict interpretation of method 8081. Otherwise, the mid-concentration standard will suffice.

8.4.3.2 Examples of GC/ECD chromatograms generated by instruments with wide-bore capillary columns are presented in Figures 1 through 7.

8.4.3.3 If the peak response is less than 2.5 times the baseline noise level, the validity of the quantitative result may be questionable. The analyst should consult with the

source of the sample to determine whether further concentration of the sample is warranted by the context in which the result is to be used.

8.4.3.4 If the peak response exceeds the range of the highest standard, dilute the extract and reanalyze.

8.4.3.5 Identification of mixtures (i.e. PCBs and Toxaphene) is based on the characteristic "fingerprint" retention time and pattern of the indicator peak(s); and quantitation is based on the area under the characteristic peaks as compared to the area under the corresponding calibration peak(s) of the same retention time and pattern generated using external calibration procedures.

8.4.3.6 Identify compounds in the sample by comparing the retention times of the peaks in the sample chromatogram with those of the peaks in standard chromatograms. The retention time windows used to make identifications are the retention time windows listed in Table 6. Take into account the variance caused by matrix effect in samples.

8.4.3.7 Quantitation of the compound(s) of interest is premised on: 1) a linear response of the ECD to the ranges of concentrations of the compound(s) of interest encountered in the sample extract and the corresponding calibration extract; and 2) a direct linear proportionality between the magnitude of response of the ECD over the width(s) of the retention window(s) (the area under the characteristic or "fingerprint" peak[s]) in the sample and calibration extracts. Proper quantitation requires the appropriate selection of a baseline from which the area under the characteristic peak(s) can be calculated.

8.4.3.8 If compound identification or quantitation are precluded due to interference (e.g., broad, rounded peaks or ill-defined baselines are present), cleanup of the extract or replacement of the capillary column or detector may be warranted.

8.4.4 Quantitation of Multiple Component Analytes:

8.4.4.1 Residues which are mixtures of two or more components present problems in measurement. When they are found together, e.g., Toxaphene and DDT, the problem of quantitation becomes even more difficult. Suggestions are offered in the following sections for handing Toxaphene and PCBs.

8.4.4.2 Toxaphene

8.4.4.2.1 Quantitative calculation of Toxaphene is difficult, but reasonable accuracy can be obtained. To calculate Toxaphene on GC/ECD, quantitate using the average of the four (4) major peaks or the total area of the Toxaphene pattern.

8.4.4.3 Polychlorinated biphenyls (PCBs)

8.4.4.3.1 Quantitation of residues of PCB involves problems similar to those encountered in the quantitation of Toxaphene. In each case, the chemical is made up of numerous compounds which generate multi-peak chromatograms. Also, in each case, the chromatogram of the residue may not match that of the standard.

8.4.4.3.2 PCB residues are quantitated by comparison to one or more of the Aroclor materials, depending on the chromatographic pattern of the residue. A choice must be made as to which Aroclor or mixture of Aroclors will produce a chromatogram most similar to that of the residue.

8.4.4.3.3 PCB Quantitation — Quantitate the PCB residues by comparing the responses of 3 to 5 major peaks in each appropriate Aroclor standard with the peaks obtained from the chlorinated biphenyls in the sample extract. The amount of Aroclor is

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calculated using each of the major peaks, and the results of the 3 to 5 determinations are averaged. Major peaks are defined as those peaks in the Aroclor standards that are at least 30% of the height of the largest Aroclor peak. Later eluting Aroclor peaks are generally the most stable in the environment. Smaller peaks may be chosen for quantitation if major Aroclor and pesticide peaks coelute.

8.4.5 See the "Standard Operating Procedure for the Preventive Maintenance of the Gas Chromatographs" for specific GC maintenance procedures.

9.0 Calculations

9.1 Calibration Factor = $\frac{\text{Peak Area (or Height) of the Compound in the Standard}}{\text{Mass of the Compound Injected}}$

9.2 Mean CF = $\frac{\text{Sum of CF for each level}}{\text{number of levels}}$

9.3 SD = Square root $\left[\frac{\text{Sum of (CF of each individual level - Mean CF)}^2}{\text{(number of levels - 1)}}\right]$

9.4 RSD = $\frac{\text{SD}}{\text{Mean CF}} \times 100$

9.5 See SW-846, Revision 2, January 1995, page 8000B - 20 through 22 for linear and higher order fit equations.

9.6 % Difference = $\frac{\text{Mean CF - CF from the calibration verification standard}}{\text{Mean CF}} \times 100$

9.7 % Drift (used for linear and higher order fits) = $\frac{\text{Calculated conc. - Nominal conc.}}{\text{Theoretical conc.}} \times 100$

9.8 Calculate percent breakdown as follows:

$$\text{\% Breakdown DDT} = \frac{\text{Area of (DDD + DDE)}}{\text{Area of DDT + Area DDD + Area DDE}} \times 100$$

$$\text{\% Breakdown Endrin} = \frac{\text{Area of (Endrin aldehyde + Endrin ketone)}}{\text{Area of Endrin aldehyde + Area of Endrin ketone + Area of Endrin}} \times 100$$

9.9 Determine the concentration of individual compounds in the sample.

9.9.1 Sample Concentration Calculation:

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9.9.1.1 Aqueous Samples

Concentration (ug/L)=[As] [V(t)][D] / [Mean CF][V(i)] [V(s)]

where:

As = Area (or height) of the peak for the analyte in the sample.
V(t) = Total volume of concentrated extract (uL).
D = Dilution factor.
Mean CF = Mean calibration factor.
V(i) = Volume of extract injected (uL).
V(s) = Volume of aqueous sample extracted (mL).

9.9.1.2 Nonaqueous Samples

Concentration (ug/Kg)=[As] [V(t)][D] / [Mean CF][V(i)] [W(s)]

where:

As = Area (or height) of the peak for the analyte in the sample.
V(t) = Total volume of concentrated extract (uL).
D = Dilution factor.
Mean CF = Mean calibration factor.
V(i) = Volume of extract injected (uL).
W(s) = Weigth of sample extracted (g).

9.4 For multicomponent mixtures (chlordane, toxaphene. and PCBs) match retention times and patterns (relative ratios) of peaks in the standards with peaks in the sample. Quantitate every identifiable peak unless interference with individual peaks persist after cleanup. Add peak height or peak area of each identified peak in the chromatogram. Calculate as total response in the sample versus total response in the standard.
9.5 Report results in ug/L without correction for recovery data. All QC data obtained should be reported with the sample results.

10.0 Quality Control

10.1 Refer to Chapter One of SW-846 for specific quality control (QC) procedures. Quality control to validate sample extraction is covered in Method 3500 and in the extraction method utilized. If the extract cleanup was performed, follow the QC in Method 3600 and in the specific cleanup method.

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10.2 DDT and endrin are easily degraded in the injection port, if the injection port or front of the column is contaminated with buildup of high boiling residue from sample injection. Check for degradation problems by injecting a mid-concentration standard containing 4,4'-DDT and endrin. Look for the degradation products of 4,4'-DDT (4,4'-DDE and 4,4'-DDD) and endrin (endrin ketone and endrin aldehyde). If degradation of either DDT or endrin exceeds **15%**, take corrective action before proceeding with calibration, (refer to Method 8000A and Section 4.4 of Method 8081). See section 9.0, Calculations, for the procedure for calculating % breakdown.

10.3 Calculate surrogate standard recovery on all samples, blanks, and spikes. Determine if the recovery is within limits established. If recovery is not within limits, the following are required:

10.3.1 Check to be sure there are no errors in calculations, surrogate solutions and internal standards. Also, check instrument performance.

10.3.2 Recalculate the data and/or reanalyze the extract if any of the above checks reveal a problem.

10.3.3 If the method blank recoveries are out of the advisory limits, reanalyze the sample to rule out instrument problems. Re-extract and reanalyze the samples if none of the above are a problem.

10.4 The breakdown of DDT and endrin should be measured before samples are analyzed. Injector maintenance and recalibration should be completed if the breakdown is greater than **15%** for either compound.

10.5 Include a mid-concentration calibration standard every 12-hour shift (not to exceed 20 samples) in the analysis sequence as a calibration check. The response factors for the mid-concentration calibration should be within **15 percent** of the average values for the multi-concentration calibration. When this continuing calibration is out of this acceptance window, the laboratory should stop analyses, clean the injector, replace the septum and recalibrate the system. Samples that are not bracketed by acceptable standards must be reinjected to avoid errors in quantitation. If the calibration verification standard exceeds the criteria in the positive direction (upward drift from the initial calibration) and no positive identifications were made for target analytes, the samples do not require reanalysis and results may be reported. This scenario shows that any target analytes at the QL would have been identified if they were present.

10.6 Instrument blanks must not contain any confirmed target analyte greater ½ of the reporting limit for the associated samples. Method blanks must not contain any confirmed target analyte greater than the reporting limit for the associated samples. If contamination is noted, stop all analyses, determine the source of the contamination, remove the source, and confirm the source is gone by reanalyzing an instrument blank and/or method blank as appropriate.

10.7 Laboratory control spike and matrix spike/matrix spike duplicate recoveries and RPDs are compared to client specified control limits if available. Otherwise, in-house generated control limits will be used (See "Blue Book" for most recent control limits).

11.0 Waste Disposal

See the Standard Operating Procedure for the Disposal of Waste.

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12.0 Method Performance

12.1 The MDL concentrations are updated annually and are obtained using organic-free reagent water and sandy loam soil. The MDL results are maintained in SWLO's "Red book" by the QA Department. Details for determining MDLs are given in Chapter One of SW-846 and the SOP for the Definition and Determination of Detection Limits, SWL-GA-113. The MDL actually achievable in a given analysis will vary depending on detector response characteristics, irreducible noise from instrument electronics and matrix effects.

12.2 The accuracy and precision obtainable following this method will be determined by the sample matrix, sample preparation technique, optional cleanup techniques, and calibration procedures used.

12.3 The laboratory should maintain control charts of LCS, MS, and MSD recoveries and relative percent deviations between MS and MSD recovery for accuracy and precision data. Surrogate recovery should also be plotted. The control charts are monitored monthly for LCS and quarterly for MS and are maintained on file in the laboratory. Control limits are updated on an annual basis.

12.4 Table 1 gives the retention times that can be achieved by this method for the organochlorine pesticides. Examples of the separations on a DB-608 column are shown in Figures 1 through 7. QLs are calculated by multiplying the MDL value for an analyte by a number ranging from 5-10. The QL will, in no instance, be less than the MDL. See "Red Book" for SWLO's QLs.

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Table 1: Gas Chromatographic Retention Times for the Organochlorine Pesticides for method 8081

Compound	Retention Time (min) DB 608(1)	DB 1701(1)
Aldrin	11.84	12.50
alpha-BHC	8.14	9.46
beta-BHC	9.86	13.58
delta-BHC	11.20	14.39
gamma-BHC (Lindane)	9.52	10.84
alpha-Chlordane	15.24	16.48
gamma-Chlordane	14.63	16.20
4,4'-DDD	18.43	19.56
4,4'-DDE	16.34	16.76
4,4'-DDT	19.48	20.10
Dieldrin	16.41	17.32
Endosulfan I	15.25	15.96
Endosulfan II	18.45	19.72
Endosulfan Sulfate	20.21	22.36
Endrin	17.80	18.06
Endrin aldehyde	19.72	21.18
Heptachlor	10.66	11.56
Heptachlor epoxide	13.97	15.03
4,4'-Methoxychlor	22.80	22.34
Toxaphene	MR	MR
Aroclor-1016	MR	MR
Aroclor-1221	MR	MR
Aroclor-1232	MR	MR
Aroclor-1242	MR	MR
Aroclor-1248	MR	MR
Aroclor-1254	MR	MR
Aroclor-1260	MR	MR

MR = Multiple peak responses.
NA = Data not available.

(1) Temperature program — These times are representative only.

12.5 Concentrations of Calibration standards are given in Tables 2-7:

Table 2: Pesticide Calibration Standards -- Individual Standard Mixture A - Low Point Concentration ng/mL (Level 1)

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Individual Standard Mixture A - Low Point Concentration ng/mL (Level 1)	
alpha-BHC	3.75
Heptachlor	3.75
gamma-BHC	3.75
Endosulfan I	3.75
Dieldrin	7.5
Endrin	7.5
4,4'-DDD	7.5
4,4'-DDT	7.5
Methoxychlor	37.5
Tetrachloro-m-xylene	3.75
Decachlorobiphenyl	7.5

Table 3: Individual Standard Mixture B — Low Point Concentration ng/mL (Level 1)

Individual Standard Mixture B — Low Point Concentration ng/mL (Level 1)	
beta-BHC	3.75
delta-BHC	3.75
Aldrin	3.75
Heptachlor epoxide	3.75
alpha-Chlordane	3.75
gamma-Chlordane	3.75
4,4'-DDE	7.5
Endosulfan sulfate	7.5
Endrin aldehyde	7.5
Endrin ketone	7.5
Endosulfan II	7.5
Tetrachloro-m-xylene	3.75
Decachlorobiphenyl	7.5

Note: Level 2 concentration = 2 x Level 1 Level 3 concentration = 4 x Level 1
Level 4 concentration = 8 x Level 1 Level 5 concentration = 16 x Level 1
Prepare all solutions in hexane or isooctane. Keep stored at 4°C (±2°C) no longer than 6 months.

Table 4: Multi-response calibration standards – Low Point Concentration ng/mL (Level 1)

Aroclor/TOX	Low Point Concentration (ng/mL) (Level 1)
Toxaphene	250

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AR1221	100
AR1232	100
AR1242	100
AR1248	100
AR1254	200
AR1016	100
AR1260	200

Note: Each Aroclor standard is prepared independently with the surrogate DCB and TCX at 5 ng/mL except that AR1016 and AR1260 are combined as AR1660. The low standard concentration standard may vary as long as the concentration is less than or equal to the PQL.

Level 2 concentration = 2 x Level 1 Level 3 concentration = 4 x Level 1
Level 4 concentration = 8 x Level 1 Level 5 concentration = 16 x Level 1
Prepare all solutions in hexane or isooctane. Keep stored at 4°C (±2°C) no longer than 6 months.

Table 5: Performance Evaluation Mixture (ng/mL)

Performance Evaluation Mixture — (ng/mL)	
gamma-BHC	10.0
alpha-BHC	10.0
beta-BHC	10.0
4,4'-DDT	100.0
Endrin	50.0
Methoxychlor	250.0
Tetrachloro-m-xylene	20.0
Decachlorobiphenyl	20.0

Performance Evaluation Mixture (PEM) - prepare the PEM in hexane or isooctane at the concentration levels listed below. The PEM must be prepared every six months, or more often if the solution has degraded or concentrated.

Table 6: Retention Time Windows

ANALYTE:	RT Window (± minutes)
Aldrin	0.05
Alpha-BHC	0.05

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Beta-BHC	0.05
Delta-BHC	0.05
Gamma-BHC	0.05
alpha-Chlordane	0.07
gamma-Chlordane	0.07
4,4'-DDD	0.07
4,4'-DDE	0.07
4,4'-DDT	0.07
Dieldrin	0.07
Endosulfan I	0.07
Endosulfan II	0.07
Endosulfan Sulfate	0.07
Endrin	0.07
Endrin Aldehyde	0.07
Endrin Ketone	0.07
Heptachlor	0.05
Heptachlor Epoxide	0.07
4,4'-Methoxychlor	0.07
Toxaphene	0.07
Aroclor-1016	0.07
Aroclor -1221	0.07
Aroclor -1232	0.07
Aroclor -1242	0.07
Aroclor -1248	0.07
Aroclor -1254	0.07
Aroclor -1260	0.07
TCX	0.05
DCB	0.10

13.0 References

U.S.EPA SW-846, "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods:, Revision 2, September 1994, Methods 8000A, 8080A, and 8081.

Standard Operating Procedure, "Control Charts", SWL-GA-112.

Standard Operating Procedure, "Definition and Determination of Detection Limits", SWL-GA-113.

14.0 Definitions

N/A = not applicable

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PROCEDURAL DOCUMENTS - SYSTEM TRANSITION

Please note that the following document is in transition from a previous system. The document identification number (listed below) has been assigned in compliance with the SWL SOP titled:

"Preparation, Review, Revision, and Control of Procedural Documents", SWL-GA-101, Rev. 1.0 — 05/14/97

The ID given below integrates the following SOP into the new system set forth in GA-101 prior to its annual review (or conversion of format and numbering). Each ID is unique and a "key" field in the new document control database system. The assignment of a new number is therefore necessary for its tracking in the new system.

Upon revision or review, the following document will be re-issued in full compliance with the guidelines set forth in GA-101, therefore consistent with newly converted or written procedural documents.

Existing ID: MS340

NEW DOCUMENT IDENTIFICATION NUMBER:

SWL-OV-309

Standard Operating Procedure

"Volatile Organics -- TCLP"
Rev. No. 3.2
Issued: 11/26/96

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AMERICAN ANALYTICAL & TECHNICAL SERVICES, INC./SOUTHWEST LABORATORY OF OKLAHOMA, INC.

STANDARD OPERATING PROCEDURE FOR THE ANALYSIS OF VOLATILE ORGANICS TOXICITY CHARACTERISTIC LEACHATE PROCEDURE (TCLP)

MS340

REV 3.2— 11/26/96

Document Control #AATSWL-MS340-2028

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GC/MS ANALYSIS OF PURGEABLE ORGANICS

I. SCOPE/SUMMARY OF METHOD

A. An inert gas, helium, is bubbled through a 5 mL sample contained in a specifically designed purging chamber at ambient temperature. The purgeables are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column where the purgeables are trapped. After purging is completed, the sorbent column is heated and backflushed with the helium to desorb the purgeables onto a gas chromatographic column. The gas chromatograph is temperature programmed to separate the purgeables which are then detected with a mass spectrometer.

B. An aliquot of the sample is diluted with reagent water when dilution is necessary. A 5 mL aliquot of the dilution is taken for purging.

C. If the amount of a TCLP compound detected is outside the linear range of the instrument, then a smaller sample size is analyzed.

II. SAMPLE PRESERVATION/HOLDING TIMES

A. TCLP leachates are analyzed within 14 days of Zero Headspace Extraction.

B. Waters should be preserved with HCL (pH <2) and received and stored cool (4°C) until analysis. Sodium Thiosulfate (4 drops of 10%) is also used when residual chlorine is present in the water.

III. APPARATUS AND MATERIALS

A. *Micro syringes* — 10 µL and larger, 0.006 inch ID needle.

B. *Syringe valve* — two-way, with Luer ends (three each), if applicable to the purging device.

C. *Syringe* — 5 mL, gas tight.

D. *Balance, Analytical* — capable of accurately weighing 0.0001 g. and a top-loading balance capable of weighing 0.1 g.

E. *Glassware*

1. Bottle — 40 mL, screw cap, with Teflon cap liner.

F. *Purge and trap device* — The purge and trap device consists of three separate pieces of equipment; the sample purger, trap and the desorber. (Tekmar 2000 with 2016 and 2032 autosamplers).

IV. GC/MS SYSTEM

A. *Gas chromatograph* — An analytical system complete with a temperature programmable gas

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chromatograph suitable for on-column injection and all required accessories including syringes, analytical column, and gases.

B. *Column* — DB624, 75m, 0.53mm ID, 3.0 um (J&W Scientific Cat. No. 125-1334) or equivalent.

C. *Jet Separator* — Glass (SIS Cat. No. 113505).

D. *Mass spectrometer* — Capable of scanning from 35 to 300 amu every three seconds, utilizing seventy volts (nominal) electron energy in the electron impact ionization mode and producing a mass spectrum which meets all the criteria when 50 ng of 4-Bromofluorobenzene (BFB) is injected through the gas chromatograph inlet (see Table 4).

E. Calibration requirements can be found in Tables 8 and 9.

V. REAGENTS AND STANDARDS

A. *Reagent water* — Produced by filtering tap water through an organic filtration system containing activated charcoal prior to use.

1. *Methanol* — Pesticide quality or equivalent.

2. *Stock standard solutions* — Stock standard solutions are purchased and are traceable to EPA supplied standards. These solutions are stored in Teflon sealed screwcap bottles at 10°C to 20°C.

B. *Secondary dilution standards* — Using stock standard solutions, prepare secondary dilution standards in methanol that contain the compounds of interest, either singly or mixed together. (See GC/MS Calibration in Exhibit E). Secondary dilution standards should be stored with minimal headspace and should be checked frequently for signs of degradation or evaporation especially just prior to preparing calibration standards from them.

C. *Surrogate Standard Spiking Solution.*

1. Prepare stock standard solutions for toluene-d_8, p-bromofluorobenzene, and 1,2-Dichloroethane-d_4 in methanol. Prepare a surrogate standard spiking solution from these stock standards at a concentration of 25 µg/mL in methanol.

D. *Purgeable Organic TCLP Matrix Standard Spiking Solution.*

1. Prepare a spiking solution in methanol that contains each TCLP compound at a concentration of 50 µg/mL.

E. *Internal Standard Solution*

1. The three internal standards are Bromochloromethane, 1,4-Difluorobenzene and chlorobenzene-d_5. Prepare a spiking solution containing each of the internal standards.

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It is recommended that the secondary dilution standard be prepared at concentration of 25 µg/mL of each internal standard compound. The addition of 10 µL of this standard to 5.0 mL of sample or calibration standard would be equivalent of 50 µg/L.

VI. GC/MS OPERATING CONDITIONS

A. These performance test require the following instrumental parameters:

1. Electron Energy: 70 Volts (nominal)
2. Mass Range: 35-300
3. Scan Time: to give at least 5 scans per peak, not to exceed one second per scan

B. All samples and standard solutions must be allowed to warm to ambient temperature before analysis.

C. Recommended operating conditions for the gas chromatograph - Column Conditions: DB-624, 75m, 0.53mm ID, 3 um film thickness or equivalent at a flow rate of 30 mL/min. Column temperature is isothermal at 45°C for five minutes, then programmed at 5°C per minute to 105°C. further programmed at 10°C/min. to 180°C.

D. Adjust the purge gas (helium) flow rate to 35 ± 3 mL/min. Variations from this flow rate may be necessary to achieve better purging and collection efficiencies for some compounds, particularly chloromethane and bromoform.

VII. CALIBRATION

A. *Tune*

1. Before analysis of samples can begin, the instrument must meet all calibration criteria. The first step in the calibration of Volatiles GC/MS system is to demonstrate the ionization and fragmentation of standard mass spectral tuning compounds. This is accomplished, as well as sensitivity check, with the use of 4-Bromofluorobenzene. This standard is run daily to validate the GC/MS system and to begin the 12-hour clock. BFB is analyzed by direct injection of a 50 ng aliquot into the instrument.
2. When the analysis is complete, the tune is checked against standard criteria (See Table 3 for Tune Criteria). SW846 Method 8240 tune criteria for BFB is used for all volatile test codes, and before any other calibration standards or sample analysis is performed.
3. If the tune fails to meet the criteria, rerun BFB. If it fails again, the instrument must be retuned through "Auto Tune" or "Manual Tune". BFB should be run again. If it meets criteria, continue with the calibration sequence. If it fails again, contact your supervisor. Instrument maintenance such as source cleaning may be required.

B. *Initial Calibration*

1. Once BFB has met its tune criteria, calibration may begin. Initial calibration of the GC/MS is established and validated by the analysis of EPA traceable standards at five concentration levels which are 5, 20, 50, 100, and 200 ppb. These standards are made up in 5 mL of reagent water

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and purged on the Tekmar before analysis by GC/MS. The internal standard calibration procedure is used. In addition to surrogate recovery compounds, standards and sample extracts are spiked with internal standards that span the retention time range of the analytes of interest.

2. The Response Factors (RF) for the analytes in the standard are calculated and stored in a Target Method File. The concentration of the analytes during sample analysis is calculated using the RF of each analyte determined in each day's calibration. These equations are shown in Table 13.

3. The calibration standards are prepared by filling a syringe with 5 mL of reagent water and spiking with the standards as shown below.

Internal Std. (µL)	Surrogate (µL)	25 ppm Stock Std. (µL)	Int. Std. Final conc. (ng/mL)	Stock Std. Final conc. (ng/mL)
10	1	1	50	5
10	4	4	50	20
10	10	10	50	50
10	20	20	50	100
10	40	40	50	200

4. Attach the syringe to the syringe valve assembly on the Tekmar. Open the syringe valve and inject the sample into the purging chamber. Close the purging device valve. Low level soil calibrations must be heated to 40°C before purging. Water, medium level soils, and high level soils purge at ambient temperature. Purge the sample for 8.0 min. At the conclusion of the purge time, introduce the trapped materials to the GC column by the Desorb Mode where the trap is rapidly heated to 180°C for four minutes.

5. While the trap is being desorbed into the GC, you may empty the purging chamber. After desorbing the sample for four minutes, bake the trap for four minutes at 225°C. After bake, allow the trap to cool to less than 30°C making it ready to purge the next standard or sample.

6. The standard will automatically be quantitated into a report from which concentrations, areas, response factors, standard deviations, and percent differences are calculated.

7. SW846 Method 8240 criteria for Volatiles Calibration Check Compounds (CCC) is employed:

CCC
1,1-Dichloroethene
Chloroform
1,2-Dichloropropane
Toluene
Ethylbenzene
Vinyl Chloride

8. The %RSD is calculated automatically for all compounds. This %RSD must be within 30% for all CCC Compounds.

9. SW846 Method 8240 criteria for System Performance Check Compounds (SPCC) is employed for the compounds below.

SPCC
Chloromethane
1,1-Dichloroethane

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Chlorobenzene
Bromoform
1,1,2,2-Tetrachloroethane

10. The average RF is calculated for all compounds and the RF for SPCC compounds must be at least 0.300, except for Bromoform, which has a minimum RF of 0.100. If the five-point does not meet criteria, check to see if a standard needs to be rerun. The standards could have been added at the wrong amount or the purge may have been bad. If reanalyzing a standard will not solve the problem, contact your supervisor. It could be related to purge chambers, multiplier, flows, etc.

11. If the five-point does meet criteria, and there is time remaining in the 12-hour time period, samples may be run. The 50 ppb Standard's response factors are used to calculate the concentrations in samples of the TCLP compounds.

C. *Continuing Calibration*

1. A continuing calibration of the GC/MS uses the same EPA traceable standards, but at only one level. This level is 50 ppb The analysis is performed in the same manner as stated for the initial calibration. The continuing calibration checks the same CCC and SPCC compounds as the initial calibration. The CCC compounds must have a %Difference (%D) of less than 20% and the SPCC must have an RF from that particular day's calibration of greater than 0.300, except for Bromoform, which must be greater than 0.100. The continuing calibration is performed after the BFB and must meet all criteria before any other analysis continues.

2. If the criteria is not met, the standard must be rerun. It is possible it was not prepared correctly, or that there was a bad purge. However, if the second standard still doesn't meet criteria, an initial calibration must be run or corrective action performed.

3. Despite the fact that only 10 of the compounds in the calibration standards are TCLP compounds, we ensure that all of the compound list meets CCC/SPCC criteria before continuing with the Method Blank. This allows operators to analyze more than one test code within a twelve hour calibration sequence. The criteria that must be met is described in the Initial and Continuing Calibration Sections of this Standard Operation Procedure.

VIII. QC SAMPLES

A. *Method Blank*

1. The method blank is analyzed once the calibration has been met, whether it be an initial or continuing calibration. The method blank, called the "VBLK", consists of loading a syringe with 10 mL of reagent water spiked with 10 µL of Internal Standard and 10 µL of Surrogate which yields a concentration for both of 50 ppb. It is used to establish the analytical curve by taking into account any background responses during the calibration process. It is also used to check for carryover contamination after a standard run or after a contaminated sample run.

2. An acceptable blank must meet the internal standard criteria stated in the "Sample Analysis" section and the surrogate recoveries in Table 4. It must also contain less than or equal to five times the Practical Quantitation Limits (PQL) of Methylene Chloride, Acetone, Toluene, and 2-Butanone. For all other TCLP compounds, the blank must contain less than or equal to the PQL of any single TCLP analyte.

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3. The laboratory meets this criteria which includes all of the Target Compound List to allow operators to analyze more than one test code within a twelve hour calibration sequence.
4. If a blank exceeds this criteria, corrective measures must be taken immediately. This action first consists of immediately reanalyzing the blank. If it still exceeds criteria, consult your supervisor immediately.

B. *Laboratory Control Spike (LCS and LCSD)*

1. The Laboratory Control Spikes (LCS and LCSD) are the last analysis of the calibration sequence. They are used as internal QA/QC checks. The LCS and LCSD are analyzed daily following the method blank to check the proficiency of the analysis in terms of working standards preparation, to monitor standard degradation, and to check traceability of the prepared standards from commercial reference materials to EPA reference materials. These results are also used to create our laboratory control charts.
2. The sample is prepared by loading a syringe with 5 mL of reagent water spiked with 10 µL of Internal Standard, 10 µL of Surrogate, and 10 µL of LCS Mix. This yields a final concentration for all three standards of 50 ppb.
3. The LCS and LCSD contain most of the compounds found in the daily calibration standards. Their limits may be found in Table 6. The LCS and LCSD must meet these limits, as well as the surrogate and internal standard criteria. If these criteria are not met, corrective action must be taken immediately. This includes rerunning the LCS and/or LCSD. If the compounds are still out of limits, a new standard should be prepared. LCS limits are advisory like Matrix Spike Compounds, but since they are internal QC limits set forth by our laboratory, every effort should be made to meet these limits.

IX. SAMPLE ANALYSIS

A. All TCLP samples are transferred to our AATS-TUL facility. If the sample is less than 0.5% solids, it is liquid filtered and sent back to our laboratory for analyses of this filtered sample. If the sample is greater than 0.5% solids, it undergoes a zero headspace extraction. This TCLP extract is then analyzed by our laboratory. Because of these procedures, all TCLP samples are analyzed under a water calibration.

B. An extraction sheet accompanies all TCLP samples whether they are liquid filtered or extracted. This sheet contains pertinent information used in the final report including date of sample receipt, extraction or filter date, and any comments related directly to the appearance of the sample. The percent moisture is also noted on this extraction sheet.

C. Before analysis, note any headspace in the bottle. Open the sample/extract bottle which has been allowed to come to ambient temperature, and carefully fill the sample into the syringe barrel. The Zero Headspace Extraction Blank requires a volume of 5mL, but all samples only require a maximum volume of 1mL due to the higher detection limits. A total of 5mL volume is still needed for purging though requiring the addition of 4mL of reagent water.

D. Add 10 uL of the Surrogates spiking solution and 10 uL of the Internal Standard spiking

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solution through the valve bore of the syringe.

E. Water samples are loaded in the following manner to ensure the proper sample is placed into its designated purge chamber on the Tekmar Autosampler:

1. A test tube rack is filled with the samples' 40 mL Voa vials. The syringes to be used are placed in front of the samples.
2. All sample purge tubes are labeled with the SWLO ID Number and Autosampler position. Each analyst verifies as he/she loads each sample into the purge chambers that the syringe matches the ID on the purge tube.
3. Follow the same purging method as stated during calibration.

F. TCLP protocol requires only a matrix spike and not a duplicate. However, clients often request a standard MS/MSD which is provided for them in the same manner as a CLP MS/MSD. The MS/MSD Mix contains all TCLP Compounds (Table 5).

X. INTERFERENCES

A. Interferences purged or coextracted from the samples will vary considerably from source to source, depending upon the particular sample or extract being tested. The analytical system, however, should be checked to ensure freedom from interferences, under the analysis conditions, by analyzing method blanks.

B. Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) through the septum seal into the sample during shipment and storage. A zero headspace extraction blank can serve as a check on such contamination.

C. Cross contamination can occur whenever high-concentration and low-concentration samples are analyzed sequentially. Whenever an unusually concentrated sample is analyzed, it should be followed by the analysis of organic-free reagent water to check for cross contamination. The purge-and-trap system may require extensive bake-out and cleaning after a high-concentration sample.

D. The laboratory where volatile analysis is performed should be completely free of solvents.

E. Impurities in the purge and gas and from organic compounds outgassing from the plumbing ahead of the trap account for the majority of contamination problems. The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running reagent blanks. The use of non-TFE plastic coating, non-TFE thread sealants, or flow controllers with rubber components in the purging device should be avoided.

XI. QUALITY ASSURANCE/QUALITY CONTROL (QA/QC)

A. The GC/MS system should be tuned to meet the BFB specifications in Section VI.A. and Table 3.

B. There should be an initial calibration of the GC/MS system as specified in Section VI.B. or a continuing calibration (Section VI.C.)

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C. Before processing any samples, the analyst should demonstrate, through the analysis of a method blank, that interferences from the analytical system, glassware, and reagents are under control. Each time a set of samples is extracted, or there is a change in reagents, a method blank should be processed as a safeguard against chronic laboratory contamination. The blank samples should be carried through all stages of sample preparation and measurement.

D. A Laboratory Control Spike (LCS) and a Laboratory Control Spike Duplicate (LCSD) are analyzed for each batch of 20 samples. The percent recoveries of the LCS/LCSD are compared to historically generated control limits to determine whether the analytical system is in control (see table 6.)

E. A Matrix Spike (MS) and a Matrix Spike Duplicate (MSD) are analyzed as per client request to determine whether there are matrix effects on the recoveries of particular compounds. Percent Recoveries are compared to Method Limits and outliers are flagged to alert the data user of matrix effects (see table 5.)

F. Surrogate Recoveries from every sample are compared to method control limits (see table 4.) If any surrogates are out of control, the sample is reanalyzed. The reanalysis is then reported if surrogates are within limits or the reanalysis is used to establish a matrix effect on the surrogate recoveries.

XII. ANALYSIS/REPORT CRITERIA/VALIDATION

A. A sample is acceptable when it meets the following criteria:

1. The internal standards' EICP areas are within a factor of 2(-50%/+100%) of the calibration's internal standards.
2. The internal standards' retention times are within ±30 seconds of the calibration's internal standards.
3. Surrogate recoveries are within the limits stated in Table 4.
4. Any TCLP compounds found in the sample are within the linearity range, with any dilutions showing compounds in an acceptable range of linearity. If a sample analysis contains any target compounds above the linear range, all subsequent samples will be reviewed for those target compounds. The target compounds must be below PQL to be acceptable; otherwise they must be reanalyzed.
5. Chromatogram shows no signs of carryover or instrument deficiencies.

B. If sample does not meet internal standards' or surrogates' criteria, it must be reanalyzed. If the reanalysis meets all criteria, it is submitted. If the reanalysis duplicates the results from the first analysis, both analyses are submitted.

C. A sample report is complete when it contains all of the following in order:

1. Chromatogram
2. Quant Report
3. Manual Integrations
4. Spectra of any TCLP compounds, excluding internal standards and surrogates.
5. Tentatively Identified Compounds (TICs)
6. Internal Standards/Surrogates

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XIII. HEALTH & SAFETY

A. Care must be used when handling standards and samples. All samples should be treated as if they were toxic. Consult the Material Safety Data Sheets for information on the hazards involved with each compound and/or standard mixes. Any spills that may occur are cleaned up immediately.

XIV. DATA ARCHIVAL

A. Instrument data is stored in two separate locations before being archived to ensure data does not become erased or corrupt. Data is stored on both the instrument's Chemstation and the Unix-based processing system. Data is archived from the Unix-based system using the "TAR" format onto 60 m DAT tapes. No more than two months worth of data is archived onto the same tape. Once a tape contains two months of data, a tape directory is printed and bound for permanent storage. Each tape is labeled with the instrument ID and the date range of data contained on the tape.

XV. REFERENCES

A. EPA's "Test Methods for Evaluating Waste" (SW846, 3rd Edition, Rev. 1, July, 1992 and September, 1994).

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TABLE 1.

Characteristic Ions for Surrogates and
Internal Standards for Volatile Organic Compounds

Compound	Primary Ion	Secondary Ion(s)
SURROGATES		
4-Bromofluorobenzene	95	174, 176
1,2-Dichloroethane d-4	65	102
Toluene d-8	98	70, 100
INTERNAL STANDARDS		
Bromochloromethane	128	49, 130, 51
1,4-Difluorobenzene	114	63, 88
Chlorobenzene d-5	117	82, 119

TABLE 2.

Characteristic Ions for Volatile TCLP Compounds

Parameter	Primary Ion	Secondary Ion(s)
Vinyl chloride	62	64
1,1-Dichloroethene	96	61,98
Chloroform	83	85
1,2-Dichloroethane	62	64,100,98
2-Butanone	43	57,72
Carbon tetrachloride	117	119,121
Trichloroethene	130	95,97,132
Benzene	78	
Tetrachloroethene	164	129,131,166
Chlorobenzene	112	114

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TABLE 3.

BFB Key Ions and Abundance Criteria

Mass	Ion Abundance Criteria
50	15.0 - 40.0 percent of mass 95
75	30.0 - 60.0 percent of mass 95
95	base peak, 100 percent relative abundance
96	5.0 - 9.0 percent of mass 95
173	less than 2.00 percent of mass 174
174	greater than 50.0 percent of mass 95
175	5.0 - 9.0 percent of mass 174
176	greater than 95.0 percent but less than 101.0 percent of mass 174
177	5.0 - 9.0 percent of mass 176

TABLE 4.

Surrogate Recovery Limits

Fraction	Surrogate	Water	Soil
Volatile	Toluene-d_8	88-110	81-117
Volatile	4-Bromoflourobenzene	86-115	74-121
Volatile	1,2-Dichloroethane-d_4	76-114	70-121

TABLE 5.

TCLP Matrix Spike Recovery Limits

Fraction	Matrix Spike Compound	Water
Volatile	Vinyl Chloride	80-120
Volatile	1,1-Dichloroethene	80-120
Volatile	Chloroform	80-120
Volatile	1,2-Dichloroethane	80-120
Volatile	2-Butanone	80-120
Volatile	Carbon Tetrachloride	80-120
Volatile	Trichloroethene	80-120
Volatile	Benzene	80-120
Volatile	Tetrachloroethene	80-120
Volatile	Chlorobenzene	80-120

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TABLE 6.

Laboratory Control Spike Recovery Limits

Fraction	Laboratory Control Spike Compound	Water
Volatile	Benzene	70-134
Volatile	Chlorobenzene	72-135
Volatile	Chloroform	57-152
Volatile	Carbon Tetrachloride	37-182
Volatile	1,1-Dichloroethene	54-161
Volatile	Trichloroethene	64-149
Volatile	Tetrachloroethene	61-141
Volatile	Vinyl Chloride	65-167
Volatile	2-Butanone	42-173
Volatile	1,2-Dichloroethane	59-151

TABLE 7.

Volatile Internal Standards with Corresponding
TCLP Analytes Assigned for Quantitation

Bromochloromethane	1,4-Difluorobenzene	Chlorobenzene-d_5
Vinyl Chloride	Carbon Tetrachloride	Tetrachloroethene
1,1-Dichloroethene	Trichloroethene	Chlorobenzene
Chloroform	Benzene	
1,2,-Dichloroethane		
2-Butanone		

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TABLE 8.

Volatile Calibration Check Compounds (CCC)

1,1-Dichloroethene Chloroform 1,2-Dichloropropane Toluene Ethylbenzene Vinyl Chloride	Initial Calibration 5,20,50,100,and 200 ppb Max %RSD: 30% Continuing Calibration 50 ppb Max %D: 20%

TABLE 9.

Volatile System Performance Check Compounds (SPCC)

Chloromethane 1,1-Dichloroethane Bromoform 1,1,2,2-Tetrachloroethane Chlorobenzene	Minimum Relative Response factor (RRF): 0.300 (0.100 for Bromoform)

TABLE 10.

Target Compound List (TCL) and
Practical Quantitation Limits (PQL)

Volatiles	CAS Number	Quantitation Limits Water µg/L
Vinyl Chloride	75-01-4	10
1,1-Dichloroethene	75-35-4	5
Chloroform	67-66-3	5
1,2-Dichloroethane	107-06-2	5
2-Butanone	78-93-3	10
Carbon Tetrachloride	56-23-5	5
Trichloroethene	79-01-6	5
Benzene	71-43-2	5
Tetrachloroethene	127-18-4	5
Chlorobenzene	108-90-7	5

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TABLE 11.

Standards Preparation

Compound or Standard Mix	Begin Conc. µg/mL	Aliquot µL	Total Vol.	Final Conc. µg/mL	Volume to be used
Restek Internal Standard					
VOA Internal Std. Mix	2,500	200	20 mL	25	10µL/5mL
Restek Surrogate					
VOA Surrogate Spike Mix	2,500	200	20 mL	25	10µL/5mL
Restek Calibration Mix					
VOA Calibration Mix 1	5,000	100	20 mL	25	10µL/5mL
VOA Calibration Mix 2	2,000	250		25	
VOA Calibration Mix 3	2,000	250		25	
VOA Calibration Mix 4	2,000	250		25	
VOA Calibration Mix 5	2,000	250		25	
*Trichlorofluoromethane	5,000	100		25	
*1,3-Dichlorobenzene	5,000	100		25	
*1,4-Dichlorobenzene	5,000	100		25	
*1,2-Dichlorobenzene	5,000	100		25	
*2-Chloroethyl Vinyl ether	10,000	5	2 mL	25	10µL/5mL
Restek VOA TCLP Matrix Spike Mix	2,000	125	5 mL	50	5µL/5mL

*Compound contained in calibration standard, but not required by SW846/8240, PQL Table II.

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TABLE 12.

Response Factor and Concentration Calculation

$$RF = \frac{(A_x)(C_{is})}{(A_{is})(C_x)} \quad \text{and} \quad conc_x = \frac{(A_x)(C_{is})}{(A_{is})(RF_x)}$$

Where:

A_x = Area of characteristic ion for the measured compound.
A_{is} = Area of the characteristic ion for the specified internal standard
C_{is} = Concentration of the internal standard (ng/µL)
C_x = Concentration of the measured compound (ng/µL)
RF_x = Response Factor of the measured compound.

Surrogate Recovery:

$$\% \text{ Surrogate Recovery} = \frac{Qd}{Qa} \times 100\%$$

Where:

Qd = Quantity determined by analysis
Qa = Quantity added to sample

Matrix Spike Recovery and RPD:

$$\text{Matrix Spike Recovery} = \frac{SSR - SR}{SA} \times 100$$

Where:

SSR = Spiked Sample Result
SR = Sample Result
SA = Spike Added

$$\text{Matrix Spike RPD} = \frac{|MSR - MSDR|}{(½)(MSR + MSDR)} \times 100$$

Laboratory Control Spike (LCS) Recovery:

$$\text{LCS Spike Recovery} = \frac{SSR - SR}{SA} \times 100$$

Where:

SSR = Spiked Sample Result
SR = Sample Result
SA = Spike Added

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PROCEDURAL DOCUMENTS - SYSTEM TRANSITION

Please note that the following document is in transition from a previous system. The document identification number (listed below) has been assigned in compliance with the SWL SOP titled:

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The ID given below integrates the following SOP into the new system set forth in GA-101 prior to its annual review (or conversion of format and numbering). Each ID is unique and a "key" field in the new document control database system. The assignment of a new number is therefore necessary for its tracking in the new system.

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Existing ID: IN170

NEW DOCUMENT IDENTIFICATION NUMBER:

SWL-IN-503

Standard Operating Procedure

"TKN (Total KJELDAHL Nitrogen)"
Rev. No. 3.0
Issued: 4/14/97

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SOUTHWEST LABORATORY OF OKLAHOMA, INC.

STANDARD OPERATING PROCEDURE FOR THE DETERMINATION OF TKN (TOTAL KJELDAHL NITROGEN)

REV3.0 – 4/14/97

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Section 1.0 Scope And Application

1.1. This method covers the determination of total Kjeldahl nitrogen in drinking, surface and saline waters, domestic and industrial wastes. The procedure converts nitrogen components of biological origin to ammonia.

1.2 After distillation: the ammonia is determined by the titrimetric method which is applicable to concentrations above 1 mg N/liter.

Section 2.0 Summary Of Method

2.1 The sample is heated in the presence of conc. sulfuric acid, K_2SO_4 and $HgSO_4$ and evaporated until SO_3 fumes are obtained and the solution becomes colorless or pale yellow. The residue is cooled, diluted, and is treated and made alkaline with a hydroxide-thiosulfate solution. The ammonia is distilled and determined after distillation by titration.

Section 3.0 Sample Handling And Preservation

3.1 Samples may be preserved by addition of 2 ml of conc. H_2SO_4 per liter and stored at 4°C. Even when preserved in this manner, conversion of organic nitrogen to ammonia may occur. Preserved samples should be analyzed as soon as possible.

Section 4.0 Interference

4.1 High nitrate concentrations (10X or more than the TKN level) result in low TKN values. The reaction between nitrate and ammonia can be prevented by the use of an anion exchange resin (chloride form) to remove the nitrate prior to the TKN analysis.

Section 5.0 Reference

5.1 *Digestion apparatus:* A Kjeldahl digestion operates with 250 ml Erlenmeyer flasks.

5.2 Hot Plate

5.3 500 ml double-neck boiling flasks with side-arm inlet tubes.

5.4 Distillation rack.

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Section 6.0 Reagents

6.1 Distilled water free of ammonia. Such water is prepared by the passage of distilled water through an ion exchange column containing a strongly acidic cation exchange resin mixed with a strongly basic anion exchange resin.

NOTE 1: All solutions must be made with ammonia-free water.

6.2 Mercuric sulfate solution: Dissolve 8 grams red mercuric oxide (HgO) in 100 ml of 1:5 sulfuric acid (10.0 ml conc. H_2SO_4:50 ml distilled water) or $6NH_2SO_4$.

6.3 Sulfuric acid-mercuric sulfate-potassium sulfate solution: Dissolve 268 grams K_2SO4 in 1300 ml distilled water and 400 ml conc. H_2SO4. Add 50 ml mercuric sulfate solution (see VII.B) and dilute to 2 liters with distilled water.

6.4 Sodium hydroxide-sodium thiosulfate solution: Dissolve 500 grams NaOH and 25 grams $Na_2S_2O_3 \bullet 5\ H_2O$ in distilled water and dilute to 1 liter.

6.5 Mixed indicator: Mix 2 volumes of 0.2% methyl red in 95% ethanol with 1 volume of 0.2% methylene blue in ethanol. Prepare fresh every 30 days.

6.6 Boric acid solution: Dissolve 20 grams boric acid, H_3BO_3 in water, add 10 ml indicator (see VI.F), and dilute to 1 liter with distilled water.

6.7 Sulfuric acid, standard solution: (0.02 N) 1 ml = 0.28 mg NH_3-N. Prepare a stock solution of approximately 0.1 N acid by diluting 3 ml of conc. H_2SO_4 (sp. gr. 1.84) to 1 liter with CO_2-free distilled water. Dilute 200 ml of this solution to 1 liter with CO_2 free distilled water. Standardize the approximately 0.02 N acid so prepared against 0.0200 N Na_2CO_3 solution. This last solution is prepared by dissolving 1.0000 gram anhydrous Na_2CO_3 oven-dried at 140°C, and diluting to 1 liter with CO_2-free distilled water. By weight, 4.08 grams conc. H_2SO_4 diluted to 4000 ml = 0.02 N H_2SO_4.

6.8 Ammonium chloride, stock solution: 1.0 ml = 1.0 mg NH_3-N. Dissolve 3.819 grams NH_4Cl , dried at 100 C, in water and make up to 1 liter in a volumetric flask with distilled water. (Final concentration of 1000 mg/L.)

Section 7.0 Procedure

7.1 The distillation apparatus should be precleaned before use by distilling a 9:1 mixture of distilled water and sodium hydroxide-sodium thiosulfate solution (see VI.D) until the distillate is ammonia-free. This operation should be repeated each time the apparatus is out of service long enough to accumulate ammonia (usually 4 hours or more).

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7.2 *Macro Kjeldahl system:*

7.2.1 Place 100 ml of sample (or an aliquot diluted to 100 ml) into a 250 ml Erlenmeyer flask and add 50 ml sulfuric acid-mercuric sulfate-potassium sulfate solution. Evaporate the mixture in the Kjeldahl apparatus until SO_3 fumes are given off and the solution turns colorless or pale yellow. Cool the residue and add 30 ml distilled water. Transfer to a 500 ml boiling flask and dilute to approximately 300 ml.

7.2.2 For Soils: Weigh 1.00 gram sample; add 50 ml H_2O; add reagent.

7.2.3 Connect the boiling flask to the condenser with an extension of the condenser tip below the level of the boric acid solution in the 500 ml receiving flask.

7.2.4 Make the digestate alkaline by careful addition of 50 ml of sodium hydroxide-thiosulfate solution. Add through the side-arm tube. Mix well.

7.2.5 Steam distill 100 ml to 150 ml at the rate of 6-10 ml/min., into 50 ml of 2% indicating boric acid solution (see VI.F). Begin at "8" setting. After boiling begins, set to "10".

7.3 Determination of ammonia in distillate by titration.

7.3.1 *Titrimetric determination:* Titrate the ammonia with 0.02 N H_2SO_4 (see VI.G), matching the endpoint against a blank containing the same volume of distilled water and H_3BO_3 solution.

Section 8.0 Qa/Qc Requirements

8.1 An ICV will be digested with each batch of samples. The percent recovery limits will be ± 10%.

8.2 A preparation blank(PB), Laboratory Control Sample (LCS), and Laboratory Control Sample Duplicate (LCSD) will be analyzed with every batch of samples. Pipet 2.00 mls of 1000 mg/L NH_4Cl into 100 mls DI water for LCS/LCSD. The percent recovery control limits for the LCS/LCSD are ± 15% and 0-20% RPD. The reporting limit for the PB is < reporting limit.

8.3 A Matrix Spike (MS) and Matrix Spike Duplicate (MSD) will be performed on every batch of 20 samples or less. The control limits and spiking procedure are the same as the LCS/LDS.

Section 9.0 Calculation

9.1 Calculate the Total Kjeldahl Nitrogen, in mg, in the original sample as follows:

TKN (mg) = (A - B)N x F

where:

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A= milliliters of standard 0.020 N H_2SO_4 solution used in titrating sample.

B= milliliters of standard 0.020 N H_2SO_4 solution used in titrating blank.

N= normality of sulfuric acid solution.

F= milliequivalent weight of nitrogen (14 mg).

9.2 To calculate the Total Kjeldahl Nitrogen in mg/l:

TKN (mg/l) = (TKN (mg) x 1000) / V

Where:

V = milliliters of sample digested.

9.3 To calculate the Total Kjeldahl Nitrogen in mg/kg:

TKN (mg/kg) = (TKN (mg) x 1000) / g

Where:

g = grams of sample digested

NOTES:

If sample plus digestion mixture turns black, dilution will probably be necessary. Do not allow digestion to go to dryness or fall below 10 ml before turning pale.

Soils may need to use 0.10 gram of sample if they are oily or black.

Section 10.0 Reference

10.1 EPA 351.3 (modified).

10.2 18th Edition Standard Methods 4500-N_{org} (modified).

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APPENDIX 1

MODIFICATION TO PROCEDURE FOR : URS CONSULTANTS— NAVY CLEAN PROJECT # CTO-0039

Perform a 1:5 soil to water extraction stirring constantly for one hour. Allow extract to settle, and filter through Whatman number one filter. Proceed with method as written. Back calculate results to original soil sample weight (dry weight basis).

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NEW DOCUMENT IDENTIFICATION NUMBER:

SWL-IN-311

Standard Operating Procedure

"Total Organic Carbon (TOC) in Soil/Sediment"
Rev. No. 2.2
Issued: 12/17/96

C2-289

SOUTHWEST LABORATORY OF OKLAHOMA, INC.

STANDARD OPERATING PROCEDURE FOR THE DETERMINATION OF TOTAL ORGANIC CARBON IN SOIL/SEDIMENT

IN045S

REV 2.2 —12/17/96

Document Control # SWL-IN045S-_____

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Signature: _____

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I. SCOPE AND APPLICATION

A. This method describes protocols for the determination of organic carbon in soil or sediment.

B. The applicable range of this analysis is 100 to 999,900 mg/Kg organic carbon. The Southwest Laboratory of Oklahoma reporting limit is 100 mg/Kg.

C. Data is reported in mg/Kg on a dry weight basis.

II. SUMMARY OF METHOD

A. Inorganic carbon from carbonates and bicarbonates is removed by acid treatment.

B. The organic compounds are decomposed by pyrolysis in the presence of oxygen.

C. The carbon dioxide that is formed is determined by direct non-dispersive infrared detection.

D. Water content is determined on a separate portion of sediment.

III. SAMPLE HANDLING AND PRESERVATION

A. Collect sediments in glass jars with teflon or aluminum foil lined lids. Cool and maintain 4°C.

IV. INTERFERENCES

A. Volatile organics in the soil/sediment samples may be lost in the process of air drying samples as well as decarbonation resulting in a low bias.

B. Bacterial decomposition and volatilization of the organic compounds are miinimized by maintaining the sample at 4°C, analyzing within the specified holding time, and analyzing the wet sample.

V. APPARATUS

A. Drying oven maintained at 103° to 105°C.

B. Analytical Instrument: Leco Model CR-12 Carbon determinator with chlorinetrap and non-dispersive infrared detector.

C. Carrier Gas: High Purity Oxygen.

D. Integral Alphanumeric Printer.

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VI. REAGENTS

A. Distilled water used in preparation of standards and for dilution of samples should be ultra pure to reduce the carbon concentration of the blank.

B. Potassium hydrogen phthalate certified A.C.S. grade: 47.05% carbon for instrument calibration.

C. Calcium carbonate certified A.C.S. grade: 12% carbon for instrument calibration check.

D. Hydrochloric acid solution, 1:1 by volume.

VII. PROCEDURE

A. Sample Preparation

1. Take 0.50 grams of well mixed sample and place in a quartz combustion boat.
2. Air dry this sample in desiccator for minimum of 24 hours.
3. Add 1:1 HCl solution dropwise until the effervescence stops.
4. Dry the samples in a drying oven at 75°C. The sample is now ready for analysis.

B. Calibration

1. Instrument run temp = 2500°C, and standby mode = 1500°C
2. Select the channel to be calibrated.
3. Add a combustion boat to the built-in analytical balance.
4. Press tare. Weigh approximately 0.2 grams of potassium hydrogen phthalate in combustion boat.
5. After the balance has stabilized press enter.
6. Press analyze and wait for the digital readout to read "Load Furnace". Slide in calibration standard and wait for the result to print-out.
7. Repeat steps 2 through 5 a minimum of three more times.
8. Press the SYSTEM UPDATE key, then the 1 key. The message center will display: CALIBRATE SYSTEM YES/NO
9. Press the YES key. The message will display: CALIBRATE, BY STD YES/NO
10. Press the YES key. The printer will print the answer stack for the previous 10 runs.
11. The message center will display: CARBON STD - % ??? MOD BY KBD enter the percent carbon contained in the standard sample (47.05%).
12. Enter the percent carbon obtained in the standard sample (47.05%).
13. The printer will print the standard entered and the old calibration.

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14. The message will display: SELECT FROM LAST xx RESULTS

15. Enter the total number of calibration runs to be included in the calibration calculation. After a number is entered, the message center will display one-at-a-time the results for the number of results entered. Answer YES or NO to each prompt as it appears. The printer will print each answer as the YES key is pressed.

16. After the operator responds to the last prompt, the printer will print the new calibration and the answer stack, recalculated according to the new calibration value.

17. Analyze the 12% calcium carbonate standard and a duplicate to verify the calibration. The percent recovery must be within ± 10% and RPD <20 or recalibration will be necessary.

18. Analyze a blank combustion boat. The result must be below 0.01% or recalibration will be necessary.

19. The 12% calcium carbonate standard and blank is analyzed after every 15 samples and must meet the above criteria or recalibration and reanalysis of the previous 15 samples will be necessary.

C. Sample Analysis

1. Press the ID CODE key. Enter the sample number to be analyzed.

2. Enter the weight used in the sample preparation and press enter.

3. Press the ANALYZE key. The PURGE flow will start immediately. The LANCE flow will start later in the analysis cycle.

4. Wait until the message center display "LOAD FURNACE" and then slide the combustion boat. Immediately after inserting the combustion boat, press the ANALYZE key again since the amount of carbon released will be insufficient to start analysis automatically.

5. When the analysis is complete, the CARBON display will indicate the results and the printer will print the value.

6. Remove the expended combustion boat from the furnace.

VIII. QUALITY ASSURANCE/QUALITY CONTROL

A. With each batch of samples prepared an empty combustion boat will be treated with 1:1 HCl and carried through the drying procedure with the samples. The result of the method blank must be below 0.01%. If all results are below the Reporting Detection Limit (RDL) or above 10x the blank value the results may be used. If not, the samples must be reprepared and analyzed.

B. Due to the nature of the analysis and the high levels of organic carbon found in soils an sediments

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a matrix spike is not required to establish method accuracy. Accuracy of the analysis is determined by two sources. The calcium carbonate standard used for the initial calibration verification and continuing calibration verification was discussed in Section VII. Accuracy will also be determined using a privately prepared sample of Chatanooga Shal containing 3.6% total organic carbon. The recovery requirements for this sample will be ±15%. If the recovery is not within control limits, the sample will be reanalyzed once more. If the recovery is still not within control limits the instrument will be recalibrated and analysis restarted.

C. As stated in Section VII. B. a continuing calibration standard and blank will be analyzed every fifteen samples.

D. A matrix spike and matrix spike duplicate must be analyzed with every batch of ≤ 20 samples. Add 1 gram of Potassium Hydrogen Phthalate (Sec. VI.b.) after step A.4 in section VII.

IX. DATA REDUCTION

A. The concentration printed out in % organic carbon shall be converted to mg/Kg organic carbon by the following formula:

Total organic carbon, mg/Kg:

% Total Organic Carbon (air dried) x 10,000

X. DATA REPORTING REQUIREMENTS

A. Total organic carbon results will be reported in a tabular format in mg/Kg dry weight.

B. The results of the method blank, initial and continuing calibration, and duplicate analysis associated with each batch will also be reported in a tabular format.

XI. REFERENCES

A. LECO Corp., CR-12 Carbon System Instruction Manual, 1987

B., SW-846, Method 9060, Rev. 2, June, 1990

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PROCEDURAL DOCUMENTS - SYSTEM TRANSITION

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SWL-IN-310

Standard Operating Procedure

"Total Organic Carbon (TOC) in Water"
Rev. No. 2.4
Issued: 3/10/97

C2-296

SOUTHWEST LABORATORY OF OKLAHOMA, INC.

STANDARD OPERATING PROCEDURE FOR THE DETERMINATION OF TOTAL ORGANIC CARBON IN WATER

IN045W
REV 2.4 – 3/10/97

Document Control # SWL-IN045W

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Section 1.0 ↳ Scope And Application

1.1 This method includes the measurement of organic carbon in drinking, surface, and saline waters, domestic and industrial wastes.

Section 2.0 ↳ Summary Of Method

2.1 Organic carbon in a sample is converted to carbon dioxide (CO_2) by catalytic combustion. The CO_2 formed is measured by a nondispersive infrared gas analyzer (NDIR).

Section 3.0 ↳ Sample Handling And Preservation

3.1 Sampling and storage of samples in glass bottles is preferable. Sampling and storage in plastic bottles is permissable if it is established that the containers do not contribute contaminating organics to the samples.

3.2 Samples should be acidified with HC1 or H_2SO_4 to a pH <2. Store samples at 4^0 C until time of analysis.

3.3 Holding times are 28 days from sample collection.

Section 4.0 ↳ Interferences

4.1 This procedure applicable only to homogenous samples which can be injected into the apparatus by reproducible means of a microliter-type syringe. The openings of the syringe limit the maximum size of particle which may be included in the sample.

4.2 Removal of carbonate and bicarbonate by acidification and purging with nitrogen can result in the loss of volatile organic substances.

Section 5.0 ↳ Apparatus

5.1 Shimadzu Total Organic Carbon Analyzer, model TOC-5050 or equivalent.

5.2 Carrier Gas: High Purity Air not to contain more than 1 ppm CO_2, CO, and HC (hydrocarbon) respectively.

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Section 6.0 ↳ Reagents

6.1 *Distilled water* used in preparation of satandards and for dilution of samples should be ultra pure to reduce the carbon concentration of the blank.

6.2 *Potassium hydrogen phthalate, stock solution, 1000 mg carbon/liter* ↳ Dissolve 2.125g of potassium hydrogen phthalate (Primary Standard Grade) in distilled water and dilute to one liter. The obtained solution contains 1,000 mg/l of carbon (1,000 C/1) or equivalent to 1,000 ppm C.

6.3 *IC Reagent* ↳ 25 ml phosphoric acid, 75 ml DI water.

6.4 *Humidifier* ↳ Fill humidifier with distilled water to the upper line, add 2 pellets of NaOH, and mix.

6.5 *Working Calibration Standards* ↳ Using the following table, dilute the standards using organic-free water.

mLs of Stock Solution (6.2) per 200 mL Final Volume	Conc. of Standard mg/l
0.4	2.0
4.0	20.0
10.0	50.0
20.0	100.0

6.6 *Blank Solution* ↳ Use the same distilled water used for the preparation of the standard solution.

Section 7.0 ↳ Calibration

7.1 Summary

7.1.1 Lower the pH of the standards to <2 with H_2SO_4.

7.1.2 Load the standards into the autosampler and purge with oxygen for 5 minutes.

7.1.3 Calibrate instrument starting with high standard and working down towards low standard.

7.1.4 Resulting calibration curve must have a correlation coefficient ≥ 0.995.

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7.2 Calibration Frequency:

7.2.1 A new calibration must be performed on a monthly basis, or if one of the following action items exist (whichever is more frequent).

a) When a new Stock standard solution is made.

b) When any of the instrument parameter's are changed (combustion, tube, carrier gasa flow rate, injection needle, or anything that might change the sensitivity of the instrument).

c) If the Independent Calibration Standard (ICV), Continuing Calibration Standard (CCV), Independent Calibration Blank (ICB), or Continuing Blank (CCB) fails their respective control limits.

7.3 Procedure

7.3.1 Remove gas-line from probe.

7.3.2 Bring up Main Menu; Press #8 (Maintenance menu). Arrow down to gas-flow (Press enter.) This will turn the gas on. Open front cover of the instrument and adjust the gas-flow all the way open.

7.3.3 Place gas-line in standard container and sparge for five minutes. Do all standards, cleaning gas-line between each standard.

7.3.4 Fill standard vials and place in turntable starting with high standard. The setup will be as follows.

CALIBRATION ONE

100.0.....S1

50.0.....S2

20.0.....S3

2.0.....S4

7.3.5 Check baseline by selecting #6 (Monitor) and correct if necessary. Press F2 to return to the Main Menu.

7.3.6 Next select #9 (Autosampler) and press enter.

7.3.6.1 Press F3 (Edit)

7.3.6.2 Press F1 (All Clear)

7.3.6.3 Press F2 (All Clear)

7.3.7 Arrow over to C1 (Calibration One.) Here you may enter any number one through eighteen and press enter. The calibration will be stored in that file number.

7.3.7.1 Type will be set as #1 (TC). Press enter.

7.3.7.2 The first standard concentration will be 50.0; press enter. Next enter where the standard will be placed. See VII.B.4 above for placement (i.e., the first standard will be S1.)

7.3.7.3 Range will be set at x5; press enter.

7.3.7.4 Injection volume will be 30 ug/l; press enter.

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7.3.7.5 Number of injections will be 3; press enter.

7.3.7.6 Max. number of injections will be set at 5; press enter.

7.3.7.7 SD and CV will be set at 200 and 2.0%.

7.3.7.8 Shift to origin should be OFF.

7.3.8 Press F2 (Return) to return to autosampler screen.

7.3.9 Press F1 (Next).

7.3.10 ASI Conditions:

7.3.10.1 Rinse 1

7.3.10.2 No. of Needle Washes 2

7.3.10.3 Flow-line washes 4

7.3.10.4 Calibrate before 2

7.3.10.5 Print 3

7.3.10.6 Screen will skip down to:

Key Lock 2

7.3.11 Press F1 (Next).

7.3.12 Press Start.

Section 8.0 Sample Analysis

8.1 Summary

8.1.1 Load samples onto autosampler.

8.1.2 Set purge time at 2.0 minutes using high purity air as purge gas.

8.1.3 Analyze the samples using triple injection.

8.2 Press #8 (Maintenance); press enter

8.2.1 Arrow down to mechanical check; press enter.

8.2.2 Press ASI.

8.2.3 Press AT-R.

8.2.3.1 Remove probe wash and clean with soap and water. Rinse with 1:1 HCL. Fill with DI water and replace; press return; press end.

8.2.4 Remove sample and gas-line from probes.

8.2.5 Remove gas and sample probe from arm.

8.2.5.1 Place both needles in a very mild HCL solution (1:9) and place in sonicator for fifteen minutes.

8.2.5.2 Remove from sonicator and rinse.

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8.2.5.3 Fill container with DI water and sonicate for an additional fifteen minutes. Replace in arm.

8.3 Regeneration of IC

8.3.1 Press enter once regeneration is complete (refill if necessary.)

8.3.1.1 Arrow down to mechanical check; press enter.

8.3.1.2 Under IC-D press on and let any excess reagent drain.

8.3.1.3 While on move, drain tube up or down to prevent tube being crimped or broken. Press off; then press end.

8.3.2 Arrow up to regeneration of TC.

8.3.2.1 Place 2N HCL in S1 and press start.

8.3.2.2 Remove top lid off of TOC 5050.

8.3.2.3 Watch injection to make sure injection is centered and does not splash on sides of combustion tube. If injection is not correct, replace injection needle (see Schimazdu TOC 5050 Operating Manual, page 123.)

8.3.3 Connect gas and sample lines to proper probe.

8.3.3.1 Arrow down to sparge gas flow and press enter. This turns the gas on.

8.3.3.2 Open front cover; set sparge gas to 40 ml/min using flowmeter.

8.3.3.3 Set carrier gas at 150 ml/min.

8.3.3.4 Arrow up to sparge gas-flow; press enter (this turns off the gas.)

8.3.3.5 Press F2 to return to Main Menu

8.4 Baseline check

8.4.1 Press #6; press enter.

8.4.2 If baseline is zero, press F2. If not, adjust baseline using the optical zero on top of the instrument.

8.4.3 Press F2 to return to Main Menu.

8.5 Analysis

8.5.1 Press #9 (Autosampler); press enter.

8.5.2 Press F3 (Edit.)

8.5.3 Press F1 (All Clear.)

8.5.4 Press F2 (All Clear.)

8.5.5 Choose type of measurement: 1=TC, 2=IC, 3=TOC, 4=NPOC. Press enter.

8.5.6 IS is the initial sample. Most often it will be number one, but it can be two, three, and so on.

8.5.7 FS is final sample and is always a CCB (each run ends with a CCV and a CCB.)

8.5.8 Arrow over to F1 and choose F1 or F2. F1 is calibration one; F2 is the second calibration used for samples that are well above calibration one. At this point #2 is

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entered. When #2 is entered, instrument will automatically move to W (which is number of washes.) Enter four. NO is the number of injections; in most cases three will be used. max is maximum number of injections; in most cases five will be used. If for instance, quads are required it is best to run four out of six. Arrow over to SP where sparge gas is set for 2.0 minutes per sample. Dl will remain as is.

8.5.9 ASI Conditions

8.5.9.1 Rinse 1

8.5.9.2 Number of needle washes 2

8.5.9.3 Flow-line washes 4

8.5.9.4 Calibrate before 2

8.5.9.5 Print Info 3

8.5.9.6 Auto add of acid 1

8.5.9.7 Acid volume ..... (auto)

8.5.9.8 Rinse after addition of acid 1

8.5.9.9 Key Lock 2

8.5.9.10 Finish or running 3

8.5.10 Press start.

Section 9.0 Quality Control

9.1 All quality control data should be maintained and available for easy reference or inspection.

9.2 Calibration: See Section 7.0.

9.3 Initial Demonstration of Performance:

1) Linear Calibration Range(LCR) -- The LCR must be determined initially and verified every 6 months. The verification on linearity must use a minimum of a blank and three standards. If any verification data exceeds the initial values by +/- 10%, linearity must be re-established. If any portion of the range is shown to be nonlinear, sufficient standards must be used to clearly define the nonlinear portion.

2) Method Detection Limit (MDL) -- MDL's must be determined, according to the SWL-GENMDLS SOP (most current version), every six (6) months.

9.4 Assessing Laboratory Performance:

1) A method blank must be distilled with every batch of 20 samples or less. If the value exceeds the reporting limit, laboratory or reagent contamination should be suspected and the samples re-distilled.

2) Laboratory Control Samples (LCW and LDW) will be distilled with every batch. With a true value of 25.0 mg/l the LCW and LDW have control limit of 90-110% recovery. If out of control the samples will be re-distilled.

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3) Independent Calibration Verification (ICV) and Continuing Calibration Verification (CCV): this must be a mid-range check standard from an independent source from the calibration standards. It will be ran immediately after calibration, a frequency of 10% and at the end of run. Control limits are 90-110% recovery, if failure occurs the system must be re-established and re-analysis of all samples that are not surrounded by CCV's that are in control.

4) Independent Calibration Blank (ICB) and Continuing Calibration Blank (CCB): A blank must be ran immediately after each ICB or CCV. The value must be less than the reporting limit, if failure occurs the system must be re-calibrated and re-analyzed of all samples that are not surrounded by CCB's that are in control.

9.5 Assessing Analyte Recovery and Data Quality

1) A Matrix Spike (MS) and (MSD) must be performed with every batch at value of 25.0 mg/l level. Data will be flagged if the control of 90-110% is not met.

Section 10 References

10.1 U.S. EPA "Methods for Chemical Analysis of Water and Wastes", EPA 600/9-91/034, September, 1991. Method 415.1.

10.2 Instruction Manual, Total Organic Carbon Analyzer Model TOC-5050.

10.3 U.S. EPA SW846 Method 9060, Revision 1, November, 1990.

10.4 Standards Methods, 18th Edition, Method 5310B.

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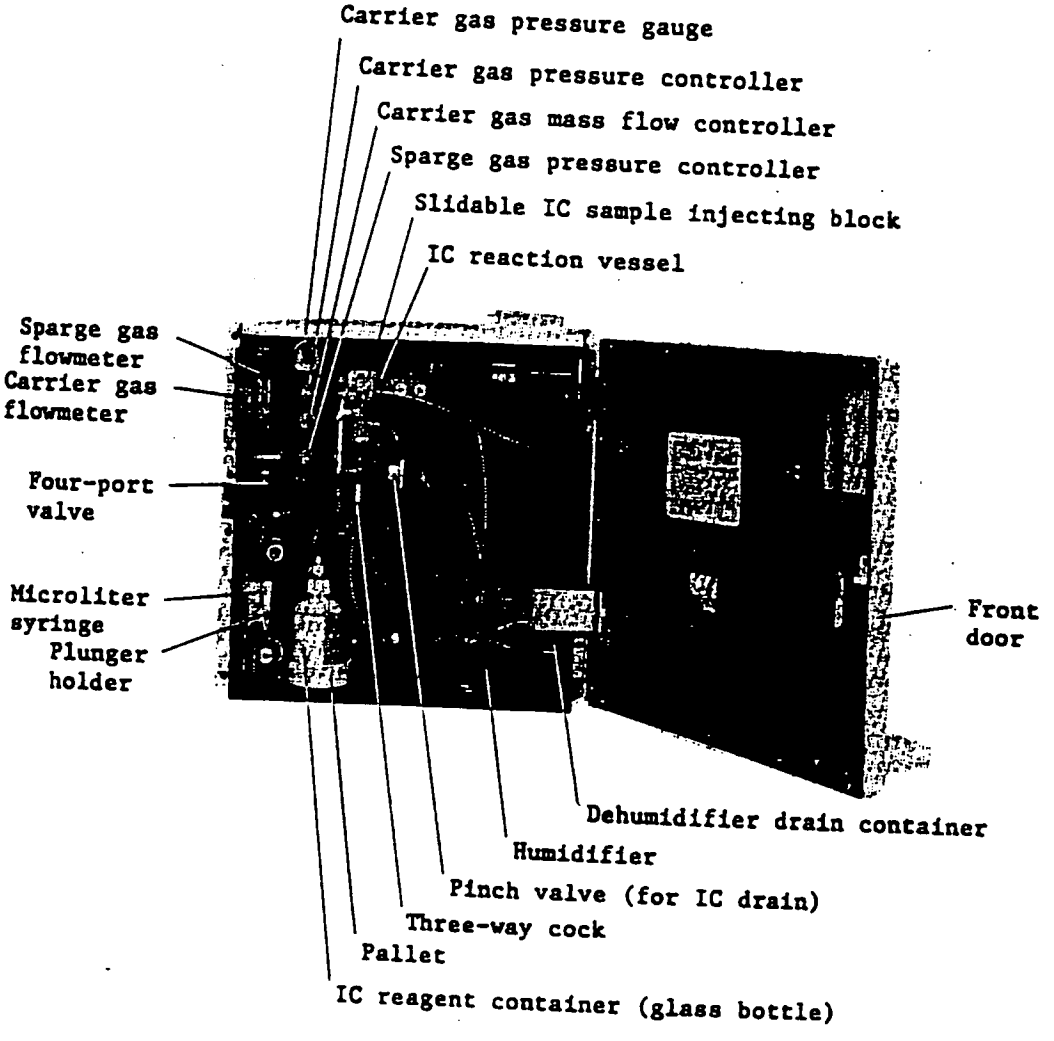
FIGURE 1.
FRONT VIEW OF THE TOC-5050 INTERIOR WITH FRONT DOOR OPEN



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SOUTHWEST LABORATORY OF OKLAHOMA, INC.

TM

Standard Operating Procedure

Ammonia as Nitrogen (Titrimetric with Distillation)

Document No.: SWL-IN-504

Rev No. / Date: Rev. 2.0 — 08/14/97

APPROVALS	DATES
Steve L. Mauldin Procedure Prepared By	08/14/97 Date
[illegible] Program Manager or Section Supervisor	8/15/97 Date
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(Effective Date is 5 calendar days after the last signature above - QA/QC Officer)

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AMMONIA AS NITROGEN (TITRIMETRIC WITH DISTILLATION)

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1.0 SCOPE AND APPLICATION

1.1 This method covers the determination of Ammonia as Nitrogen in drinking, surface, and saline waters, soils, sediments, and sludges.

1.2 After distillation, the Ammonia is determined by the titrimetric method which is applicable to concentrations above 1 mgN/L (waters) and 10 mgN/Kg (soils).

2.0 METHOD SUMMARY

— This method is based on the distillation of ammonia in a borate buffer at pH 9.5 and subsequent reaction of the distillate with indicating boric acid solution, which is then titrated with sulfuric acid.

3.0 HEALTH AND SAFETY

3.1 Refer to the current version of the "Laboratory Safety Plan" SOP.

3.2 Each chemical and sample should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. This may be done by wearing gloves, eye protection, and laboratory coat during distillation and analysis.

3.3 The following chemicals have the potential to be highly toxic or hazardous, consult MSDS: Sulfuric Acid, Sodium Hydroxide.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING & STORAGE

4.1 Samples may be preserved by H_2SO_4 at pH between 1.5 and 2 and stored at 4°C.

4.2 Samples should be collected in plastic or glass bottles. All containers must be thoroughly cleared and rinsed with reagent water.

5.0 INTERFERENCES

— Volatile alkaline compounds such as hydrazine and amines will influence titrimetric results.

6.0 APPARATUS

6.1 Distillation Apparatus

6.1.1 500 mL borosilicate boiling flask

6.1.2 Vertical condenser

6.1.3 500 mL receiving beakers

6.1.4 Boiling flask heating mantel

6.1.5 pH paper

6.2 Titration apparatus

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6.2.1 50 mL Class A T-buret with Teflon stopcock

6.2.2 Buret support

7.0 REAGENTS

7.1 ASTM Type II Water (ASTMD 1193): Water should be monitored to be Ammonia free.

7.2 Sulfuric Acid, H_2SO_4: Concentrated, Tracepur.

7.3 Borate buffer solution: Add 88 mL 0.1 N NaOH solution to 500 mL approximately 0.025 M sodium tetraborate ($Na_2B_4O_7$) solution (9.5 g $Na_2B_4O_7 \cdot 10\ H_2O$/L) and dilute to 1 liter.

7.4 Sodium Hydroxide, 6N: Dissolve 240 g NaOH in beaker and dilute to 1 L.

7.5 Mixed indicator solution: Mix 2 volumes of 0.2% methylene in 95% ethanol with 1 volume of 0.2% methylene blue in ethanol.

7.6 Indicating boric acid solution: Dissolve 20 g H_3BO_3 in water. Add 10 mL mixed indicator solution and dilute to 1 L.

7.7 Standard sulfuric acid titrant, 0.02N: Standardize with Na_2CO_3.

8.0 PROCEDURE

8.1 Distillation

8.1.1 The distillation apparatus should be precleaned before use to make sure it is Ammonia free.

8.1.2 Place 100 mL (water) or 10 g (soils) of sample into distillation boiling flask with 20 mL of borate buffer solution and 100 mL of distilled H_2O. Adjust pH to 9.5 with NaOH solution. Attach to condenser.

8.1.3 Put 50 mL of indicating boric acid solution into receiving beaker, and put tip of the delivery tube below the surface of acid solution.

8.1.4 Distill at a rate of 6-10 mL/min. until about 100 mL of distillate has been collected.

8.1.5 Remove delivery tube from collection beaker and titrate.

8.2 Titration

8.2.1 Titrate Ammonia in distillate with standard 0.02 N H_2SO_4. Titrate until indicator turns a pale lavender.

8.2.2 Carry a blank through all steps of the procedure and apply the necessary correction to the results.

9.0 CALCULATION

9.1 Liquid Samples

$$\text{mg } NH_3 - N/L = \frac{(A-B) \times 280}{\text{mL sample}}$$

9.2 Soil Samples

$$\text{mg } NH_3 - N/Kg = \frac{(A-B) \times 280}{\text{g weight sample}}$$

where:

A = Volume of H_2SO_4 titrated for sample (mL)
B = Volume of H_2SO_4 titrated for blank (mL)

10.0 QUALITY ASSURANCE/QUALITY CONTROL

10.1 Ensure all quality control documentation is readily available.

10.2 Analyze at least one method blank carried through the whole procedure (PBW).

10.3 Analyze a laboratory control sample (LCW) and laboratory control sample duplicate (LDW) with each batch of samples.

10.4 At the client's request a matrix spike (MS) and matrix spike duplicate (MSD) will be analyzed with each batch of samples.

11.0 **WASTE DISPOSAL** — All samples and chemicals will be disposed of as per instruction in SOP SWL-GA-114 "Hazardous Waste Management Plan".

12.0 METHOD PERFORMANCE

12.1 The result of the method blank must be below the reporting limit.

12.2 The control limit of the laboratory control sample is 20% RPD and 80-120% recovery.

13.0 REFERENCES

13.1 Standard Methods for the Examination of Water and Wastewater, 19th Edition, APHA-AWWA-WPCF, Method 4500-NH_3-B &E.

13.2 U.S. Environmental Protection Agency, Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-010, March 1983, Method 350.2.

14.0 DEFINITIONS

14.1 Material Safety Data Sheet (MSDS): ..... Information provided by vendors for the chemical products prepared by their company. The information on the MSDS includes but is not limited to composition, physical properties, hazards identification, first aid measures, emergency clean-up and/or response, exposure and storage.

14.2 Sample Batch: ..... Consists of a maximum of twenty samples per matrix.

14.3 Method Blank: ..... A distilled water sample carried through the entire preparation and analysis procedure to check for contamination.

14.4 Laboratory Control Sample: ..... A spiked sample taken through distillation and analysis to check the methods accuracy.

14.5 Matrix Spike (MS): ..... A spiked sample taken through the distillation and analysis to check the effect of the matrix on the accuracy of the method.

14.6 Matrix Spike Duplicate (MSD): ..... A spiked sample taken through the distillation and analysis to check the effect of the matrix on the precision and accuracy of the method.

Request for New or Revised Procedure, SWL-GA-101

SWL SOP Rev. # 1.0 - 05/14/97; Form ID: GA101-DOCREQ-F: Location/File ID: F:\SOP\GA\101-ATT5

REVISED (includes Forms/Logs) Submitted By: SRM Date: 08/12/97

Document Title: Ammonia -Titrimetric w/Distillation

Document No.: SWL-I A-504 Rev. No. 1.1 — 07125196

Reference: Page ____ of ____ Section: ___.___; Subpart: _____

Attachment No. _____ — Title: _____

Brief Description (need for revision): Annual Review. Need to put into new Format. Rev. 2.0.

Approved: Steve L. Markho Dated: 08/12/97 Denied: ☐ PM/SS Initials: _____
Program Manager/Section Supervisor (see attached explanation)

NEW (Procedural Document) Submitted By: _____ Date: _____

Justification for New Procedure: _____

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Program Manager/Section Supervisor (see attached explanation)

Author: _____ Assigned By: _____ Date: _____

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SOUTHWEST LABORATORY OF OKLAHOMA, INC.

STANDARD OPERATING PROCEDURE FOR THE DETERMINATION OF AMMONIA AS NITROGEN (TITRIMETRIC WITH DISTILLATION)

IN020

REV 1.1 — 7/25/96

Document Control #SWL-IN020-_____

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I. Scope and Application

A. This method covers the determination of Ammonia as Nitrogen in drinking, surface, and saline waters, soils, sediments, and sludges.

B. After distillation, the Ammonia is determined by the titrimetric method which is applicable to concentrations above 1 mg N/L (waters) and 10 mg N/kg (soils).

II. Summary of Method

A. This method is based on the distillation of Ammonia in a borate buffer at pH 9.5 and subsequent reaction of the distillate with indicating boric acid solution, which is then titrated with sulfuric acid.

III. Interferences

A. Volatile alkaline compounds such as hydrazine and amines will influence titrimetric results.

IV. Sample Handling & Preservation

A. Samples may be preserved by H_2SO_4 at pH between 1.5 and 2 and stored at 4°C.

V. Apparatus

A. Distillation Apparatus

1. 500 mL borosilicate boiling flask
2. Vertical condenser
3. 500 mL receiving beakers
4. Boiling flask heating mantel
5. pH paper

B. Titration apparatus

1. 50 mL Class A T-buret with Teflon stopcock
2. Buret support

VI. Reagents

A. ASTM Type II Water (ASTMD 1193): Water should be monitored to be Ammonia free.

B. Sulfuric Acid, H_2SO_4: Concentrated, Tracepur.

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C. Borate buffer solution: Add 88 mL 0.1 N NaOH solution to 500 mL approximately 0.025 m sodium tetraborate ($Na_2B_4O_7$) solution (9.5 g $Na_2B_4O_7 \cdot 10\ H_2O$ /L) and dilute to 1 liter.

D. Sodium Hydroxide, 6N: Dissolve 240 g NaOH in beaker and dilute to 1 L.

E. Mixed indicator solution: Mix 2 volumes of 0.2% methylene in 95% ethanol with 1 volumes of 0.2% methylene blue in ethanol.

F. Indicating boric acid solution: Dissolve 20 g H_3BO_3 in water. Add 10 mL mixed indicator solution and dilute to 1 L.

G. Standard sulfuric acid titrant, 0.02N: Standardize with Na_2CO_3.

VII. Procedure

A. Distillation

1. The distillation apparatus should be precleaned before use to make sure it is Ammonia free.
2. Place 100 ml (water) or 10 g (soils) of sample into distillation boiling flask with 20 ml of borate buffer solution and 100 ml of distilled H_2O. Adjust pH to 9.5 with NaOH solution. Attach to condenser.
3. Put 50 mL of indicating boric acid solution into receiving beaker, and put tip of the delivery tube below the surface of acid solution.
4. Distill at a rate of 6-10 ml/min. until about 100 ml of distillate has been collected.
5. Remove delivery tube from collection beaker and titrate.

B. Titration

1. Titrate Ammonia in distillate with standard 0.02 N H_2SO_4. Titrate until indicator turns a pale lavender.
2. Carry a blank through all steps of the procedure and apply the necessary correction to the results.

VIII. Calculation

A. Liquid Samples

$$\text{mg } NH_3\text{ - N/L} = \frac{(A-B) \times 280}{\text{mL sample}}$$

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B. Soil Samples

$$\text{mg } NH_3\text{ - N/Kg} = \frac{(A-B) \times 280}{\text{g weight sample}}$$

where:

A = Volume of H_2SO_4 titrated for sample (mL)

B = Volume of H_2SO_4 titrated for blank (mL)

XI. Quality Control

A. Ensure all quality control documentation is readily available.

B. Analyze at least one method blank carried through the whole procedure (PBW). The result for the PBW must be below the reporting limit.

C. Analyze a laboratory control sample (LCW) and laboratory control sample duplicate (LDW) with each batch of samples. The control limit for the LCW-LDW is 20 % RPD and 80-120% recovery.

D. At the client request a matrix spike (MS) and matrix spike duplicate MSD) will be analyzed with each batch of samples.

X. References

A. Standard Methods for the Examination of Water and Wastewater, 16th Edition, APHA-AWWA-WPCF, Method 417A and 417D.

B. U.S. Environmental Protection Agency, Methods for Chemical Analysis of Water and Wastes, CPA-600/4-79-010, March 1983, Method 350.2.

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OFFICIAL COPY RED STAMP INDICATES ORIGINAL

SOUTHWEST LABORATORY OF OKLAHOMA, INC.

STANDARD OPERATING PROCEDURE FOR THE DETERMINATION OF INORGANIC ANIONS IN WATER & SOIL BY ION CHROMATOGRAPHY (DIONEX 2010I)

IN030, IN055, IN135, IN185, IN195, IN255, IN295

REV 2.4 — 10/30/96

Document Control #SWL-IN030B-006

CONTROLLED DOCUMENT: DO *NOT* COPY!

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I. Scope and Application

A. This method covers the determination of the following inorganic anions; Bromide, Chloride, Nitrate-N, Nitrite-N, Ortho-Phosphate-P, Sulfate, and Fluoride.

B. This method is applicable to the determination of the above anions in drinking water, surface water, mixed domestic and industrial wastewaters, groundwater, reagent waters, and solids (after extraction).

C. This method is recommended for use only by or under the supervision of analysts experienced in the use of ion chromatography and in the interpretation of the resulting chromatogram. Each analyst must demonstrate the ability to generate acceptable results with this method.

D. When this method is used to analyze unfamiliar samples for any of the above anions, anion identification should be supported by the use of sample matrix spikes covering the anions of interest.

II. Summary of Method

A. A 1.0 millileter volume of sample is introduced into the ion chromatograph. The anions of interest are separated and measured, using a system comprised of a guard column, separator column, suppressor device, and conductivity detector.

B. In order to use this method for solids, an extraction procedure must be performed.

III. Interferences

A. Interferences can be caused by substances with retention times that are similar to and overlap those of the anion of interest. Large amounts of an anion can interfere with the peak resolution of an adjacent anion. Sample dilution and/or spiking can be used to solve most interference problems.

B. Method interferences may be caused by contaminants in the reagent water, reagents, glassware, and other sample processing apparatus that lead to discrete artifacts or elevated baseline in ion chromatograms.

C. Samples that contain particles larger than 0.45 microns and reagent solutions that contain particles larger than 0.20 microns require filtration to prevent damage to instrument columns and flow systems.

IV. Safety

A. Normal, accepted laboratory safety practices should be followed during reagent preparation and instrument operation. No known carcinogenic materials are used in this method.

1. Safety glasses must be worn at all times within the laboratory.
2. Gloves should be worn during the filtration of the samples.

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V. Apparatus and Materials

A. Balance - Mettler/H80, capable of accurately weighting to the nearest 0.0001 g.

B. Ion Chromatograph - Dionex Model 2010i, with required accessories including syringes, analytical columns, compressed gases, and detectors.

C. Anion guard column - Dionex Ion Pac AG4A-SC

D. Anion separator column - Dionex Ion Pac AS4A-SC

E. Anion suppressor device - Dionex Anion Micro-Membrane Suppressor (P/N 43074).

F. Detector - Dionex Conductivity Cell

G. VG Chromatography server and mini-chrom data system.

VI. Reagents and Consumable Materials

A. Reagent water - Deionized water, free of the anions of interest. The water must be filtered through a 0.20 micron filter.

B. Eluent Solution - Sodium bicarbonate 1.7/ mM, sodium carbonate 1.8 mM. Dissolve 0.2856 g sodium bicarbonate ($NaHC0_3$) and 0.3816 g of sodium carbonate (Na_2C0_3) in reagent water and dilute to 2 liters.

C. Regeneration solution - Sulfuric acid, 0.025 N. Dilute 2.8 ml conc. sulfuric acid (H_2S0_4) to 4 liters with reagent water.

D. Stock Standard solutions, 10,000 mg/l - Certified stock standards for all anions are purchased from SPEX Industries, Inc. (or another NIST-certified manufacturer), and are the basis for all working standards.

E. Intermediate Standard - Dilute the 10000 ppm stock standards to 500 ml according to the following schedule: 2.5 ml Br^- (50 ppm), 2.5 ml Cl^- (50ppm), 12.5 mls $N0_3^-$ (25 ppm), 1.0 ml $N0_2^-$ (20 ppm), 1.0 mls $P0_4^-$ (20 ppm), 1 ml F^- (20 ppm), and 5.0 ml $S0_4^-$ (100 ppm).

F. Working Standards (Method 2010i):

1. Number 1 Standard - Dilute 125 ml of the Intermediate Standard (VI.E) to 250 ml in reagent water to obtain the following concentrations: 25 ppm Br^-, 25 ppm Cl^-, 12.5 ppm $N0_3^-$, 10 ppm $N0_2^-$, 10 ppm $P0_4^-$, 10 ppm F^-, and 50 ppm $S0_4^-$.

2. Number 2 Standard - Dilute 25 ml of the Intermediate Standard (VI.E) to 250 ml in reagent water to obtain the following concentrations: 5 ppm Br^-, 5 ppm Cl^-, 2.5 ppm $N0_3^-$, 2 ppm $N0_2^-$, 2 ppm $P0_4^-$, 2 ppm F^-, and 10 ppm $S0_4^-$.

3. Number 3 Standard - Dilute 5 ml of the Intermediate Standard (VI.E) to 100 ml in reagent water to obtain the following concentrations: 2.5 ppm Br^-, 2.5ppm Cl^-, 1.25 ppm $N0_3^-$, 1.0 ppm $N0_2^-$, 1.0 ppm $P0_4^-$, 1.0 ppm F^-, and 5.0 ppm $S0_4^-$.

4. Number 4 Standard - Dilute 2 ml of the Intermediate Standard (VI.E) to 100 ml in reagent water to obtain the following concentrations: 1.0 ppm Br^-, 1.0 ppm Cl^-, 0.5 ppm $N0_3^-$, 0.4 ppm $N0_2^-$, 0.4 ppm $P0_4^-$, 0.4 ppm F^-, and 2.0 ppm $S0_4^-$.

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V. Apparatus and Materials

A. Balance - Mettler/H80, capable of accurately weighting to the nearest 0.0001 g.

B. Ion Chromatograph - Dionex Model 2010i, with required accessories including syringes, analytical columns, compressed gases, and detectors.

C. Anion guard column - Dionex Ion Pac AG4A-SC

D. Anion separator column - Dionex Ion Pac AS4A-SC

E. Anion suppressor device - Dionex Anion Micro-Membrane Suppressor (P/N 43074).

F. Detector - Dionex Conductivity Cell

G. VG Chromatography server and mini-chrom data system.

VI. Reagents and Consumable Materials

A. Reagent water - Deionized water, free of the anions of interest. The water must be filtered through a 0.20 micron filter.

B. Eluent Solution - Sodium bicarbonate 1.7/ mM, sodium carbonate 1.8 mM. Dissolve 0.2856 g sodium bicarbonate ($NaHCO_3$) and 0.3816 g of sodium carbonate (Na_2CO_3) in reagent water and dilute to 2 liters.

C. Regeneration solution - Sulfuric acid, 0.025 N. Dilute 2.8 ml conc. sulfuric acid (H_2SO_4) to 4 liters with reagent water.

D. Stock Standard solutions, 10,000 mg/l - Certified stock standards for all anions are purchased from SPEX Industries, Inc. (or another NIST-certified manufacturer), and are the basis for all working standards.

E. Intermediate Standard - Dilute the 10000 ppm stock standards to 1000 ml according to the following schedule: 2.5 ml Br^- (50 ppm), 2.5 ml Cl^- (50ppm), 12.5 mls NO_3^- (25 ppm), 1.0 ml NO_2^- (20 ppm), 1.0 mls PO_4^- (20 ppm), 1 ml F^- (20 ppm), and 5.0 ml SO_4^- (100 ppm).

F. Working Standards (Method 2010i):

1. Number 1 Standard - Dilute 125 ml of the Intermediate Standard (VI.E) to 250 ml in reagent water to obtain the following concentrations: 25 ppm Br^-, 25 ppm Cl^-, 12.5 ppm NO_3^-, 10 ppm NO_2^-, 10 ppm PO_4^-, 10 ppm F^-, and 50 ppm SO_4^-.

2. Number 2 Standard - Dilute 25 ml of the Intermediate Standard (VI.E) to 250 ml in reagent water to obtain the following concentrations: 5 ppm Br^-, 5 ppm Cl^-, 2.5 ppm NO_3^-, 2 ppm NO_2^-, 2 ppm PO_4^-, 2 ppm F^-, and 10 ppm SO_4^-.

3. Number 3 Standard - Dilute 5 ml of the Intermediate Standard (VI.E) to 100 ml in reagent water to obtain the following concentrations: 2.5 ppm Br^-, 2.5ppm Cl^-, 1.25 ppm NO_3^-, 1.0 ppm NO_2^-, 1.0 ppm PO_4^-, 1.0 ppm F^-, and 5.0 ppm SO_4^-.

4. Number 4 Standard - Dilute 2 ml of the Intermediate Standard (VI.E) to 100 ml in reagent water to obtain the following concentrations: 1.0 ppm Br^-, 1.0 ppm Cl^-, 0.5 ppm NO_3^-, 0.4 ppm NO_2^-, 0.4 ppm PO_4^-, 0.4 ppm F^-, and 2.0 ppm SO_4^-.

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5. Number 5 Standard - Dilute 0.5 ml of the Intermediate Standard (VI.E) to 100 ml in reagent water to obtain the following concentrations: 0.25 ppm Br^-,0.25 ppm Cl^-, 0.125 ppm NO_3^-, 0.10 ppm NO_2^-, 0.10 ppm PO_4^-, 0.10 ppm F^-, and 0.5 ppm SO_4^-.

F. Initial Calibration Verification - A certified premixed standard is purchased from SPEX Industries, Inc. (or another NIST-certified manufacturer) and should have the following approxamate concentrations: 10 ppm Br^-, 25 ppm Cl^-, 10 ppm NO_3^-, 5 ppm NO_2^-, 10 ppm PO_4^-, 5 ppm F^-, and 25 ppm SO_4^-.

VII. Sample Collection, Preservation, and Storage

A. Samples should be collected in scrupulously clean glass or polyethylene bottles.

B. Sample preservation and holding times for the anions that can be determined by this method are as follows:

Analyte	Preservation	Holding Time
Bromide	None required	28 days
Chloride	None required	28 days
Nitrate-N	Cool to 4°C	48 hours
Nitrite-N	Cool to 4°C	48 hours
NO_3-NO_2	Cool to 4°C	28 days
0-Phosphate-P	Cool to 4°C	24 hours
Sulfate	Cool to 4°C	28 days
Fluoride	Cool to 4°C	28 days

VIII. Calibration and Standardization

A. Start the pump by pressing the number one eluant line and the pump start button. Gradually increase flow to 2.0 ml/min. by increments of 0.5 until ready line comes on.

B. Start regeneration flow by pressing B channel button on the column box.

C. Turn on the cell and computer. Range and pump will be set automatically by Dionex mini-chrome Software.

D. Wait for system to stabilize, generally 10 - 15 minutes.

E. Inject 1.0 ml of the mid-range calibration standard.

F. Using the retention times from this standard, build your calibration table entering the concentrations for each standard, in Calibration Menu of Dionex Mini-chrome software.

G. Using injections of 1.0 ml of each calibration standard, tabulate peak area against the concentration. The results will be used to prepare a calibration curve for each analyte. The coefficient for each anion should be 0.995 or better in the Cal Plot Menu of the mini-chrome software.

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H. The calibration curve must be verified on each working day, or whenever the anion eluent is changed, and after every 10 samples. If the response or retention time for any analyte varies from the expected values by more than ± 10 %, the test must be repeated, using fresh calibration standards. If the results are still more than ± 10%, a new calibration curve must be prepared for that analyte.

IX. Procedure

A. Start the system and prime the column by injecting a standard. Then check the calibration as described in Section VIII.

B. Load and inject 1.0 - 2.0 ml of well mixed sample. Flush the injection loop thoroughly, using each new sample. Use the same size loop for standards and samples.

C. The width of the retention time window used to make identifications should be based upon measurements of actual retention time variations of standards.

D. If the response for the peak exceeds the working range of the system, dilute the sample with an appropriate amount of reagent water and reanalyze.

E. If the resulting chromatogram fails to produce adequate resolution, or if identification of specific anions is questionable, fortify the sample with an appropriate amount of standard and reanalyze.

F. The following extraction should be used for solid materials. Weigh 10.0 g of sample into a plastic 4 oz sample container. Add 100 ml of deionized water. Shake for 10-20 minutes in a mechanical shaker. Pour slurry into a 40 ml VOA vial, and centrifuge for 30 min. Filter the supernatant through a 0.2 micron I.C. filter, and load autosampler as normal.

X. Quality Control

A. Immediately after the calibration has been established, the calibration must be verified for every analyte by the analysis of an Independent Standard (ICV). When measurements exceed the 90 - 110% confidence interval, the analysis must be terminated, the problem corrected, the instrument recalibrated, and the calibration reverified.

B. A Calibration Verification (CCV) and a Calibration Blank (CCB) must be ran every 10 analytical samples and after the last sample. The CCV must agree within 90-110% Recovery of the expected value, and the CCB must be below the reporting limit. If they are not, terminate the analysis, correct the problem, and recalibrate the instrument.

C. The following QC samples must be analyzed with every batch of 20 samples per matrix:

1. Preparation Blank - result must be below the Reporting limit for the analyte of interest, or the analysis must be terminated and samples re-prepared.

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2. Laboratory Control Sample and Laboratory Control Sample Duplicate - Measurements must be within 90-110% recovery, or the analysis must be terminated and samples re-prepared.

3. Matrix Spike and Matrix Spike Duplicate - If Measurements fall outside of 75-125%R the data will be flagged. The following procedure will be followed for matrix spikes:

   a. Waters - Add 10 µl of a 10,000 mg/l stock standard for each analyte and bring up to a final volume of 5 ml with the sample, giving a spike value of 20 mg/l.

   b. Soils - Add 0.2 ml of 10,000 mg/l stock standard for each analyte to 10 g of sample. Follow the soil extraction procedure in IX.F., thus giving a 20 mg/l or 200 mg/kg spike value.

XI. CALCULATIONS

A. Results are printed in mg\l. No calculations are necessary for waters.

B. For Soils:

$$mg/kg = (R \times V) / g$$

Where:

R = result in mg/l

V = Volume of reagent water added for the extraction (milliliters)

g = the grams of the sample used in the extraction

XII. DATA VERIFICATION

A. Regulations covering Data Verification are located with the General Inorganics Standard Operating Procedures.

XIII. REFERENCES

A. U.S. Environmental Protection Agency, Methods for the Determination of Inorganic Substances in Environmental Samples, August 1993, Method 300.0

B. Standard Methods for the Examination of Water and Wastewater, 18th Edition, APHA-AWWA-WPCF, Method 4110, pp. 4-1 to 4-5

C2-326

GC VOLATILES DEPARTMENT

PROCEDURAL DOCUMENTS - SYSTEM TRANSITION

Please note that the following document is in transition from a previous system. The document identification number (listed below) has been assigned in compliance with the SWL SOP titled:

"Preparation, Review, Revision, and Control of Procedural Documents", SWL-GA-101, Rev. 1.0 — 05/14/97

As a matter of priority, the following document has been reviewed and re-issued in accordance with the laboratory's annual review policy. Although its approval/cover page is as set forth in the above-referenced SOP, the full document has yet to be fully converted into it's required format. This document is scheduled to be re-issued within 60 days of the revision date reflected below in full compliance with all guidelines set forth in GA-101. Please note the corrective action report following this transitional cover.

Existing ID: SWL-AATIN200

NEW DOCUMENT IDENTIFICATION NUMBER:

SWL-OG-105

Standard Operating Procedure

"Oil & Grease (IN200/IN205)"

Rev. No. 3.3
Issued: 02/10/97

C2-327

AMERICAN ANALYTICAL & TECHNICAL SERVICES, INC. / SOUTHWEST LABORATORY OF OKLAHOMA, INC.

STANDARD OPERATING PROCEDURE FOR THE ANALYSIS OF OIL & GREASE

IN200, IN205
REV 3.2 — 1/05/96

Document Control # AATSWL-IN200-2028 2/4/98
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MM.

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Section 1.0 – Scope and Application

1.1 This SOP is applicable to the analysis of solid and liquid samples for oil and grease. Concentrations from 0.5 mg/L and upward for water and 10 mg/kg and upward for solids are obtainable using the methods described herein.

Section 2.0 – Method Summary

2.1 Samples are extracted with trichlorotrifluoroethane (freon) or n-hexane (gravimetric only) to remove petroleum hydrocarbons. Water samples must be extracted and analyzed within 28 days and soils must be extracted within 14 days and analyzed within 40 days of sample collection. Oil and grease in the extract is then determined by infrared analysis according to EPA Method 413.2 or gravimetrically, after driving off the solvent, according to EPA Method 413.1 or 1664.

Section 3.0 – Sample Preservation, Containers, Handling & Storage

3.1 Soil samples should be collected and stored in airtight glass containers with teflon lined lids. A minimum of 100 grams of sample should be collected. Samples should be maintained at 39°F ± 4° (4°C ± 2°) during shipment and storage prior to analysis. Aqueous samples should be collected in airtight glass containers with teflon lined lids. A minimum of 1 L of sample should be collected. Aqueous samples should be preserved with sulfuric acid to <2 pH. Samples should be maintained at 39°F ± 4° (4°C ± 2°) during shipment and storage prior to analysis.

Section 4.0 – Interferences and Potential Problems

4.1 Solvents, reagents, and processing hardware may yield artifacts leading to misinterpretation of data. The system must be demonstrated to be free from interferences under the conditions of the analysis by running method blanks. Specific selection of reagents and purification of solvents may be required.

Section 5.0 – Equipment/Apparatus

5.1 *Pipets* — 1 mL, 5 mL and 10 mL

5.2 *Volumetric Flasks* — 100 mL, 1000 mL

5.3 *Round-bottomed (Florence) flasks* — 500 mL

5.4 *Three-ball Snyder columns*

5.5 *Separatory Funnels* — 2000 mL glass with Teflon stopcock

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5.6 *Beakers* — 400 mL

5.7 *Filter Funnels* — Glass with six-inch tip

5.8 *Filter Paper* — S&S #595 or equivalent

5.9 *Graduated Cylinder* — 1000 ml

5.10 *Horn Sonicator* — Tekmar Sonic Disrupter with 3/4 inch horn or equivalent

5.11 *Separatory Funnel Shaker* — Glas-Col, or equivalent

5.12 *Spectrophotometer* — Perkin Elmer 1600 FTIR or equivalent

5.13 *Scintillation Vials* — 20 mL

Section 6.0 – Reagents and Standards

6.1 *Anhydrous Sodium Sulfate* — Mallinckrodt #8024KJXE, or equivalent

6.2 *Freon* — EM TX1165-1, or equivalent

6.3 *n-Hexane* — Mallinckrodt ChromAR, or equivalent

6.4 *Iso-octane* — Baker #9335-3, or equivalent

6.5 *n-Hexadecane* — Fluka AG #CH9470, or equivalent

6.6 *Chlorobenzene* — Aldrich #28,451-3, or equivalent

Section 7.0 – Procedure

7.1 Sample Preparation

7.1.1 Water Samples — Separatory Funnel Technique

7.1.1.1 Sample pH is checked with pH paper. Those samples with pH >2.0 are acidified to <2.0 with sulfuric acid.

7.1.1.2 Mark the water level on the side of the sample container. Decant the entire sample into a 2 liter separatory funnel. Using a one liter graduated cylinder, decant tap water into the sample container to the mark. Measure the amount poured from the graduate and record as the sample volume. Add 30 mL solvent to the sample bottle and shake vigorously to remove hydrocarbons remaining in the container. Vent the container to prevent pressure build-up. Add this solvent to the separatory funnel.

7.1.1.3 Perform the extraction based on SW846 Method 3510. Vigorously shake the separatory funnel for two minutes to thoroughly mix the two phases, venting as necessary to prevent pressure buildup. Allow the phases to separate (at least ten minutes). Filter the solvent phase through enough sodium sulfate to resolve any emulsion and remove entrained water. Collect in a 120 mL glass container for Infrared (IR) analysis or a round-bottomed flask for gravimetric analysis. Repeat the extraction twice more using 30 additional ml solvent each time.

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7.1.1.4 After filtering the last of the extract through the sodium sulfate, rinse the separatory funnel tip and the sodium sulfate twice to remove traces of hydrocarbon. Proceed to the desired analysis step B.1. or B.2.

7.1.2 Soil/Sludge Samples — Sonication Technique

7.1.2.1 Weigh 15-30 grams of sample into a clean 400 ml beaker. Stir in anhydrous sodium sulfate and mix until the mixture is of uniform consistency and free-flowing.

7.1.2.2 Add sufficient solvent to completely cover the sample and allow immersion of the sonicator probe tip one-fourth inch into the liquid.

7.1.2.3 Perform the extraction according to SW846 Method 3550. Immerse the probe tip into the liquid (1/8 to 1/4 inch). Sonicate three minutes at 50-80% duty cycle and 7-9 output control.

7.1.2.4 Decant the extract through sodium sulfate and collect in the glass extract container or round-bottomed flask. Repeat the extraction steps twice more. Proceed to the desired analysis step.

7.2 Standardization/Instrument Calibration

7.2.1 Infrared Analysis

7.2.1.1 Reference Oil Concentrate Preparation

7.2.1.1.1 Prepare reference oil concentrate by combining 15 mL n-hexadecane, 15 mL isooctane and 10 mL chlorobenzene in a 40 mL VOA vial with teflon liner. Keep tightly sealed and refrigerated when not in use.

7.2.1.2 Stock Standard Preparation

7.2.1.2.1 Prepare the stock standard by pipetting 1 g of reference oil into a 100 ml volumetric flask and dilute to volume with freon. Prepare monthly or sooner as needed. Record this and all subsequent standards in a standards log book, uniquely identifying each standard. Record this unique identity in the bench notebook each time the standard is used.

7.2.1.3 Initial Calibration

7.2.1.3.1 From the stock standard, prepare working standards at five concentrations (e.g. 5, 10, 20, 30 & 40 mg/100mL) by diluting with freon.

7.2.1.3.2 Prepare a calibration curve by plotting absorbance (at 2930 cm^{-1}) vs concentration for the five working standards. The acceptance limit is a minimum correlation coefficient of 0.99. If the acceptance limit is not met, prepare new standards and perform initial calibration again. All standards should be replaced and new initial calibration performed at a minimum of every six months.

7.2.1.4 Continuing Calibration

7.2.1.4.1 At the beginning of each day, adjust the absorbance to zero with fresh freon. Read and record the absorbance for a mid level standard (e.g. 20 mg/100 mL) daily. If the absorbance is within ±15% of the

original value as determined from the initial calibration the system is in calibration and sample analysis can proceed. Otherwise, prepare a fresh mid level standard. If the absorbance of this fresh standard is also out of limits, prepare new working standards and perform initial calibration again.

7.2.2 Gravimetric Analysis

7.2.2.1 Primary Spiking Standard Preparation

7.2.2.1.1 Prepare primary spiking standard by weighing 1.0 g #2 fuel oil and 1.0 g corn oil into a 100 mL volumetric flask. Dilute to volume with the solvent. Prepare the working spiking standard by measuring 25 mL primary standard into a 1000 mL volumetric flask and diluting to volume. Replace primary and working standards at a minimum of every 12 months.

7.3 Sample Analysis

7.3.1 IR Analysis

7.3.1.1 For IR analysis, bring the extract volume to the 100 ml mark on the extract container. Measure the absorbance at 2930 cm^{-1} and record.

7.3.1.2 Oil and Grease is calculated as described in Section VIII.

7.3.2 Gravimetric Analysis

7.3.2.1 For gravimetric analysis, place two boiling chips in the flask and place a three-ball Snyder column atop the flask. Place the flask in a water bath at 70°C and allow the solvent to boil and reflux until the volume of solvent remaining in the flask is about the diameter of a half dollar. Remove the flask from the bath and allow to cool. Decant the remaining extract into a clean, previously weighed scintillation vial. Rinse the flask three times with 1-2 mL each time and decant each rinse into the scintillation vial. Place the open scintillation vial under the nitrogen blow-down apparatus until all solvent is evaporated. Cool the vial in a desiccator one hour and reweigh to a constant weight as determined by two consecutive weighings.

Section 8.0 -- Calculations

8.1 Oil and Grease by IR is calculated as follows:

$$C = (A_{smp}/A_{std})(C_{std})(100/V)$$

Where:

C = concentration (mg/l or mg/kg)
A_{smp} = absorbance of sample extract
A_{std} = absorbance of standard
C_{std} = concentration of standard
V = sample volume (L) or weight (g)

8.2 Oil and Grease, Gravimetric is calculated as follows:

$$C = [(W_r - W_t)*1000]/V$$

Where:

C = concentration (mg/l or mg/kg)
W_r = weight of vial + residue (g)
W_t = tare weight of empty vial
V = sample volume (L) or weight (Kg)

Section 9.0 -- Quality Control

9.1 Instrument Baseline

9.1.1 For IR analysis the absorbance must be brought to zero before analyses are performed. The baseline is established using pure freon and is checked after each ten sample readings, or fewer if instrument drift is suspected.

9.2 Blanks

9.2.1 Prepare one blank per batch of up to 20 samples. Water blanks are prepared using 1000 mL deionized tap water. Soil blanks are prepared using 30 g sodium sulfate. Blanks are taken through the entire extraction process.

9.3 Matrix Spikes/Duplicates

9.3.1 Prepare a spike/spike duplicate pair for each batch of 20 or fewer soil samples. For IR analysis, spikes are prepared by adding 2 mL of the stock standard to the already mixed soil/sodium sulfate prior to sonication. For gravimetric analysis, spikes are prepared by adding 20 mL of the working spiking standard to the already mixed soil/sodium sulfate prior to sonication. Analyst discretion is used to determine which soil sample to spike.

9.3.2 When sufficient sample volume is provided (i.e. three separate one liter containers), matrix spikes/duplicates for waters are prepared by adding 2 mL of the stock standard (IR analysis) or 20 mL of the working standard

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(gravimetric analysis) to the separatory funnel containing the sample to be spiked.

9.4 Laboratory Control Spikes

9.4.1 For soils, provide one laboratory control spike per day by adding 2 mL of the stock standard (IR analysis) or 20 mL of the working standard (gravimetric analysis) to 30 g of sodium sulfate and carry through the extraction procedure.

9.4.2 For waters, provide a spike/duplicate pair per batch of 20 or fewer samples by adding 2 mL of stock standard (IR analysis) or 20 mL of the working standard (gravimetric analysis) to one liter of deionized tap water and carry through the extraction process.

9.5 Control Limits

9.5.1 If the soil blank contains >10 mg/kg oil and grease the system is out of control. Extract a new blank and reextract all samples associated with that blank.

9.5.2 If a water blank contains >0.5 mg/l oil and grease, so note on the report to the client.

9.5.3 The acceptance range for soil spike recoveries is 50-120%. If a spike is out of limits, reextract. If the reextract is also out of limits the system is out of control and the batch of samples associated with that spike must be reextracted with a new spike/duplicate pair. Find and correct the problem before proceeding.

9.5.4 The acceptance limit for relative percent difference (RPD) for soil spike/duplicates is 20%. If RPD >20%, reextract the spike/duplicate pair, taking care to homogenize the sample. If the RPD is still >20%, evaluate the laboratory control spike (LCS). If the LCS is within control limits the system is in control and a matrix effect has resulted in the out-of-limit spike/spike duplicate RPD. Submit the LCS result with the report and no further action is necessary. If the LCS is out of acceptance limits the system is out of control and the batch of samples associated with that spike/duplicate pair must be reextracted. Ascertain and correct the problem prior to reextracting.

9.5.5 The acceptance range for water control spike recoveries is 50-115%. If a spike is out of limits, extract a new spike. If the new extract is out of limits the system is out of control and results are so noted to the client. Find and correct the problem before proceeding.

9.5.6 RPD of water control spikes should be <20%. Otherwise, extract a new spike or spike duplicate or both. If the RPD is still >20% the system is out of control and the problem must be ascertained and corrected before more samples are extracted and analyzed.

Section 10.0 – Data Verification

10.1 The analyst checks all calculations and initials the appropriate page in the bench notebook. The supervisor periodically reviews the bench notebook for accuracy and completeness and initials pages reviewed. All reports are reviewed by the supervisor before issuance of the final report.

Section 11.0 – Health and Safety

11.1. Good laboratory practices dictate the careful handling of all laboratory samples and reagents.

Section 12.0 – References

12.1 EPA Methods 413.1, 413.2, and 1664

12.2 SW-846, 9070

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PROCEDURAL DOCUMENTS - SYSTEM TRANSITION

Please note that the following document is in transition from a previous system. The document identification number (listed below) has been assigned in compliance with the SWL SOP titled:

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Existing ID: IN030D

NEW DOCUMENT IDENTIFICATION NUMBER:

SWL-IN-300

Standard Operating Procedure

"Inorganic Ions in Soil & Water
by Ion Chromatography (Dionex DX300)"
Rev. No. 1.3
Issued: 7/31/96

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SOUTHWEST LABORATORY OF OKLAHOMA, INC.

STANDARD OPERATING PROCEDURE FOR THE DETERMINATION OF INORGANIC ANIONS IN WATER & SOIL BY ION CHROMATOGRAPHY (DIONEX DX 300)

IN030, IN055, IN135, IN185,
IN195, IN255, IN295
REV 1.3 — 7/31/96

Document Control # SWL-IN030D-2033 2/6/98 MM
Alan Fosdick

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Signature: Chuck Hoover

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I. SCOPE AND APPLICATION

A. This method covers the determination of the following inorganic anions; Bromide, Chloride, Nitrate-N, Nitrite-N, Ortho-Phosphate-P, Sulfate, and Fluoride.

B. This method is applicable to the determination of the above anions in drinking water, surface water, mixed domestic and industrial wastewaters, groundwater, reagent waters, and solids (after extraction).

C. This method is recommended for use only by or under the supervision of analysts experienced in the use of ion chromatography and in the interpretation of the resulting chromatogram. Each analyst must demonstrate the ability to generate acceptable results with this method.

II. SUMMARY OF METHOD

A. A 1.0 milliliter volume of sample is introduced into the ion chromatograph via an autosampler. The anions of interest are separated and measured, using a system comprised of a guard column, separator column, suppressor device, and conductivity detector.

B. In order to use this method for solids, an extraction procedure must be performed.

III. INTERFERENCES

A. Interferences can be caused by substances with retention times that are similar to and overlap those of the anion of interest. Large amounts of an anion can interfere with the peak resolution of an adjacent anion. Sample dilution and/or spiking can be used to solve most interference problems.

B. Method interferences may be caused by contaminants in the reagent water, reagents, glassware, and other sample processing apparatus that lead to discrete artifacts or elevated baseline in ion chromatograms.

C. Samples that contain particles larger than 0.45 microns and reagent solutions that contain particles larger than 0.20 microns require filtration to prevent damage to instrument columns and flow systems.

IV. SAFETY

A. Normal, accepted laboratory safety practices should be followed during reagent preparation and instrument operation. No known carcinogenic materials are used in this method.

V. APPARATUS AND MATERIALS

A. *Balance* — Mettler/H80, capable of accurately weighting to the nearest 0.0001 g.

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B. *Ion Chromatograph* — Dionex Model DX300, with required accessories including syringes, analytical columns, compressed gases, 0.2 micron filters, and detectors.

C. *Anion guard column* — Dionex Ion Pac AG4A-SC

D. *Anion separator column* — Dionex Ion Pac AS4A-SC

E. *Anion suppressor device* — Dionex Anion Micro-Membrane Suppressor (P/N 43074).

F. *Detector* — Dionex Conductivity Cell

G. Dionex AI 450 Software and Computer

H. Dionex Automated Sampler supplied with polyvials and filtercaps

VI. REAGENTS AND CONSUMABLE MATERIALS

A. *Reagent water* — Deionized water, free of the anions of interest. The water must be filtered through a 0.20 micron filter.

B. *Eluent Solution* — Sodium bicarbonate 1.7/ mM, sodium carbonate 1.8 mM. Dilute 20 ml of Dionex AS4A Eluent Concentrate (P/N 39513) in reagent water and dilute to 2 liters. If concentrate is unavailable, dissolve 0.2856 g sodium bicarbonate ($NaHCO_3$) and 0.3816 g of sodium carbonate (Na_2CO_3) in reagent water and dilute to 2 liters. Sparge the eluent for 20 min with high purity Helium before use.

C. *Stock standard solutions, 10,000 mg/l* — Certified stock standards for all anions are purchased from SPEX Industries, Inc. (or another NIST-certified manufacturer), and are the basis for all working standards.

D. *Working Standards (Method DX300):*

1. *Number 1 Standard* — Dilute the 10000 ppm stock standards to 500 ml according to the following schedule: 2.5 ml Br (50 ppm), 5.0 ml Cl^-(100ppm), 2.5 ml NO_3^-N (50 ppm), 1.0 ml NO_2^-N (20 ppm), 1.0 ml PO_4^-P (20 ppm), 1.0 ml F^-(20 ppm), and 5.0 ml SO_4 (100 ppm).
2. *Number 2 Standard* — Dilute 125 ml of the No. 5 Standard to 250 ml in reagent water to obtain the following concentrations: 25 ppm Br, 50 ppm Cl, 25 ppm NO_3^-N, 10 ppm NO_2^-N, 10 ppm PO_4^-P, 50 ppm SO_4, and 10 ppm F.
3. *Number 3 Standard* — Dilute 25 ml of the high No. 5 Standard to 250 ml in reagent water to obtain the following concentrations: 5 ppm Br, 10 ppm Cl, 5 ppm NO_3^-N, 2 ppm NO_2^-N, 2 ppm PO_4^-P, 10 ppm SO_4, and 2 ppm F.
4. *Number 4 Standard* — Dilute 1 ml of the No. 5 Standard to 100 ml to obtain the following concentrations: 0.5 ppm B^-r; 1.0 ppm Cl; 0.5 ppm NO_3^-N, 0.2 ppm NO_2^-N, 0.2 ppm PO_4^-P, 1.0 ppm SO_4, 0.2 ppm F.
5. *Number 5 Standard* — Dilute 0.5 ml of the No. 5 Standard to 100 ml to obtain the following

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concentrations: 0.25 ppm Br, 0.5 ppm Cl, 0.25 ppm NO_3^-N, 0.1 ppm NO_2^-N, 0.1 ppm PO_4^- P, 05 ppm SO_4, 0.1 ppm F.

E. *Initial Calibration Verification* — A certified premixed standard is purchased from SPEX Industries, Inc. (or another NIST- certified manufacturer) with the following concentrations: 10 ppm Br, 25 ppm Cl, 10 ppm NO_3^-N, 5 ppm NO_2^-N, 10 ppm PO_4^-P, 25 ppm SO_4, and 5 ppm F.

VII. SAMPLE COLLECTION, PRESERVATION, AND STORAGE

A. Samples should be collected in scrupulously clean glass or polyethylene bottles.

B. Sample preservation and holding times for the anions that can be determined by this method are as follows:

Analyte	Preservation	Holding Time
Bromide	None required	28 days
Chloride	None required	28 days
Nitrate-N	Cool to 4°C	48 hours
Nitrite-N	Cool to 4°C	48 hours
NO_3-NO_2	Cool to 4°C	28 days
O-Phosphate-P	Cool to 4°C	24 hours
Sulfate	Cool to 4°C	28 days
Fluoride	Cool to 4°C	28 days

VIII. CALIBRATION AND STANDARDIZATION

A. Turn on high purity Helium and high purity Nitrogen tanks. Adjust flow pressure to 100 psi.

B. Turn on computer. Turn on DX300 power switch located at the top left corner of the instrument.

C. Turn on the system switch of the eluent degas module. Set the number 2 switch to the up position, and the corresponding SPARGE/PRESSURIZE switch to PRESSURIZE.

D. Turn on the cell. Press start on the gradient pump. Flow is set at 1.90 ml/ min. Pressure must be >1000 psi. Wait for system to stabilize (generally 10 - 15 minutes), until the conductivity is between 14.0 and 18.0 uS.

E. Select a calibration schedule from the Schedule Menu of Dionex AI-450 software. Use AUTODX5.SCH. AUTOCAL1 is always the highest working standard, while AUTOCAL5 is always the lowest working standard. If the instrument has not been used in the past 24 hours, insert a PRIME sample at the beginning of the schedule to flush the system. Any standard may be used as the PRIME.

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F. Load autosampler cartridges following the selected schedule. Fill supplied polyvials to the mark, and cap with a filtercap. Use supplied tool so that the top of the cap is flush with the top of the vial. Place the cartridge in the autosampler, and set the RUN/HOLD switch to RUN. Set the MODE switch to CONST.

G. Enter the Run Menu of the Dionex AI-450 software. Select the DX300 window. Under the Load Menu, select SCHEDULE, and then load the schedule selected in Sec. VIII. F. At the LOAD SCHEDULE prompt, choose the "Store Data Files in the Following Directory" option, and set the directory as "C:\DX\DATA\DX300 \X," where X is either WATER or SOIL.

H. Set both the cell and gradient pump to REMOTE operation. DO NOT set the autosampler to remote operation; it must remain in the LOCAL mode.

I. Under the Run Menu, select START. Select YES at the prompt. The instrument will start the run. Each sample takes approximately 12 minutes to run.

G. After the run has completed, go to the Cal Plot Menu of the AI-450 software Main Menu. Load the appropriate METHOD from the File Menu (DX300A5.MET for waters). This will graphically show the calibration curve for each analyte. The coefficient for each anion must be 0.995 or better.

H. The calibration curve must be verified on each working day, or whenever the anion eluent is changed, and after every 10 samples. If the response or retention time for any analyte varies from the expected values by more than ±10 %, the test must be repeated, using fresh calibration standards. If the results are still more than ±10%, a new calibration curve must be prepared for that analyte.

IX. PROCEDURE

A. Start the system as described in Section VIII. A-D.

B. Go to the Schedule Menu of the AI-450 software Main Menu. Under Sample Name, enter the SWLO sample ID number. In the method column, choose the appropriate method (DX300A5.MET) for each sample. Enter a data file using the following system: Xmmddyy, where X is the matrix (W for waters, S for soils), and mmddyy is the current date (for example, a water sample run on January 19, 1995 is entered as W011995). The following samples must be placed at the beginning of each schedule: LCS (the No.4 Standard), LCSD (a duplicate of the LCS), ICV (the certified pre-mixed standard, see Sec.VI.F.), and ICB (a blank of reagent water). In addition, a CCV (the No. 4 Standard) and a CCB (a blank of reagent water) must be run after every tenth sample. If the instrument has not been used in the past 24 hours, insert a PRIME sample at the beginning of the schedule to flush the system. Any standard may be used as the PRIME. Save the schedule following the same notation used for the data file.

C. Load autosampler cartridges following the selected schedule. Fill polyvials to the mark with sample that has been filtered through a 0.2 micron I.C. filter, and cap with a filtercap. Use supplied tool so that the top of the cap is flush with the top of the vial. Place the cartridge in the autosampler, and set the RUN/HOLD switch to RUN. Set the MODE switch to CONST.

D. Enter the Run Menu of the Dionex AI-450 software. Select the DX300 window. Under the Load

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Menu, select SCHEDULE, and then load the schedule selected in Sec. VIII. F. At the LOAD SCHEDULE prompt, choose the "Store Data Files in the Following Directory" option, and set the directory as "C:\DX\DATA\DX300 \X," where X is either WATER or SOIL.

E. Set both the cell and gradient pump to REMOTE operation. DO NOT set the autosampler to remote operation; it must remain in the LOCAL mode.

F. Under the Run Menu, select START. Select YES at the prompt. The instrument will start the run. Each sample takes approximately 12 minutes to run.

G. The width of the retention time window used to make identifications should be based upon measurements of actual retention time variations of standards.

H. If the response for the peak exceeds the working range of the system, dilute the sample with an appropriate amount of reagent water and reanalyze.

I. If the resulting chromatogram fails to produce adequate resolution, or if identification of specific anions is questionable, fortify the sample with an appropriate amount of standard and reanalyze.

J. The following extraction should be used for solid materials. Weigh 10.00 g of sample into a plastic 4oz sample container. Add 100 ml of deionized water. Shake for 10 - 20 minutes. in a mechanical shaker. Pour slurry into a 40 ml VOA vial, and centrifuge for 30 min. Filter the supernatant through a 0.2 micron I.C. filter, and load autosampler as normal.

X. QUALITY CONTROL

A. Immediately after the calibration has been established, the calibration must be verified for every analyte by an Initial Calibration Standard (ICV). The ICV must be from a different standard source then the calibration standards. When measurements are not within ±10% of the expected value, the analysis must be terminated, the problem corrected, the instrument recalibrated, and the calibration reverified.

B. A Preparation Blank (PBW or PBS) must be run with every batch of 20 samples per matrix. The PBW or PBS must be below the Reporting Limit for each analyte, or the analysis must be terminated and reprep samples.

C. A Laboratory Control Sample (LCW or LCS) and a duplicate (LDW or LDS) must be ran with every batch of 20 samples per matrix. Measurements must be within ±10% of the expected value, or the analysis must be terminated and reprep samples.

D. A Calibration Verification (CCV) and a Calibration Blank (CCB) must be ran every 10 analytical samples and after the last sample. The CCV must agree within ±10% of expected value, and the CCB must be below the reporting limit. If they are not, terminate the analysis, correct the problem, and recalibrate the instrument.

E. One set Matrix Spike (MS) and Matrix Spike Duplicate (MSD) analysis will be performed per 20 samples or batch. The following procedure will be followed for matrix spikes:

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1. For waters: Add 10 µl of a 10,000 mg/l stock standard for each analyte and bring up to a final volume of 5 ml with the sample, giving a spike value of 20 mg/l.
2. For soils: Add 0.2 ml of a 10,000 mg/l stock standard for each analyte to 10 g of sample. Follow the soil extraction procedure in IX.J., thus giving a 20 mg/l or 200.0 mg/kg spike value.

XI. CALCULATIONS

A. Results are printed in mg/L. No calculations are necessary for waters.

B. For soils

$$mg/kg = \frac{R \times V}{g}$$

Where:

R = result in mg/L

V = volume of H_2O added for the extraction (milliliters)

g = the grams of the sample used in the extraction

XII. DATA VERIFICATION

A. Regulations covering Data Verification are located within the General Inorganics Standard Operating Procedure.

XIII. HEALTH AND SAFETY

A. Safety glasses must be worn at all times within the laboratory.

B. Gloves should be worn during filtration of the samples.

XIV. REFERENCES

A. U.S. Environmental Protection Agency, Methods for the Determination of Inorganic Substances in Environmental Samples, EPA/600/R-93/100, August 1993, Method 300.0

B. Standard Methods for the Examination of Water and Wastewater, 18th Edition, APHA-AWWA-WPCF, Method 4110, pp.4-1 to 4-5

C. Dionex, DX-300 Series Chromatography System Operation Manual, Dionex Corp., Sunnyvale, CA 94086, October 1992

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D. Dionex, AI-450 Chromatography Software User's Guide, Dionex Corp., Sunnyvale, CA 94086, March 1993

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PROCEDURAL DOCUMENTS - SYSTEM TRANSITION

Please note that the following document is in transition from a previous system. The document identification number (listed below) has been assigned in compliance with the SWL SOP titled:

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Upon revision or review, the following document will be re-issued in full compliance with the guidelines set forth in GA-101, therefore consistent with newly converted or written procedural documents.

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SWL-IN-307

Standard Operating Procedure

"Analysis of Cyanide (SPECTROPHOTOMETER)"
Rev. No. 1.1
Issued: 10/28/96

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SOUTHWEST LABORATORY OF OKLAHOMA, INC.

STANDARD OPERATING PROCEDURE FOR THE ANALYSIS OF CYANIDE (SPECTROPHOTOMETER)

IN120/IN121

REV 1.1 — 10/28//96

Document Control #SWL-IN121- 2034 2/9/98

Alan Fosdick

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I. MIDI DISTILLATION PROCEDURE for TOTAL CYANIDES

A. Distillation Summary

1. The cyanide as hydrocyanic acid (HCN) is released from cyanide complexes by means of a reflux-distillation operation and absorbed in a scrubber containing sodium hydroxide solution.

B. Interferences

1. 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.
2. Oxidizing agents such as chlorine decompose most cyanides. Chlorine interferences can be removed by adding an excess of ascorbic acid.
3. Sulfide interferences can be removed by adding an excess of bismuth nitrate to the waste before distillation.
4. High results may be obtained for samples that contain nitrate and /or nitrite. The possibility of interference of nitrate and /or nitrite is eliminated by pretreatment with sulfamic acid just before distillation.

C. Apparatus

1. Midi-reflux distillation apparatus having a 100 ml boiling flask, gas-dispersion tube, allihn water-cooled condenser, air-inlet tube, vacuum source and screw clamp to adjust vacuum flow.

D. Distillation Reagents

1. Sodium Hydroxide solution, 0.25N: Dissolve 10 grams of NaOH in ASTM Type II water. Dilute to 1 liter with ASTM Type II water.
2. Sulfuric Acid: 50%: Carefully add 500 ml of concentrated H_2SO_4. After the solution cools, dilute to 1 liter with ASTM Type II water.
3. Magnesium chloride solution: Weigh 510 grams of $MgCl_2 \bullet 6H_2O$ into a 1000 ml flask. Dissolve and dilute to 1 liter with ASTM Type II water.
4. Ascorbic Acid, Analytical Reagent Grade.
5. Bismuth Nitrate: Dissolve 30.0 grams of $Bi(NO_3)_3$ in 100 ml of ASTM Type II water. While stirring, add 250 ml of glacial acetic acid. Stir until dissolved. Dilute to 1 liter with ASTM Type II water.
6. Sulfamic Acid: Dissolve 40 g of sulfamic acid in ASTM Type II water. Dilute to 1 liter.
7. Stock potassium cyanide solution: Dissolve 2.51 grams of KCN and 2 grams KOH in one liter of ASTM Type II water. Standardize with 0.0192 N $AgNO_3$ (1 ml = 1.0 mg).

   Note: Detailed procedure for $AgNO_3$ standardization is described in "Standards Methods for the Examination of Water and Wastewater", 16th edition. (1985), methods 412C and 407A.

8. Intermediate standard potassium cyanide solution: Dilute 100 ml of Stock potassium cyanide (I.D.7.) to 1000 ml with ASTM Type II water (1 ml = 100 µg CN).

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9. Working Standard potassium cyanide solution : Dilute 100 ml of intermediate stock solution (I.D.8.) to 1000 ml with 10 ml of 0.25 N NaOH (1 ml = 10 µg). Prepare Daily.

10. Standard silver nitrate solution, 0.0192 N: Prepare by crushing approximately 5 grams of $AgNO_3$ crystals and drying to constant weight at 40° C. Weigh out 3.2647 grams of dried $AgNO_3$. Dissolve in distilled water and dilute to 1000 ml (1 ml = 1 mg CN).

E. Distillation Procedure

1. Test a drop of aqueous sample for the presence of chlorine using potassium iodide starch paper. A blue color indicates the need for treatment. Add ascorbic acid a few crystals at a time until a drop of sample produces no color on the indicator. Then add an additional 0.6 g of ascorbic acid for each liter of ASTM Type II water.

2. Place 50.0 ml of sample (waters), or 1.0 g ± 0.05 g sample (soils) plus 50 ml ASTM Type II water, into 100 ml boiling flask.

3. Add 30 ml of 0.25 N sodium hydroxide to the absorbing tube.

4. Connect the boiling flask, condenser / absorber and trap in the train.

5. Start a slow stream of air entering the boiling flask by adjusting the screw clamp on the vacuum line. Adjust the vacuum so that approximately three bubbles of air per second enters the boiling flask.

NOTE: The bubble rate will not remain constant after the reagents have been added and while heat is being applied to the flask. It will be necessary to readjust the air rate occasionally to prevent the solution in the boiling flask from backing up into the air inlet tube.

6. If the sample is water, use lead acetate paper to check for the presence of sulfide. A positive test is indicated by a black color on the paper. If positive, treat the sample by adding 5.0 ml of Bismuth Nitrate solution through the air inlet tube after the air rate is set. A separate set of calibration standards must be brought through the distillation process including the addition of Bismuth Nitrate for any samples with a positive test.

7. Add 5.0 ml of sulfamic acid solution into the air inlet tube.

8. Slowly add 5.0 ml 50% sulfuric acid through the air inlet tube. Rinse the tube with distilled water and allow the airflow to mix the flask contents for 5 minutes.

9. Pour 2 mls magnesium chloride solution into the air inlet and wash down with a stream of water.

10. Heat the solution to boiling and reflux for one hour.

11. Turn off the heat and continue the air flow for at least 15 minutes.

12. After cooling the boiling flask, disconnect the absorber and close off the vacuum source. Quantitatively transfer contents of the absorber tube to a plastic, 100 ml, snap-cap container.

13. Adjust the final volume to 50 ml with NaOH solution by weighing on a top-loading balance (50.00 g ± 0.25 g).

14. Seal the receiving solutions and store at 4° C until analyzed.

15. Document all distillation details in the Sample Distillation Record Log, the analyst must then sign and date the log (see Digestion / Distillation Log SOP).

16. For QC requirements see Section III.

II. TOTAL CYANIDE ANALYSIS IN WATER AND SOIL

A. Tested Concentration Range: 20 - 400 ug/l

B. Principle

1. Total cyanide from a alkaline distillates is converted to cyanogen chloride, CNCl, by reaction with chloramine-T at pH lower than 8. The CNCl then forms a red-blue dye by reacting with pyridine-barbituric acid reagent. The color is read at 578nm.

C. Interferences

1. See distillation procedure.

D. Apparatus

1. Spectrophotometer - Bausch & Lomb Spectronic 70, capable of reading at 578 nm with a 1.0 cm cell.
2. 100 & 250 ml volumetric flasks.
3. 10 ml graduated pipettes.
4. 50 ml volumetric pipettes.

E. Reagents

1. Sodium hydroxide solution (0.25N), NaOH, Dissolve 10 g NaOH in 1 liter of ASTM Type II water.
2. Sodium phosphate monobasic (1M): Dissolve 138 g of $Na_2H_2PO_4 \bullet H_2O$ in 1 liter of ASTM Type II water. Refrigerate this solution.
3. Chloramine-T solution (0.44%): Dissolve 1.0 g of white, water soluble chloramine-T in 100 ml of ASTM Type II water and refrigerate until ready to use.
4. Pyridine - Barbituric acid reagent: Place 15g of barbituric acid in a 250 ml volumetric flask and add just enough distilled water to wash the sides of the flask and wet the barbituric acid. Add 75 ml of pyridine and mix. Add 15 ml of concentrated hydrochloric acid (HCl), mix, and cool to room temperature. If you have difficulty getting the barbituric acid to dissolve, place flask in a ultrasonic bath for 15 min. Dilute to 250 ml with ASTM Type II water. Pyridine is toxic and this procedure should be done in a hood. This reagent is stable for approximately six months if stored in a cool, dark place.
5. Stock potassium cyanide solution: see (I.D.7.)
6. Intermediate standard potassium cyanide solution: see (I.D.8.)
7. Working Standard potassium cyanide solution: see (I.D.9.)

F. Procedure

1. Preparation of standards

a. Prepare a series of standards by pipetting suitable volumes of working standard potassium cyanide solution (I.D.9.) into a 50 ml volumetric flask. To each flask, add 10 ml of 1.25 N Sodium Hydroxide and dilute to 50 ml with ASTM Type II water. Prepare using the following table. The sodium hydroxide concentration will be 0.25 N.

ml of working standard solution (1 ml = 10 µg CN)	Final Concentration µg CN / L
0.0	Blank
0.2	40
0.4	80
0.8	160
1.0	200
1.5	300
2.0	400

b. After the standard solutions have been prepared according to the table above, pipet 50 ml of each standard solution into a 100 ml volumetric flask and proceed to steps F.2.b. to obtain absorbance values for the standard curve. The final concentrations for the standard curve will be one half of the amounts in the above table (final concentrations ranging from 20 to 200 µg / L).

c. Prepare a standard curve ranging from 20 to200 µg / L by plotting absorbance of standard versus the cyanide concentration. Linear regression analysis should result in a correlation coefficient > 0.995.

2. Sample Analysis

a. Transfer the 50 ml of distillate obtained into a 100 ml volumetric flask. If the sample is later found to be beyond the linear range of the colorimetric determination, redistillation of a smaller volume will be required.

Note: Temperature of reagents and spiking solution can affect the response of the colorimetric determination. The reagents stored in the refrigerator should be warmed to ambient temperature before use.

b. Add 15 ml of 1 M sodium phosphate solution and mix. Add 2 ml of chloramine-T and mix. Some distillates may contain compounds that have chlorine demand. One minute after the addition of chloramine-T, test for excess chlorine with KI-starch paper. If the test is negative, add 0.5 ml chloramine-T. After one minute recheck with KI-starch paper. Continue to add Chloramine-T in 0.5 ml increments until an excess is maintained. After 1 to 2 minutes, add 5 ml of pyridine-barbituric acid solution and mix.

c. Dilute to 100 ml with ASTM Type II water and mix again. Allow 8 minutes for color development and then read the absorbance at 578 nm in a 1 cm cell within 15 minutes. The sodium hydroxide concentration will be 0.125 N.

III. CALCULATIONS

A. Calculate the cyanide in soil, in mg/kg as follows:

$$CN,\ mg/kg = (A \times B \times 0.05) / (C \times D)$$

Where:

A = µg/l CN read from standard curve

B = ml of sample after preparation of colorimetric analysis (usually 100 ml)

C = ml of distillate used for colormetric analysis

D = wet weight of original sample distilled, in grams.

B. Calculate the cyanide in water, in µg/l, as follows:

$$CN,\ \mu g/l = (A \times B \times C) / (D \times E)$$

Where:

A = µg/l CN read from standard curve

B = ml of sample after preparation of colorimetric analysis (usually 100 ml)

C = ml of sample after distillation (usually 50)

D = ml of original sample for distillation

E = ml used for colorimetric analysis (usually 50)

IV. QUALITY CONTROL

A. Calibration Checks

1. Initial Calibration Verification (ICV) - After the system has been calibrated, the accuracy of the initial calibration shall be verified and documented by the analysis of the ICV(which has been carried through the distillation procedure). The measurement must be between 85-115 % recovery of the true value or the analysis terminated, the problem corrected, the instrument recalibrated and the calibration reverified.

2. Initial Calibration Blank (ICB) - Immediately after the analysis of the ICV the ICB must be analyzed. If the absolute result is greater than the current PQL, terminate analysis, correct the problem and recalibrate.

3. CRA Standard - To assure linearity of calibration at the low end of the range, the low standard which was carried through the digestion procedure, shall be run after the ICB. The control limit for this standard is 75-125 % of the true value.

4. Continuing Calibration Verification (CCV) - To assure calibration accuracy during each analysis run, a mid-range standard will be analyzed at the beginning, a frequency of 10% during the run, and after the last analytical sample. If the deviation of the CCV is not within 85-115 % recovery, the instrument will be recalibrated and the proceeding samples since the last CCV will be reanalyzed.

5. Continuing Calibration Blank (CCB) - A CCB is analyzed immediately after every CCV. Blanks are to be reported down to the instrument detection limit. If the blank result is greater than the current PQL, terminate analysis, correct the problem and recalibrate.

B. Preparation Batch Controls - The following samples must be distilled with every batch of samples.

1. Preparation Blank (PBW or PBS) must be distilled with every sample batch. If the absolute value is > PQL, re-distilled all samples associated with the prep blank.
2. Laboratory Control Sample (LCW or LCS) and a Laboratory Control Duplicate (LDW or LDS) must be distilled with every sample batch. If the recovery is outside of 80-120 % recovery, re-distill all associated samples.
3. Duplicate samples - Will be analyzed upon request of the client. Control limit for the duplicate will be <20 RPD.
4. Matrix Spike (MS) and matrix spike duplicate (MSD) samples - Control limits are 75-125 % recovery, and must be prepared with every batch of samples.

V. REFERENCES

A. U.S. EPA SW846, Method 9010A

B. EPA Methods for Chemical Analysis of Water and Wastes, 3rd edition, Method 335.2

SOUTHWEST LABORATORY OF OKLAHOMA, INC.

TM

Standard Operating Procedure

Homogenization of Soil, Sediment, and Sludge Samples

Document No.: SWL-GA-140

Rev No. / Date: Rev. 1.0 — 11/20/97

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Laboratory Safety Officer	Date
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HOMOGENIZATION OF SOILS, SEDIMENTS, and SLUDGES

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TYPIST: pn

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1.0 SCOPE and APPLICATION

1.1 The scope of this document is to detail the procedures used to obtain representative samples from soils, sediments, and sludges for all analyses except where volatile compounds are to be analyzed.

1.2 The procedures described herein will apply to all technicians involved in the preparation of sample media prior to analysis. Generally this will include the Sample Custodians, Extractions Chemists/Technicians, and/or Inorganic Chemists/Technicians

2.0 METHOD SUMMARY – N/A

3.0 HEALTH and SAFETY – N/A

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE – N/A

5.0 INTERFERENCE and POTENTIAL PROBLEMS

5.1 Care must be used when handling any sample since they may contain hazardous materials.

5.2 Take measures to ensure that equipment is scrupulously cleaned.

5.3 Work space areas shall be decontaminated between samples. It is recommended that liners be used as they may help in keeping work areas clean.

6.0 EQUIPMENT/APPARATUS

6.1 Stainless Steel Scoops

6.2 500ml, Pre-cleaned Glass Jar

6.3 SS mixing bowls

6.4 Labels

6.5 Protection gear; 1) gloves; 2) lab coat; 3) safety glasses

7.0 REAGENTS – N/A

8.0 PROCEDURE

8.1 Mixing in the Sample Container. It is imperative for any analysis to start with a representative sub-sample to insure reliable results. Reliable mixing can be achieved for the majority of soils and sediments by utilizing a scoopula and the sample container which the sample came in.

8.1.1 The analyst is to observe the consistency of the sample and mix throughout. Once mixed obtain a sub-sample from the entire sample.

8.1.2 Sub-samples (which are to be weighed) will be targeted to a "weight range" and not a predetermined specific weight (i.e.; 1 to 2 grams not 1.0 grams OR a range of 30 to 35 grams not 30.0 grams). By targeting a specific weight there could be a bias in the results due to particle size.

8.2 If the sample cannot be mixed in the sample container, the sample is to be transferred to a larger vessel, such as a quart SS mixing bowl.

8.2.1 Using a Scoop, breakup the sample into uniform sizes (approximately 4mm or less fragments) and homogenize in the bowl taking care to remove any artifacts such as vegetation or rocks.

8.2.2 After thoroughly mixing – obtain a representative portion of the sample for the preparation step.

8.3 Lab Duplicates or Matrix Spikes. When LCS/LCSD or MS/MSD's are to be analyzed, the analyst, after mixing, shall obtain both sub-samples from the same bowl.

9.0 CALCULATIONS – N/A

10.0 QUALITY ASSURANCE / QUALITY CONTROL – reference *SWL-GA-100*

11.0 WASTE DISPOSAL – reference *SWL-GA-114*

12.0 METHOD PERFORMANCE – N/A

13.0 REFERENCES

13.1 All QA/QC issues shall be referenced to SWL-GA-100, "*Laboratory Quality Assurance Program*".

13.2 Waste Disposal is governed by document SWL-GA-114, *"Hazardous Waste Management Plan"*

14.0 DEFINITIONS – N/A

SOUTHWEST LABORATORY OF OKLAHOMA, INC.

Standard Operating Procedure

Corrective Action

Document No.: SWL-GA-105

Rev No. / Date: Rev.0.0 — 05/08/97

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1.0 Scope and Application

This document defines the Corrective Action Program employed by Southwest Laboratory of Oklahoma, Inc. It delineates Corrective Action (CA) procedures including when CA is needed, how to document CA, and who is responsible for each step of the process. In addition, CA is distinguished from Non-conformance (NC). Procedures invoked for non-conformances are also described.

2.0 Summary

2.1.0 CA is the step-wise process by which the laboratory identifies deficiencies, defines cause, and corrects deficiencies. It is an ongoing process by which laboratory operations are improved. Any condition determined to adversely affect quality shall be subject to CA.

2.1.1 Guidelines for use of CA -- CA is required when a laboratory system is deemed out-of-control, thus affecting the usability or quality of that system. This includes analytical and non-analytical systems. Corrective action is also required when an uncontrolled deviation from standard operating procedure is made. CA may be initiated by any laboratory personnel but should be reviewed by immediate supervisor.

2.1.2 CA Process:

- Identify problem event and assess need for CA (out-of-control or non-conformance).
- Define cause, including root cause, of the out-of-control event.
- Define the need for change in the system , if necessary, and specify changes to be made.
- Create a plan of action with implementation schedule.
- Implement the changes, including change in SOP, QA Manual, etc., which the QA Department Manager must approve.
- Follow-up to ensure the change was made and to assess effectiveness of that change.
- Quarterly review of Closed CAs to search for trends.

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2.1.3 Documentation or CA (See Figure 2 for general Flow Diagram of Documentation Process):

- Describe events including root cause,
- Identify affected samples for analytical events,
- Describe resolution,
- Describe return to analytical control (proof of effectiveness),
- Describe follow-up to prevent recurrences, and
- Describe responsibility assignments.

2.1.4 Structure – See Figure 1

2.1.4.1 Structure of the CA process is divided into two major sections or series: G-Series; and O-, I-, and R-series. The G-series CAs are those CAs which universally affect the laboratory such as Safety, Document control, Waste management, Sample log-in, Administration, Client Services, Purchasing, and Computer Services. The O-, I-, and R-series are general laboratory sections: Organic, Inorganic, and Radiation Sections, respectively. Subcategories may be found under the second level/series to further differentiate between individual departments for filing and follow-up purposes (e.g., O-GCMS for Mass Spectrometry Department).

2.1.5 Responsibility – See Figure 1

2.1.5.1 Responsibility for each Series of the CA process is delineated in Figure 1. G-Series CAs are the responsibility of the Laboratory Director and/or Laboratory Manager. The O-, I-, and R-Series CAs are the responsibility of the Program Managers of their respective sections. Responsibility for subcategories of the O-, I-, and R-Series lies with the Section Supervisors. G-Series, as described above, encompass many different sections. Supervisors/managers of these sections will be responsible for their respective CAs (e.g., Client complaint CAs will be implemented through the Project Officers). In all instances, final follow-up and approval is the responsibility of the QA Department Manager. Portions of the CA process may be delegated, however, ultimate responsibility does remain with the parties described above.

2.2 Non-conformances:

2.2.1 Non-conformances are minor, non-trending deviations from standard procedure or QC acceptance criteria which may cause reduction of future quality if trends form.

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Events requiring non-conformance reports (NCRs) generally involve re-preparation or re-extraction of a sample. A NCR is documentation of action taken when a system non-conformance is identified. A system non-conformance is not considered an out-of-control event and does not affect an entire system (usability or quality). Trends of non-conformance, however, do constitute out-of-control events.

2.2.2 NCR Process:

- Identify and assess the problem event (out-of-control or non-conformance).
- Define cause of the non-conformance.
- Define the action to be taken.
- Quarterly review of NCRs to search for trends (discovery of trends will constitute the need for CA).

2.2.3 NCR documentation:

- Describe events,
- Identify affected samples for analytical events,
- Describe action to be taken,
- Describe responsibility assignments.

2.2.4 Responsibility – See Figure 1

2.1.5.1 NCRs are generally initiated at the department level and are the responsibility of the appropriate Section Supervisor or Program Manager. Actions to be taken, such as re-preparation or re-extraction, are the responsibility of the Section Supervisor performing the re-preparation or re-extraction. Section Supervisors will pull closed files and search for trends on a quarterly basis.

3.0 Health and Safety

N/A

4.0 Sample Preservation, Containers, Handling, and Storage

N/A

5.0 Interferences and Potential Problems

N/A

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6.0 Equipment/Apparatus

N/A

7.0 Reagents

N/A

8.0 Procedure

8.1 Identify problem event and assess need for CA or NC:

8.1.1 Identify problem using resources derived from training and SOPs. If at the bench level, notify Section Supervisor of possible out-of-control event. Initiator of action and Section Supervisor or appropriate Manager will use the following guidelines to distinguish between the need for the CA or NC process and determine the action to be taken. Guidelines follow:

8.1.1.1 Out-of-control events (See definitions – Section 14.0) require CA. Out-of-control events may include but are not limited to:

Statistical outliers (as defined in the Quality Assurance Manual);
Failure to follow standard operating procedure;
System failure;
Atypical instrument failure.

NOTE: If an out-of-control event is revealed, all actions associated with the out-of-control event must be stopped until the root cause is determined and CA is taken and documented.

8.1.1.2 Non-conformances require that a NCR be completed. Non-conforming events include minor, non-trending deviations from standard procedure or QC acceptance criteria which may cause reduction of future quality if trends form. Events requiring a NCR generally involve re-preparation or re-extraction of a sample. Refer to Section 8.2 for NC process procedure.

8.1.1.3 If it is decided that CA is required, then:

- Obtain a copy of the Corrective Action Report (CAR) form [See Attachment 1—Corrective Action Report];

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- Fill out the CAR beginning with out-of-control event description, date of occurrence, name of CA initiator, samples/systems affected, Series, and Department.
- Copy initiated CA to the QA Department's OPEN file. The original copy is kept at the Responsible Party's Level in the OPEN file.

8.1.2 Define cause, including root cause, of out-of-control event:

8.1.2.1 Defining cause may be as simple as describing the event (e.g., mis-dilution of a sample because of a misunderstanding of procedure). Root cause describes why the sample was mis-diluted (e.g., needs more thorough training to understand method requirements. Both cause and root cause must be detailed in the CAR in the appropriate section.

8.1.2.2 Root causes of out-of-control events may be separated into six general categories which include:

- Need for organization,
- Need for training,
- Need for discipline,
- Need for resources,
- Need for time, and
- Need for top management support.

8.1.2.3 The following documentation must be made on the CAR at this point:

- Detail the cause and root cause of the event on the CAR in the appropriately marked section.

8.1.3 Define the need for change in the system, if necessary, and specify the changes to be made:

8.1.3.1 After determination of cause and root cause, the Responsible Party must determine whether the event warrants change in a system, SOP, and/or QA manual. A change must be made if the system, SOP, etc., might perpetuate the out-of-control event.

8.1.3.2 All changes in procedure, accompanied by change in documentation such as SOPs, QA Manual, etc., must be approved by the QA

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Department Manager. The QA Department Manager is responsible for notifying other affected Sections of the change.

8.1.3.3 Documentation must be made on the CAR:

- Note on the CAR if changes must be made, the changes to be implemented, and the accompanying documentation changes that must be made;
- Signatures and dates documenting Responsible Party and QA Manager's approval must be made on the CAR at this time.

8.1.4 Plan of action with implementation schedule:

8.1.4.1 After changes to be made have been determined, a plan of action with schedule must be documented on the CAR.

8.1.4.2 Documentation must be made on the CAR:

- Step-by-step plan of action to implement the necessary change;
- Projected date of completion of the change.

8.1.5 Implementation:

8.1.5.1 Follow the plan of action, making every effort to meet the projected schedule of completion. Completion includes procedural change, documentation change, and re-training of affected personnel.

8.1.5.2 Upon completion of the change:

- the Responsible Party will sign and date the appropriate section of the CAR.

8.1.6 Follow-up to ensure change is made and to assess effectiveness of change:

8.1.6.1 Follow-up by Responsible Party includes audits of: effectiveness of the procedural change, documentation of the procedural change, and documentation of appropriate re-training.

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8.1.6.1.1 Documentation to be made on the CAR:

- Describe the follow-up procedure;
- Note the effectiveness of the change;
- Signature and date of the Responsible Party's follow-up completion.

8.1.6.1.2 Upon completion of follow-up:

- the Responsible Party will sign and date the appropriate section of the CAR;
- Original copy of the CAR will be transferred to the QA Department. Responsible Party will file a copy of the CAR in his/her CLOSED file.
- If follow-up reveals that the change was not effective, then the CA process must be repeated starting at section 8.1.2.

8.1.6.2. Follow-up by QA Department is the final step in the CA process. Follow-up includes audits of: effectiveness of the procedural change, documentation of the procedural change, and documentation of appropriate re-training.

8.1.6.3 Upon completion of QA follow-up:

- the QA Manager will sign and date the appropriate section of the CAR, documenting that follow-up was performed;
- Original copy of the CAR is transferred the QA Department CLOSED file.
- If follow-up reveals that the change was not effective, then the CA process must be repeated by the Responsible Party starting at section 8.1.2.

8.1.7 Quarterly review of Closed CAs to search for trends:

8.1.7.1 Both the QA Department and individual Laboratory Sections must quarterly review all Closed CARs to search for trends.

8.1.7.2 Each CAR reviewed must be initialed and dated to document that the review was performed.

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8.2 Non-conformance:

8.2.1 If a decision is made that a NC has occurred then:

8.2.1.1 Obtain a copy of the Non-conformance Report (NCR) form [See Attachment 2 – Non-conformance Report].

8.2.1.2 Fill out the NCR beginning with the department affected, Section Supervisor, the name of initiator, date of occurrence, description of the event, and the samples affected.

8.2.1.3 Define the cause of non-conformance:

8.2.1.3.1 Defining cause may be as simple as describing the event (e.g., erratic QC recovery caused by poor technique). Cause and action to be taken must be detailed in the NCR in the appropriate section. The "Cause" portion of the report will be signed by the Section Supervisor.

8.2.1.3.2 Copy the initiated NCR to the department performing the re-preparation or re-extraction. The original copy is kept by the initiator's Department.

8.2.1.3.3 If re-preparation or re-extraction is required, the NCR must be signed and dated upon completion by the Section Supervisor who is responsible for re-preparation/re-extraction.

8.2.2 Check for NC trends by periodic audit of files. If trends are noted, CA must be initiated. Section Supervisors or appropriate managers are responsible for NCR follow-up.

8.2.2.1 Documentation to be made on the NCR:

- Section Supervisor or appropriate manager will initial and date the NCRs which were reviewed upon completion.

9.0 Calculations

N/A

10.0 Quality Assurance/Quality Control

N/A

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11.0 Waste Disposal

N/A

12.0 Method Performance

N/A

13.0 References

Navy Installation Restoration Laboratory Quality Assurance Guide, February 1996.

Scope of Work for Analytical Sources, April 1996, Department of Energy. Page 50-51.

Alan J. Sayle, Management Audits, the assessment of quality management systems, 2nd Edition, ASQC Quality Press, Copyright 1988. Page 1-5.

14.0 Definitions

Corrective Action (CA) – the step-wise process by which laboratory deficiencies are identified, causes are defined, deficiencies corrected, and possibility or recurrence minimized and is an ongoing process by which laboratory operations are improved. Corrective action is required when a laboratory system is deemed out-of-control, thus affecting the usability or quality of that system. This includes analytical and non-analytical systems.

Corrective Action Report (CAR) – documentation of CA with emphasis on describing events, root causes, affected samples, resolutions including return to analytical control, and follow-up for recurrence prevention.

Closed File – a closed file is a file containing a CA that has been completed through final Follow-up by the QA Department.

Open File – the open file is a file containing a CA which is still in-process.

Out-of-control event – an event which is contrary to or fails to meet the objectives of the laboratory QA/QC plan, laboratory SOPs, or good laboratory practice thus

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affecting the usability or quality of a system. This includes analytical and non-analytical systems.

N/A – not applicable.

Non-conformance (NC) – non-conformances are minor, non-trending deviations from standard procedure or QC acceptance criteria which may cause reduction of future quality if trends form. Events requiring NC generally involve re-preparation or re-extraction of a sample.

Non-conformance Report (NCR) – documentation of action taken when a system non-conformance is identified. A system non-conformance is not considered an out-of-control event and does not affect an entire system (usability or quality). Trends of non-conformance, however, do constitute out-of-control events.

Responsible Party – the responsible party is the individual who is responsible for seeing the corrective action process through to closure. See Section 2.1.5 and Figure 1 for details.

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FIGURE

Corrective Action Report (CAR) Structure

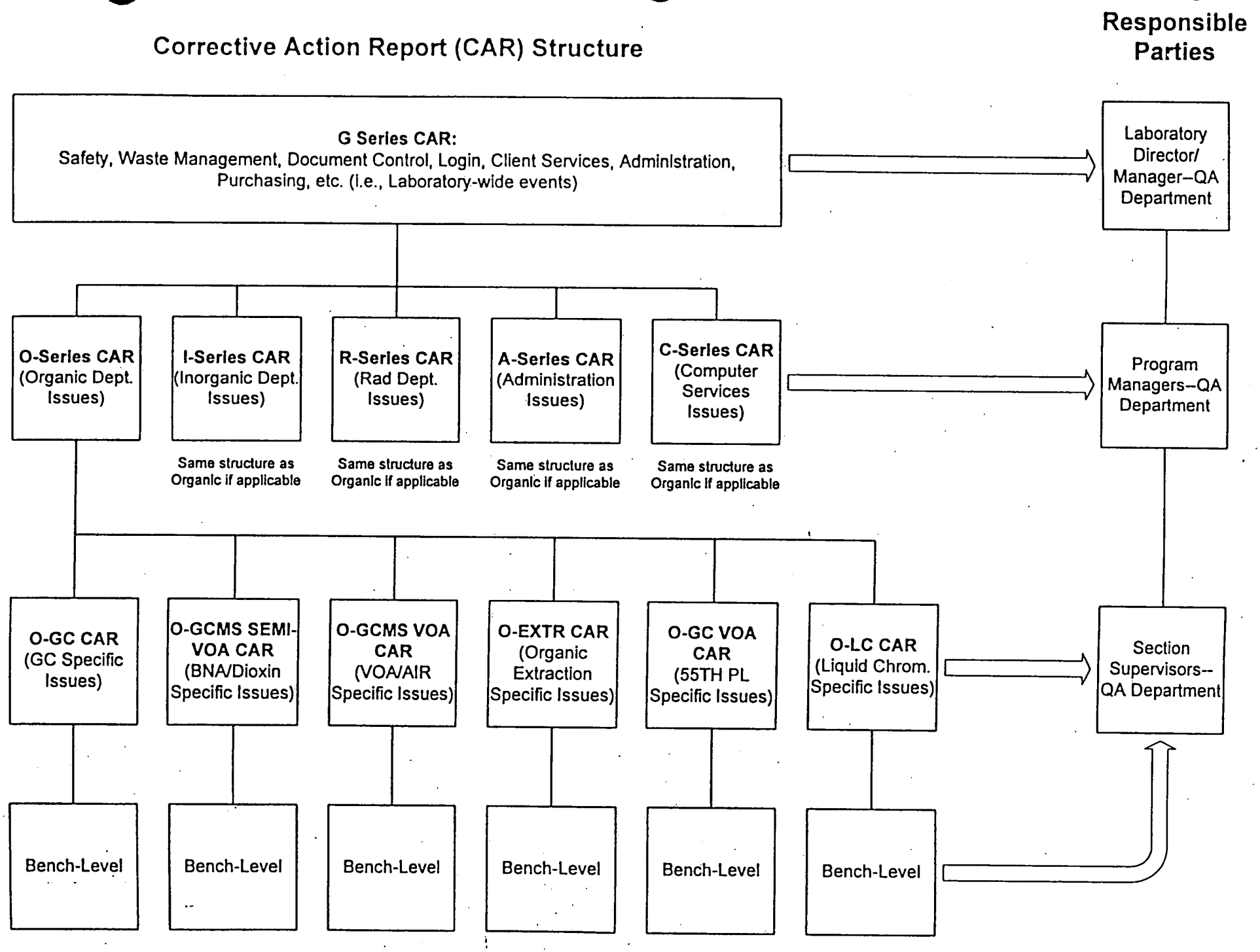

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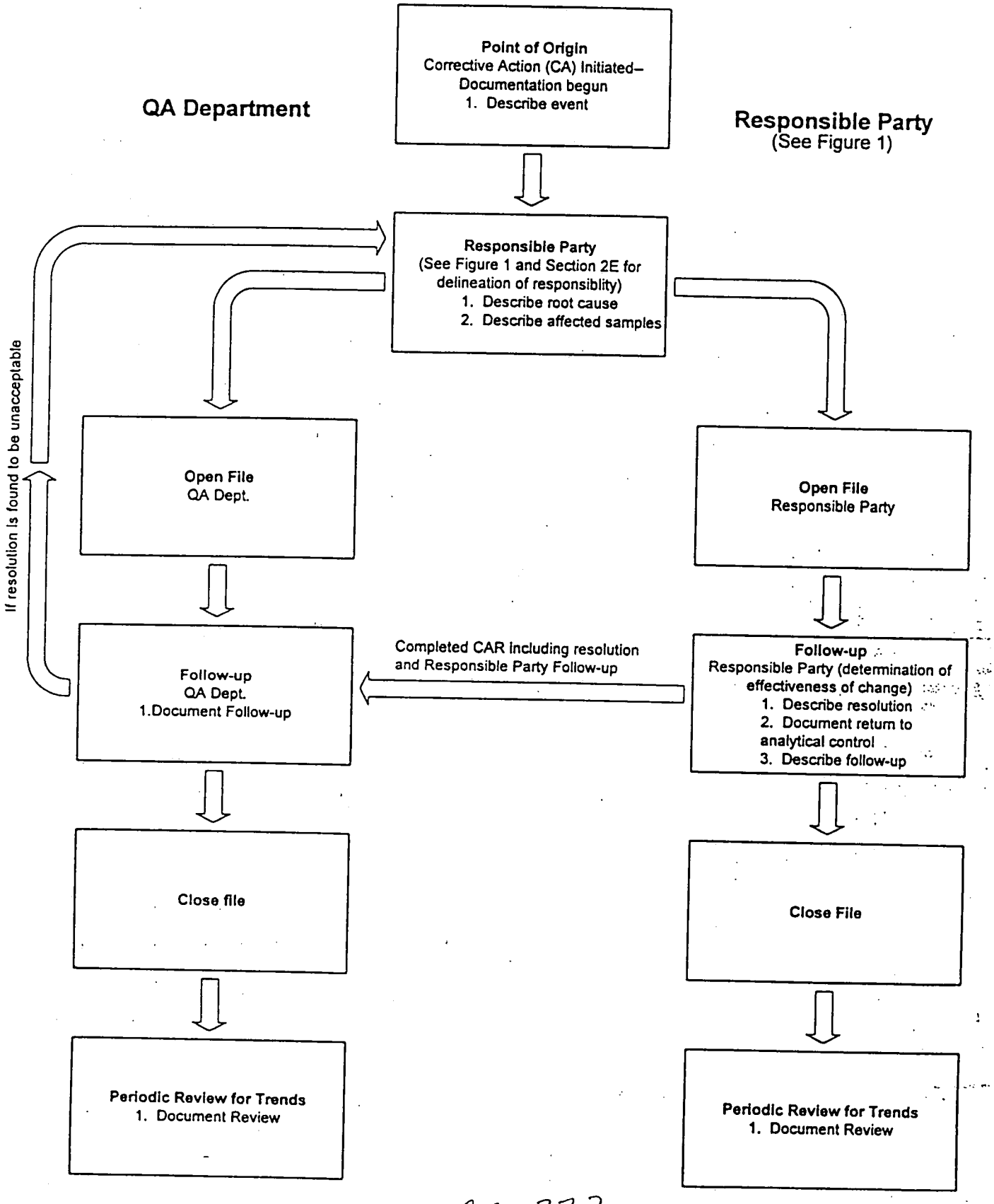
Figure 2:
Documentation Trail



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Attachment 1: **CORRECTIVE ACTION REPORT** QA DEPT.

SWL-GA-105

Series (circle one): G-, O-, I-, R-Series
Department:

8.1.1 DESCRIPTION OF OUT-OF-CONTROL EVENT

Initiated By: Date:

Responsible Party: Date:

Date of Occurrence:

Description of Event:

System and/or Samples Affected:

8.1.2 CAUSE/ROOT CAUSE

Description:

8.1.3 SYSTEM CHANGES REQUIRED [Yes/ No] (circle one)

Procedural Change Description:

Documentation Change Description:

Attachment 1: **CORRECTIVE ACTION REPORT** QA DEPT.

SWL-GA-105

SIGNATURES:

Responsible Party: Date:

QA Manager: Date:

8.1.4 PLAN OF ACTION

Step-By-Step Description:

Projected Date of Completion:

8.1.5 IMPLEMENTATION

SIGNATURE:

Completion Date: Responsible Party:

8.1.6.1 DEPARTMENTAL FOLLOW-UP

Description of Follow-up:

Was Action Effective (Yes/No):

SIGNATURE:

Responsible Party: Date:

8.1.6.2 QA FOLLOW-UP

Description of Follow-up:

Was Action Effective (Yes/No):

SIGNATURE:

QA Manager: Date:

Attachment 2: **NON-CONFORMANCE REPORT** QA DEPT.

SWL-GA-105

Department:

8.2.1.2 DESCRIPTION OF NON-CONFORMANCE

Initiated By: Date:

Section Supervisor: Date:

Date of Occurrence:

Description of Event:

System and/or Episode of Samples Affected (Client/Test code/Matrix if applicable):

8.2.1.3 CAUSE

Description:

SIGNATURE:

Section Supervisor: Date:

8.2.1.3 ACTION TO BE TAKEN

Re-preparation/ Re-extraction Required (Yes/ No)? Date Needed:

Within hold time: (Yes/No)? Sample Receipt Date (if applicable):

Description (with dates):

SIGNATURE:

Completion Date: Section Supervisor:

SOUTHWEST LABORATORY OF OKLAHOMA, INC.

1700 W. ALBANY• BROKEN ARROW, OK 74012 •OFFICE (918) 251-2858 •FAX (918) 251-2599

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Request for New or Revised Procedure, SWL-GA-101

SWL SOP Rev. # 1.0 - 05/14/97; Form ID: GA101-DOCREQ-F: Location/File ID: F:\SOP\GA\101-ATT5

REVISED (includes Forms/Logs) Submitted By: CAH Date: 6/5/97

Document Title: Corrective Action

Document No.: SWL- G A - 1 0 5 Rev. No. 0.0 — 0 5 1 0 8 1 9 7

Reference: Page 1 of 10 Section: 2.0; Subpart: 2.1.1

Attachment No. _____ — Title: ~~Add verbage re~~ CAH 6/5/97

Brief Description (need for revision): Add Verbage regarding the initiation of CARs in response to Audit Findings (see Attached)

Approved: CAH Dated: 6/5/97 Denied: ☐ PM/SS Initials: _____
Program Manager/Section Supervisor (see attached explanation)

NEW (Procedural Document) Submitted By: _____ Date: _____

Justification for New Procedure: _____

Approved: _____ Dated: _____ Denied: ☐ PM/SS Initials: _____
Program Manager/Section Supervisor (see attached explanation)

Author: _____ Assigned By: _____ Date: _____

Review/Approval by Manager/Supervisor

Draft No. ___ Author's Initials: ____ Date: ____
Reviewed: ☐ Revise: ☐ Approved: ☐
PM/SS Initials ____ Date ____ (revise as marked)

Draft No. ___ Author's Initials: ____ Date: ____
Reviewed: ☐ Revise: ☐ Approved: ☐
PM/SS Initials ____ Date ____ (revise as marked)

Draft No. ___ Author's Initials: ____ Date: ____
Reviewed: ☐ Revise: ☐ Approved: ☐
PM/SS Initials ____ Date ____ (revise as marked)

Final Draft # ___ Routed to QA Dept.: Date _____ Initials ____

Review/Approval by QA/QC Officer

Draft No. ___ Author's Initials: ____ Date: ____
Reviewed: ☐ Revise: ☐ Approved: ☐
QA/QC Initials ____ Date ____ (revise as marked)

Draft No. ___ Author's Initials: ____ Date: ____
Reviewed: ☐ Revise: ☐ Approved: ☐
QA/QC Initials ____ Date ____ (revise as marked)

Draft No. ___ Author's Initials: ____ Date: ____
Reviewed: ☐ Revise: ☐ Approved: ☐
QA/QC Initials ____ Date ____ (revise as marked)

QA-Formal Document Completed: _____ Initials: _____
Date Rec'd by Document Control: _____ Initials: _____

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4 June 97
7:58

Rob,

Minutes ago I recieved the SOP for Corrective Actions, I was told that it is needed today. ASAP - hopefully by noon.

There is no mention of CA generated from audits outside SWL. I think that as a reader, an auditee, I would like to see such a section. Auditees believe they contribute to an quality. In this SOP we tell them we think they contribute and we respect their input. The Huge success of EOE and Hazwrap to this CA Approach. and the failure of Waste where we didn't have such and approach argue for such a section. I recommend

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Add 2nd ¶ to Section 2.1.1

A unique "out of control" situation occurs when a audit, either external or internal, occurs and positive and negative findings, observations, deficiencies etc are found. These third party specific comments are grouped and form a special set of CA's. They indicate strong or weak areas of performance or perhaps only communication. After initiation of the CA audit set all other steps of this SOP are followed. Depending on the type of audit and its requirements the initial CAR set might be supplied to the auditee, as well as follow ups as appropriate.