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Work Plan
for
Phase II Ground Water RCRA Facility Investigation Solid Waste Management Unit 30 (Landfarm) Naval Surface Warfare Center (NSWC)
Crane, Indiana

Southern Division
Naval Facilities Engineering Command
Contract Number N62467-94-D-0888
Contract Task Order 0019

August 1999

049910/P

WORK PLAN
FOR
PHASE II GROUND WATER RCRA FACILITY INVESTIGATION
SOLID WASTE MANAGEMENT UNIT 30
(LANDFARM)

NAVAL SURFACE WARFARE CENTER (NSWC)
CRANE, INDIANA

COMPREHENSIVE LONG-TERM
ENVIRONMENTAL ACTION NAVY (CLEAN) CONTRACT

Submitted to:
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Naval Facilities Engineering Command
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TABLE OF CONTENTS

SECTION **PAGE**

ACRONYMS, INITIALISMS, AND ABBREVIATIONS 3

1.0 INTRODUCTION 1-1
1.1 OBJECTIVES AND SCOPE 1-1
1.2 SITE BACKGROUND AND DESCRIPTION 1-2
1.2.1 Landfarm History 1-2
1.2.2 Site Topography 1-6
1.2.3 Natural and Manmade Features 1-7
1.3 PREVIOUS SITE INVESTIGATIONS 1-7
1.3.1 Site Geology 1-11
1.3.2 Site Hydrogeology 1-11
1.4 SAMPLING PLAN RATIONALE 1-13
1.5 PROJECT MANAGEMENT AND ORGANIZATION 1-14
1.5.1 Management Responsibilities 1-15
1.5.2 Quality Assurance Responsibilities 1-18
1.5.3 Laboratory Responsibilities 1-19
1.5.4 Field Responsibilities 1-19
1.6 COMMUNITY RELATIONS 1-20
1.7 DELIVERABLES AND DATA MANAGEMENT 1-21
1.8 PROJECT SCHEDULE 1-21

2.0 SAMPLING AND ANALYSIS PLAN 2-1
2.1 FIELD OPERATIONS SUMMARY 2-1
2.2 MOBILIZATION / DEMOBILIZATION 2-1
2.3 GROUND WATER SAMPLING 2-3
2.3.1 Water-Level Measurements and Ground Water Monitoring Well Evaluations 2-3
2.3.2 Monitoring Well Development and Purging 2-3
2.3.3 Sampling 2-5
2.4 FIELD MEASUREMENTS 2-6
2.5 DECONTAMINATION 2-13
2.6 INVESTIGATION DERIVED WASTE (IDW) 2-14

3.0 SAMPLE HANDLING PROCEDURES 3-1
3.1 SAMPLE IDENTIFICATION SYSTEM 3-1
3.2 SAMPLE PACKAGING AND SHIPPING 3-1
3.3 SAMPLE CUSTODY 3-1
3.4 QUALITY CONTROL SAMPLES 3-2
3.5 RECORD KEEPING 3-3

REFERENCES R-1

TABLE OF CONTENTS (Continued)

APPENDICES

A GEOLOGIC LOGS AND WELL CONSTRUCTION DIAGRAMS
B FIELD FORMS
C STANDARD OPERATING PROCEDURES
D HEALTH AND SAFETY PLAN
E HACH TEST KIT INFORMATION
F SLUDGE SAMPLING ADDENDUM

TABLES

NUMBER PAGE

1-1A Historical Analytical Results from Sludge Samples, Anacon Analytical and Consulting Laboratories, Inc. 1-9
1-1B Historical Analytical Results from Sludge Samples, Heritage Laboratories, Inc. 1-10
2-1 Quarterly Chemical Analysis Summary 2-2
2-2 Summary of Sampling Activities 2-7
2-3 Summary of Sample Analyses, Container Types and Volumes, Preservative Requirements, and Holding Times 2-12

FIGURES

NUMBER PAGE

1-1 Location Map 1-3
1-2 Landfarm Location 1-5
1-3 Site Map 1-8
1-4 Generalized Geologic Cross-Section A-A' 1-12
1-5 Project Organization Chart 1-16

ACRONYMS, INITIALISMS, AND ABBREVIATIONS

CAP	Contamination Assessment Plan
CAR	Contamination Assessment Report
CFR	Code of Federal Regulations
CFR	Code of Federal Register
CLEAN	Comprehensive Long-term Environmental Action Navy
CMS	Corrective Measures Study
COE	U.S. Army Corps of Engineers
CTO	Contract Task Order
DOD	U.S. Department of Defense
EPA	U.S. Environmental Protection Agency
FID	Flame Ionization Detector
FOL	Field Operations Leader
FSP	Field Sampling Plan
ftASL	Feet Above Mean Sea Level
HASP	Health and Safety Plan
HSWA	Hazardous and Solid Waste Amendment of 1984
IDW	Investigation-Derived Waste
IWPT	Industrial Waste Treatment Plant
MCL	Maximum Contaminant Level
MSL	Mean Sea Level
NAD	Naval Ammunition Depot
NEESA	Naval Energy and Environment Support Activity
NAVFAC	Naval Facilities Engineering Command
NSWC	Naval Surface Warfare Center
OVA	Organic Vapor Analyzer
PCB	Polychlorinated Biphenyl
PID	Photoionization Detector
POC	Point of Contact
PPE	Personal Protective Equipment
PVC	Polyvinyl Chloride
QAM	Quality Assurance Manager
QAPP	Quality Assurance Project Plan
QA/QC	Quality Assurance and Quality Control

RAB	Restoration Advisory Board
RCRA	Resource Conservation and Recovery Act
RFI	RCRA Facility Investigation
RPM	Remediation Project Manager
SOP	Standard Operating Procedure
SOUTHDIV	Southern Division Naval Facilities Engineering Command
SSO	Site Safety Officer
SWMU	Solid Waste Management Unit
TKN	Total Kjeldahl Nitrogen
TOM	Task Order Manager
TtNUS	Tetra Tech NUS, Inc.
USCS	Unified Soil Classification System
VOC	Volatile Organic Compound
USACEWES	U.S. Army Corps of Engineers Waterways Experiment Station

1.0 INTRODUCTION

(Note: Brown & Root Environmental was purchased on January 1, 1998 and became Tetra Tech NUS, Inc. [TtNUS]). TtNUS has prepared this Work Plan for Solid Waste Management Unit (SWMU) 30, Naval Surface Warfare Center (NSWC) Crane, Indiana. This Work Plan was prepared for the U.S. Navy (Navy) Southern Division (SOUTHDIV) Naval Facilities Engineering Command (NAVFAC) under Contract Task Order (CTO) 0019, for the Comprehensive Long-Term Environmental Action Navy (CLEAN III) Contract Number N62467-94-D-0888.

Although this Work Plan was prepared as a stand-alone document, it should be used with the Quality Assurance Project Plan (QAPP) and associated standard operating procedures (SOPs). This Work Plan is an outgrowth of a January 1995 U.S. Army Corps of Engineers Waterways Experiment Station (USACEWES) Work Plan completed for SWMU 30. It is designed to provide direction for field and laboratory staff to ensure that specific procedures are properly implemented in a safe and scientifically defensible manner. It is also designed to provide a logical rationale for the investigative approach based on known regional and site-specific background information. This Work Plan is comprised of an Introduction (Section 1) that provides background information, presents the project rationale, and identifies project management issues; a Sampling and Analysis Plan (Section 2) that provides direction to the field staff; and an Environmental Sample Handling Procedures section (Section 3) that also provides direction to the field staff. Four appendices are attached to this Work Plan: Appendix A, Geologic Logs and Well Construction Diagrams (site-specific data from previous investigations); Appendix B, Field Forms; Appendix C, Standard Operating Procedures; and Appendix D, Health and Safety Plan.

1.1 OBJECTIVES AND SCOPE

The objective of this project is to determine the possible presence or absence and extent of ground water contamination at SWMU 30. SWMU 30 is commonly referred to as the "Landfarm". The work is being conducted to meet the requirements of a Phase II Ground Water Resource Conservation and Recovery Act (RCRA) Facility Investigation (RFI).

Seven ground water monitoring wells were installed around the perimeter of SWMU 30 (the Landfarm) during a previous USACEWES investigation, but these wells have not been sampled to date. The scope of the current investigation involves sampling the seven on-site ground water monitoring wells five times over a 15-month duration, laboratory analysis, analytical data validation, data management, and reporting.

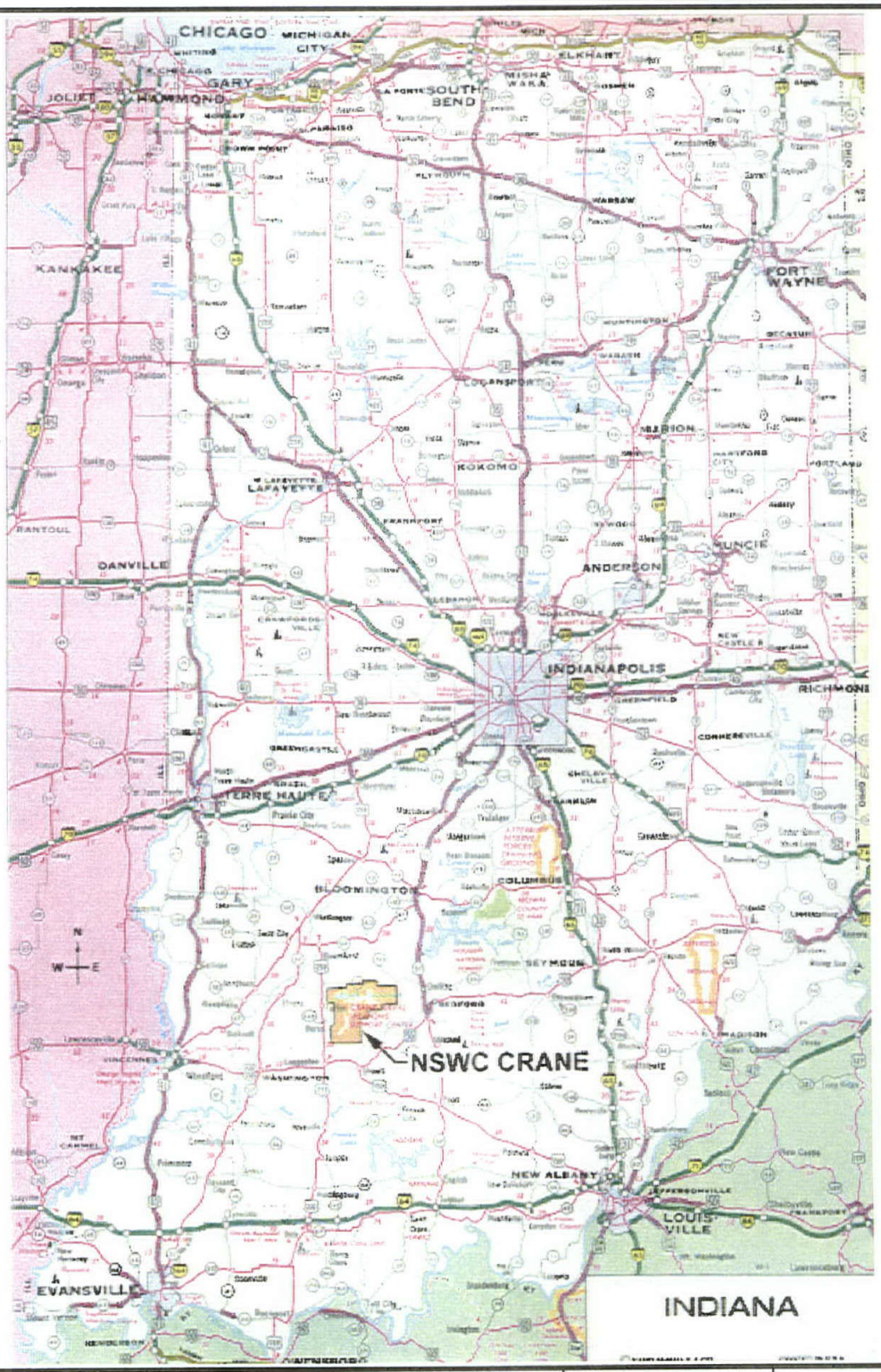
1.2 SITE BACKGROUND AND DESCRIPTION

The NSWC Crane is located in Crane, Indiana, approximately 75 miles southwest of Indianapolis and 71 miles northwest of Louisville, Kentucky (Figure 1-1). The facility encompasses more than 100 square miles (64,463 acres) (Figure 1-2). NSWC Crane provides material, technical, and logistical support to the Navy for equipment, weapons systems, and expendable and nonexpendable ordnance items. The facility was opened in 1941 as the Naval Ammunition Depot (NAD), Burns City, to serve as an inland munitions production and storage center. In 1943, the name was changed to NAD Crane in honor of Commodore William Montgomery Crane, the first chief of the Navy's Bureau of Ordnance. The name changed again in 1975 to Naval Weapons Support Center to reflect the facility's growing involvement in high-technology weapons system. In 1977, the Secretary of Defense decided to combine all conventional ammunition acquisition under the responsibility of a single service. The ammunition production and storage function was given to the Army, and the Crane Army Ammunition Activity was established as a Crane tenant to accomplish this task for Naval ammunition. In 1992, based on changing missions and alignment, the name was changed again to the Naval Surface Warfare Center Crane. The Army has assumed ordnance production, storage, and related responsibilities under the single-service management directive. All environmental activities on the installation, including permitting activities, remain the responsibility of the Navy. Although ordnance production and storage still resides on Base, Crane serves a modern and sophisticated Navy as a recognized leader in diverse and highly technical product lines, such as microwave devices, acoustic sensors, small arms, microelectronic technology, and more. The Army currently exists as a Tenant Activity on the Base.

The Landfarm (SWMU 30) is located in the west central part of the NSWC Crane, approximately 1 mile south of Burns City, Indiana (Figure 1-2) at the northeastern intersection of Highway 161 and Highway 200. Aboveground, high-voltage power lines cross the site from east to west.

1.2.1 Landfarm History

Within the facility, NSWC Crane operates a wastewater treatment plant that generates sludges. NSWC Crane has historically used land application for the disposal of sludge from its main on-site sewage treatment plant. These sludges are from the processing of domestic and process wastewater sources. Process sources included wastewater from metal finishing operations; surface coating operations; the loading, assembly, and packing of ordnance; water treatment plant backwash; boiler blowdowns; and industrial laundry.



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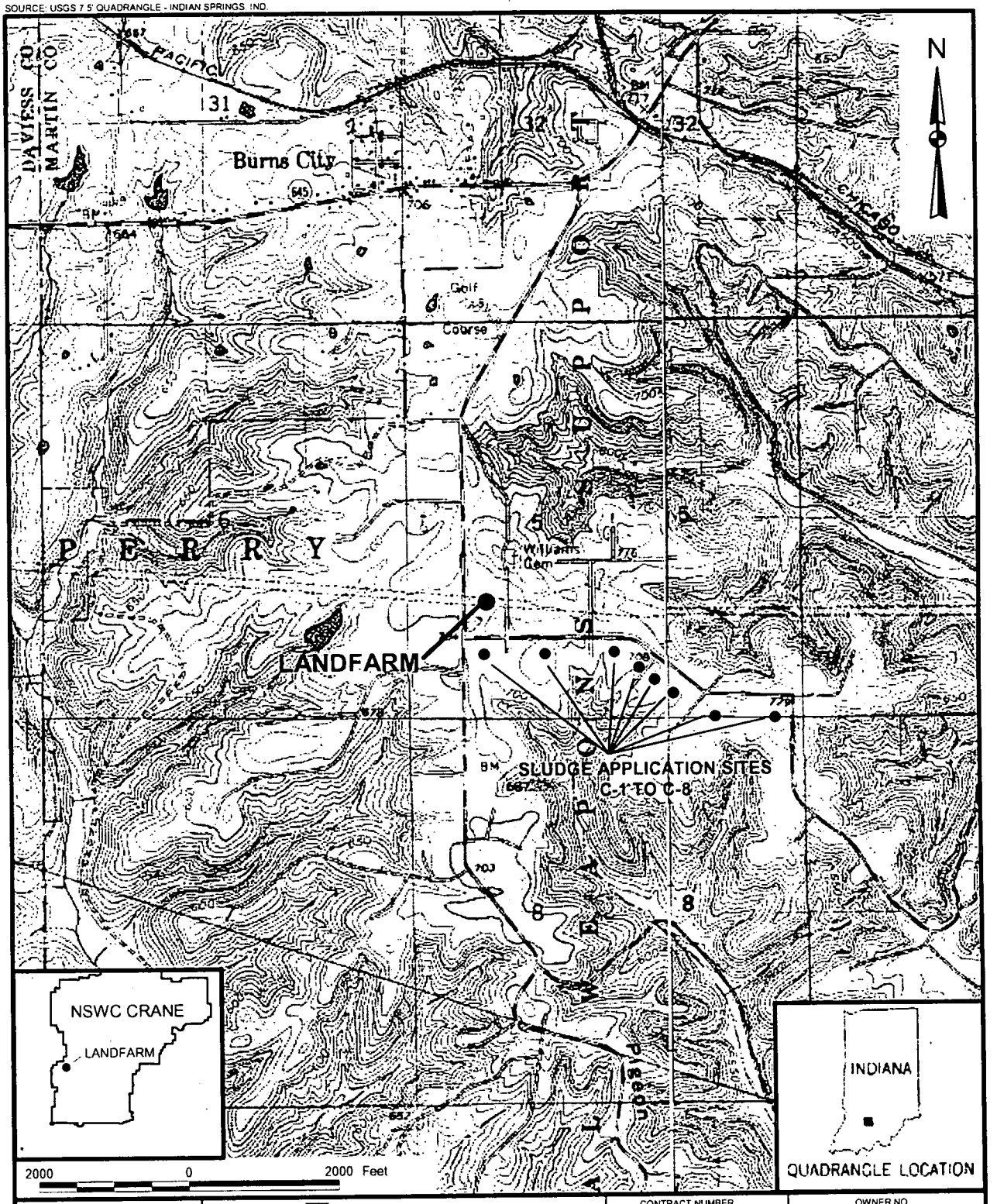
CHECKED BY —	DATE —	LOCATION MAP NAVAL SURFACE WARFARE CENTER CRANE, INDIANA	APPROVED BY — / DATE —
COST/SCHEDULE-AREA			APPROVED BY — / DATE —
SCALE AS NOTED			DRAWING NO FIGURE 1-1 / REV 0

P:\GIS\NSWC_CRANE\CRANE.APR 29-JAN-99 DNP LOCATION MAP LAYOUT

SOURCE: USGS 7.5' QUADRANGLE - INDIAN SPRINGS, IND.



DRAWN BY D. PERRY — DATE 29-JAN-99	Tetra Tech NUS, Inc.	CONTRACT NUMBER —	OWNER NO —
CHECKED BY — DATE —	LOCATION OF LANDFARM NAVAL SURFACE WARFARE CENTER CRANE, INDIANA	APPROVED BY — DATE —	
COST/SCHEDULE-AREA		APPROVED BY — DATE —	
SCALE AS NOTED		DRAWING NO. FIGURE 1-2	REV 0

P:\GIS\NSWC_CRANE\CRANE.APR 29-JAN-99 DNP LOCATION OF LANDFARM LAYOUT

In October 1980, NSWC Crane filed a RCRA Section 3010 Notification and a Part A (interim status) permit application to operate as a treatment, storage, or disposal facility. The application for the Part A permit was approved, and the facility was allowed to operate as though it had a permit. In December 1983, NSWC Crane applied for and obtained a sludge application permit to spray sludges from its wastewater treatment plant along approximately 18 miles of roadside near the western boundary of the facility. On April 13, 1988, NSWC Crane was issued a permit (effective May 6, 1988) to apply sludge to a 2.5-acre site (the Landfarm, Figure 1-2) located near an existing sanitary landfill. Sludges from the on-site wastewater treatment facility were applied to this site from November 16, 1988 through March 1995. In June of 1994, NSWC Crane began applying sludges to eight new land-application-permitted sites located south and southeast of the Landfarm (see Figure 1-2).

On December 20, 1989, a joint federal and state RCRA storage permit was issued to the Navy. The federal portion of the permit established the Hazardous and Solid Waste Amendments (HSWA) Corrective Actions Requirements and Compliance Schedules (RCRA 3004). The compliance schedule obligated NSWC Crane to perform an RFI at each on-site SWMU. A corrective measures study (CMS) and implementation of corrective measures are to be initiated if contamination is identified at a SWMU during the RFI process. The State of Indiana obtained pre-HSWA authorization and issued the Navy the state portion of the permit.

In 1992, the U.S. Environmental Protection Agency (U.S. EPA) became concerned that the sludges from the on-site treatment plant should be characterized as F006 waste (wastewater treatment sludges from electroplating operations). U.S. EPA contended that wastewaters discharging from the electroplating shop pretreatment plants (Buildings 38 and 3064) were mixing with other wastewaters in the sewer system prior to arrival at the main sewage treatment plant. In response to U.S. EPA concerns, NSWC Crane implemented measures to prevent the discharge of wastes from the plating shops and resulting mixing with other wastewaters. In 1995, U.S. EPA renewed and modified the NSWC Crane permit to include the Landfarm as SWMU 30 to settle the enforcement issue concerning F006 and to modify the HSWA corrective action requirements and schedule. U.S. EPA had already required NSWC Crane to conduct an RFI at the Landfarm as part of the 1989 permit to determine if the previous application of sludges possibly contaminated with plating wastes had adversely affected the shallow ground water regime.

1.2.2 Site Topography

The Landfarm is approximately 2.5 acres in area. The site is relatively topographically flat. Ground elevations for the monitoring wells at the Landfarm are currently not available. The tops of monitoring

well casings at the Landfarm varied in elevation from 705.03 feet above mean sea level (ftASL) at location 30-07 to 719.11 ftASL at location 30-03 (Figure 1-3).

A hill north-northeast of the site has an elevation of 741.1 ftASL. This hill slopes gently toward the Landfarm. The elevation at the intersection of Highways 200 and 161 is 705.7 ftASL. Therefore, the topography across the Landfarm slopes southwest at an average gradient of approximately 0.02 feet per feet (ft/ft).

1.2.3 Natural and Manmade Features

The Landfarm is located directly beneath two high-voltage power lines (Figure 1-3). The site is lightly vegetated and is bound on the north and south by forested areas. The Landfarm is bound to the west by Highway 161 and to the east by an unnamed road. William's Cemetery is located on top of the hill north-northeast of the site. The Landfarm is located northeast of the intersection of Highways 161 and 200.

1.3 PREVIOUS SITE INVESTIGATIONS

NSWC Crane subcontracted to the USACEWES the installation of seven ground water monitoring wells around the Landfarm. The USACEWES installed four downgradient, one cross-gradient, and two upgradient ground water monitoring wells at this site in the Fall of 1993 (Figure 1-3). These seven ground water monitoring wells were designated 30-1 through 30-7. The original well logs and reformatted well logs and well construction diagrams are presented in Appendix A.

Ground water samples have not been obtained from these seven wells. However, NSWC Crane has conducted quarterly analysis of the sludge since 1988. Although analytical data has been collected for the sludge since 1988, only data from 1990 through 1992 were available for the completion of this Work Plan.

Sludge samples were obtained before the sludge was loaded into the spreader truck. The available quarterly analyses of sludges destined for the Landfarm were performed by Anacon Laboratories in 1990, 1991, and 1992 (Table 1-1A), and by Heritage Laboratories in 1992 (Table 1-1B). The Anacon analytical data was for a limited analysis that included a modified metals analysis, polychlorinated biphenyls (PCBs), ammonia, nitrate, total Kjeldahl nitrogen (TKN), and phosphorus. The Heritage analyses were limited to include only a modified metals analysis, cyanide, toxicity characteristic leaching procedure (TCLP) analysis for volatile and semivolatile organic compounds, sulfide, ignitability, and pH. The available historic sludge analytical results (1990 through 1992) are presented in Tables 1-1A and 1-1B.

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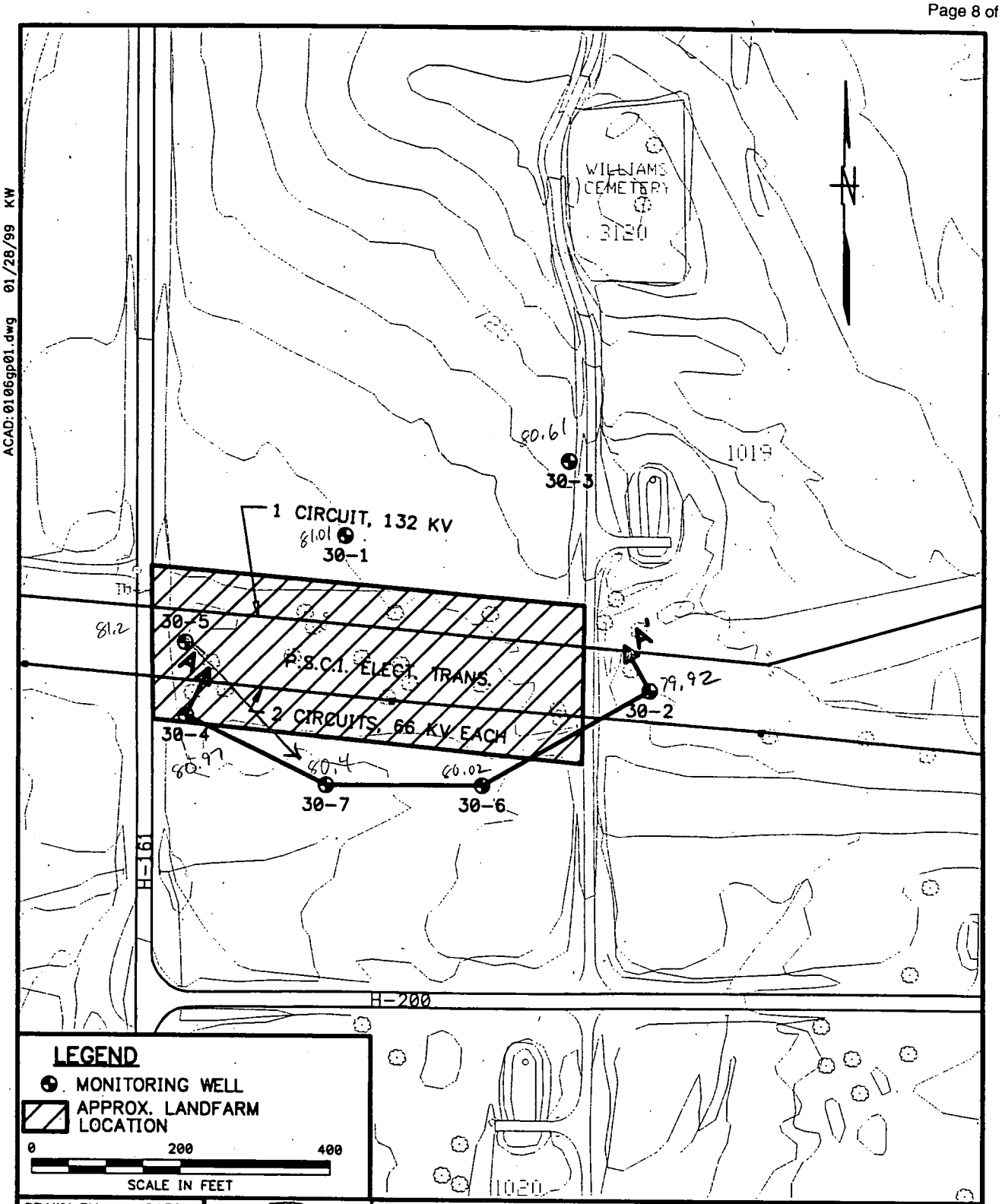

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SCALE AS NOTED			DRAWING NO. FIGURE 1-3 REV. 0

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TABLE 1-1A

HISTORICAL ANALYTICAL RESULTS FROM SLUDGE SAMPLES
ANACON ANALYTICAL AND CONSULTING LABORATORIES, INC.

Analyte	1/11/90	1/11/90	4/3/91	4/3/91	9/17/90	9/17/90	12/27/90	12/27/90	4/3/91	4/3/91	4/17/91	4/17/91	6/26/91	6/26/91	10/3/91	10/3/91	12/19/91	12/19/91	1/9/92	4/4/92	4/4/92	7/2/92	7/2/92
	mg/L	mg/kg	mg/L	mg/kg	mg/L	mg/kg	mg/L	mg/kg	mg/L	mg/kg	mg/L	mg/kg	mg/L	mg/kg	mg/L	mg/kg	mg/L	mg/kg	mg/L	mg/kg	mg/L	mg/kg	mg/L
Ammonia	2.7	76													10.85		3.6		1.84	2.13	68.71	29.4	1838
PCB	<0.1	<1													<0.1		<0.01		<0.01	<0.01	<1.0	<0.001	<0.1
TKN																				410	13226	12.6	788
Phosphorus	62	1750													16.5		0.44		0.31	256	8259	6.03	377
Total Nitrogen	170	4760													18.35				18.3				
Nitrate	8.2	230													n/a		17		9.1	<0.1	<5		
% Total Solids	3.53	n/a	0.99	n/a	1.16	n/a	0.93	n/a	0.99	n/a	0.56	n/a	2.2	n/a	0.87	n/a	0.03 to 0.59		0.21	0.32	n/a	1.4 to 1.6	n/a
Cadmium			0.1583	15.45	0.5	43.1	0.1	10.8	0.153	15.45	0.215	38.5	0.004	0.19	0.169	19.4	14	23.7		0.036	11.3	0.368	26.3
Chromium			1.25	126	2.01	173			1.25	126	1.222	219	0.009	0.4	0.46	52.9	0.58	98		0.16	50		
Copper			4.25	429	12.7	1096	4.15	446	4.25	429	5.167	924	0.14	6.24	2.05	235	2.47	417		0.63	197	3.02	216
Lead			0.91	92	8.33	718	<0.50	<0.50	0.91	92	0.006	1.074	0.003	0.014	<0.10	<1.0	<0.05	<5.0		<0.05	<0.5	<0.05	<0.50
Nickel			0.63	64	1.33	115	0.52	55.9	0.63	64	0.645	115	0.034	1.53	0.35	40.25	0.27	45.6		0.48	26.3	0.45	32.1
Silver			0.05	<1	<0.05	<2.5			0.05	<1	<0.05	<1.00	<0.05	<0.5	0.45	<1.0	4.04	682		<0.0001	26.3		
Zinc			8.3	838	15.15	1306	6.88	740	8.3	838	9.597	1719	0.293	13.2	5.63	647	0.09	15.2		1.41	438	7.99	571
Mercury			<0.0001	<0.01					<0.0001	<0.01					<0.01	<1	0.008	1.35					
Potassium					30	2586	25	2688			30.4	3170			16.6	1909	30.4	5152		18.4	5750	56.7	4050

Notes:

- mg/L milligrams per liter
- mg/kg milligrams per kilogram
- PCB polychlorinated biphenyl
- TKN total Kjeldahl nitrogen
- < less than
- n/a not available

TABLE 1-1B

HISTORICAL ANALYTICAL RESULTS FROM SLUDGE SAMPLES HERITAGE LABORATORIES, INC.

Analyte	5/18/92 mg/L	5/26/92 mg/L	6/17/92 mg/kg
Percent Total Solids	51		
Barium	0.78	0.64	
Cadmium	430	<0.02	
Chromium	41	<0.05	2600
Lead	<0.2	<0.20	
Silver	<0.04	<0.04	
Arsenic		<0.005	
Selenium		<0.005	
Mercury		<0.0050	
Copper		3.7	
Zinc		0.81	
TCLP Analysis			
1,4-Dichlorobenzene		<50 ug/L	
2,4-Dinotrotoluene		<50 ug/L	
Hexachlorobenzene		<50 ug/L	
Hexachlorobutadiene		<50 ug/L	
Hexachloroethane		<50 ug/L	
Nitrobenzene		<50 ug/L	
Pyridine		<250 ug/L	
2-Methyl Phenol		<50 ug/L	
3-Methyl Phenol		<50 ug/L	
3-Methyl Phenol		<50 ug/L	
Pentachlorophenol		<250 ug/L	
2,4,5-Trichlorophenol		<50 ug/L	
2,4,6-Trichlorophenol		<50 ug/L	
Benzene		<50 ug/L	
Carbon Tetrachloride		<50 ug/L	
Chlorobenzene		<50 ug/L	
1,2-Dichloroethane		<50 ug/L	
1,1-Dichloroethylene		<50 ug/L	
Methyl Ethyl Ketone		<100 ug/L	
Tetrachloroethylene		<50 ug/L	
Trichloroethylene		<50 ug/L	
Vinyl Chloride		<100 ug/L	
pH		4.9	
Total Cyanide		<0.1 mg/kg	1600
Sulfide		<10 mg/kg	

Notes:

mg/L	milligrams per liter
mg/kg	milligrams per kilogram
µg/L	micrograms per liter

1.3.1 Site Geology

The soils underlying the site were observed to be silty clay and ranged in thickness from 12 to 16 feet (USACEWES, 1995). These soils were classified under the Unified Soil Classification System (USCS) as being silt to silty clay (ML to CL). The soils were dry to moist and commonly were slightly to moderately plastic.

The rock underlying the Landfarm is Pennsylvanian in age and consists of 0 to 14 feet of interbedded soft clay, gray to black shale, coal, and sandstone (USACEWES, 1995). These strata overlie a massively bedded, predominantly brown, noncalcareous, quartzitic, fine- to medium-grained sandstone that becomes coarser and more friable with depth. An upper interbedded shale and sandstone sequence that was observed in most borings was absent in the easternmost boring, (30-2). The sandstone was observed to be jointed along bedding planes. An east-west cross section was constructed to show the geology across the site and is presented as Figure 1-4. The location of the cross-section line is presented on Figure 1-3.

1.3.2 Site Hydrogeology

Based on information collected by the USACEWES, the first indication of saturated subsurface conditions was noted within the sandstone bedrock at between 30 and 42 feet below ground level (USACEWES, 1995). This shallow ground water regime is suspected to be confined or semiconfined based on the observed increase in the subsequent water-level elevation data upon completion of drilling and monitoring well installation. Water levels in completed monitoring wells were approximately 26 feet below the surface. This is believed to be due to a graditional change in the character of the sandstone at depth and the presence of a clayey overburden soil (Figure 1-4). It is anticipated that the potentiometric surface of the shallow ground water regime at the site parallels the surface topography. Ground water flow within the shallow regime is believed to reflect surface topography, with flow from the north-northeast to south-southwest.

Seven ground water monitoring wells were installed in the shallow bedrock ground water regime during the USACEWES investigation (Figure 1-3). These seven monitoring wells are designated 30-1 through 30-7. The geologic and well construction diagrams for these monitoring wells are presented in Appendix A. Two monitoring wells were installed hydraulically upgradient of the site (30-1 and 30-3); one was installed hydraulically cross-gradient to the site (30-2), and four were installed hydraulically downgradient of the site (30-4, 30-5, 30-6 and 30-7). These seven wells are screened within the uppermost water-bearing zone.

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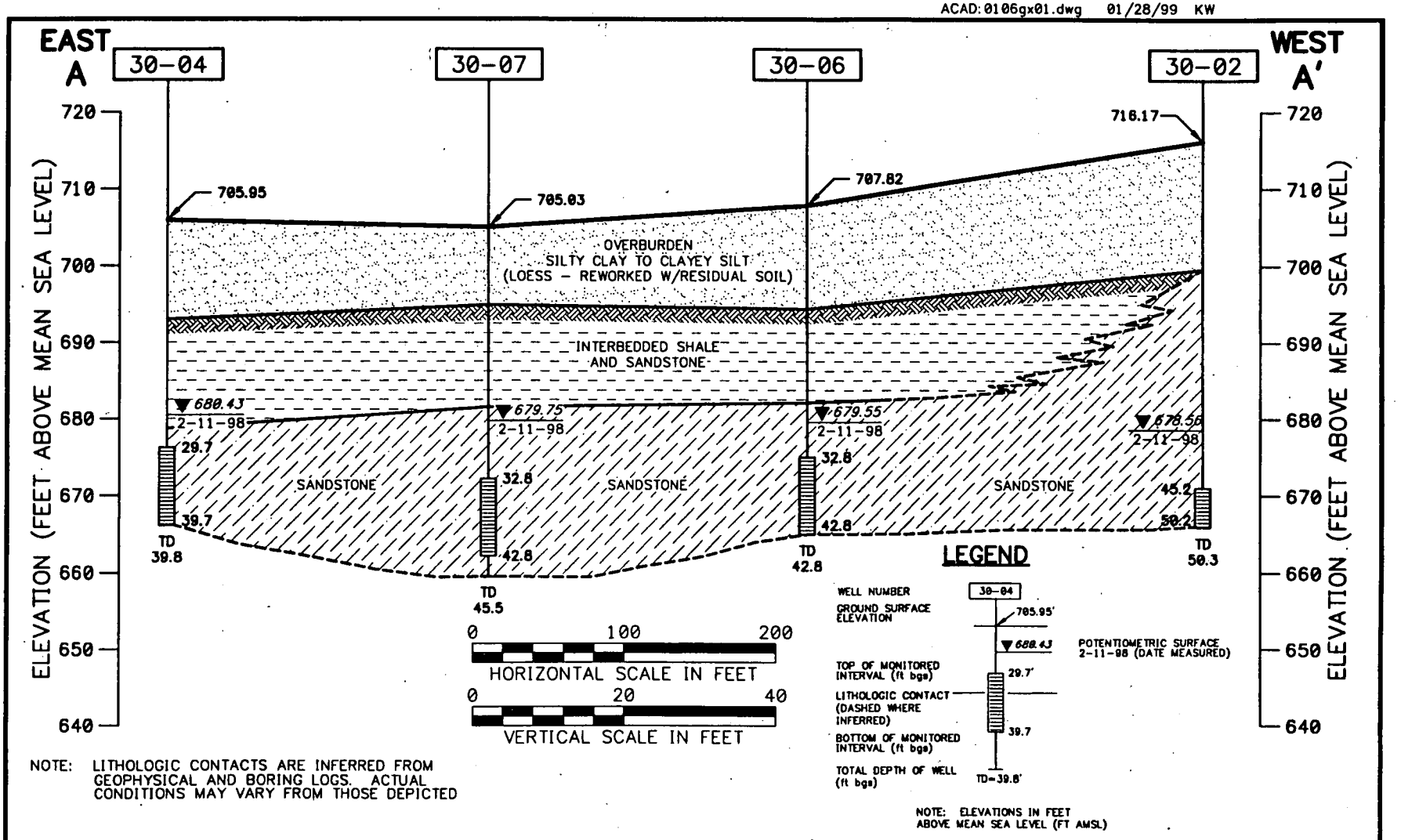

NOTE: LITHOLOGIC CONTACTS ARE INFERRED FROM GEOPHYSICAL AND BORING LOGS. ACTUAL CONDITIONS MAY VARY FROM THOSE DEPICTED

DRAWN BY KW DATE 1/28/99	DEPARTMENT OF THE NAVY · SOUTHERN DIV FACILITIES ENGINEERING COMD	GENERALIZED GEOLOGIC CROSS SECTION A-A' SWMU 30, LANDFARM SITE NSWC - CRANE, CRANE, INDIANA	CONTRACT NO. 0106
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COST/SCHED-AREA			APPROVED BY DATE
SCALE AS NOTED			DRAWING NO. FIGURE 1-4 REV. 0

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1.4 SAMPLING PLAN RATIONALE

The objective of this ground water sampling program is to obtain representative samples from the shallow ground water regime at the Landfarm. To accomplish this objective, five quarterly ground water sampling rounds will be performed for the seven on-site ground water monitoring wells. The results of the ground water sampling program will be used to determine the possible presence and extent of ground water contamination associated with application of sludge at the Landfarm. These ground water samples are to be collected at quarter-year intervals over approximately 1 year. The rationale for collecting quarterly samples is to examine the variability of the analytical data versus time and seasonal changes.

The first round of sampling will be conducted within 10 weeks of final Work Plan approval. Subsequent rounds will be collected at 3-month intervals following the first sampling event. Assuming final written approval of the Work Plan is received by September 1, 1999, the first sampling event would be completed by November 8, 1999. Subsequent rounds 2,3,4,and 5 would be conducted in February, May, August, and November of 2000, respectively.

The ground water samples will be analyzed for a list of parameters associated with single application operations. These parameters include modified list of Appendix IX volatile organic compounds (VOCs), Appendix IX metals (total and dissolved), explosives, cyanide, nitrate/nitrite, ammonia, TKN, and phosphorus (total and total dissolved). Specific analytes are presented in Table 1-2 of the QAPP. Hexavalent chromium analysis will also be performed using a U.S. EPA-accepted field test method (see Appendix E). Any exceedance of the hexavalent chromium RBTL specified in Table 1-2 of the QAPP will require a discussion with EPA Region 5 to determine if fixed-base laboratory analysis is required. Also, additional testing of hexavalent chromium using an accepted fixed-base laboratory analytical procedure may be required if hexavalent chromium results are in the range of 100 to 180 µg/L and the total chromium results are less than the field result. The rationale for using this parameter list is based on generator knowledge provided by Crane personnel.

Samples for the first two sampling rounds will be analyzed for the aforementioned list of parameters. The data from these initial two sampling rounds will be used to preliminarily characterize the upgradient and downgradient ground water and will allow TtNUS to develop a list of potential contaminants of concern for this site. The results will provide a basis for focusing the subsequent three sampling rounds on the list of potential contaminants of concern. The subsequent focused list will be based on a subset of the above parameter list. The scientific basis for determining the analysis subset will be as follows:

IF:	THEN:	ACTION:
The compound was not detected during the first and second sampling rounds.	No further analysis is recommended for this compound.	Remove compound from future sampling list.
The parameter was detected but in amounts statistically insignificant* relate to background data and state or federal standards.	No further analysis is recommended for this compound.	Remove compound from future sampling list.
The parameter was detected in amounts statistically significant* relative to background but less than state or federal standards.	Recommend continued analysis for the parameter in the subsequent three sampling rounds.	Keep compound on future sampling list.
The parameter was detected in amounts statistically significant* relative to background and greater than state or federal standards.	Recommend continued analysis for the parameter in the subsequent three sampling rounds.	Keep compound on future sampling list.

*Note: Statistical insignificance or significance will be determined based on a comparison of the maximum value downgradient to the maximum value upgradient. If one value exceeds the maximum upgradient value, that parameter will be retained for further analysis. Statistical analysis specified in 40 Code of Federal Regulations (CFR) 264 Subpart F will be conducted after four full rounds of data are available. Because metals are the primary contaminants of concern associated with this SWMU, analyses for the metals listed will be conducted for all 5 rounds, even if nondetectable values are obtained in the first 2 rounds.

1.5 PROJECT MANAGEMENT AND ORGANIZATION

TtNUS is responsible for the overall management of the project, including all field sampling activities. Navy personnel will actively support the investigation and will coordinate with TtNUS personnel during field activities. The responsible organizations and personnel involved in the project are as follows:

Southern Division
SOUTHNAVFACENGCOM
2155 Eagle Drive
P.O. Box 190010
North Charleston, South Carolina 29419-9010
(843) 820-7360

Mr. Bill Gates
Restoration Project Manager

Point of Contact
Naval Surface Warfare Center
300 Highway 361, Building 3260
Crane, Indiana 47522-5009

Mr. Thomas Brent
Installation Restoration Project Manager

Tetra Tech NUS, Inc.
Foster Plaza VII
661 Andersen Drive
Pittsburgh, PA 15220
(412) 921-7090

Ms. Debra Wroblewski, Program Manager
Dr. Roger A. Clark, Task Order Manager
Mr. Paul Frank, Quality Assurance Manager
Mr. Matt Soltis, Health and Safety Manager

This section presents the project management for the RFI to be conducted at NSWC Crane. Included in the following sections are the staffing and coordination requirements. At the direction of the Restoration Project Manager (RPM), TtNUS is responsible for the overall management, implementation, and inspection of contract field activities. Navy personnel will be actively involved and will coordinate with TtNUS personnel in a number of areas. The authorities and organizational relationship of key personnel are depicted on Figure 1-5. Responsibilities for program management, project management, field operations, and laboratory operations are discussed in the following sections.

1.5.1 Management Responsibilities

Navy Project Management

The U.S. Navy Restoration Project Manager (RPM), Bill Gates, has overall responsibility for all phases of the RFI. In general, the Navy RPM acts as the focal representative for the U.S. Navy, providing management, technical direction, and oversight for all NSWC Crane project activities performed by contractors and their subcontractors. In matters such as facilitation of access and of project activities, oversight, etc., the Navy RPM is assisted by the Installation Restoration Project Manager (IRPM), Thomas Brent.

Federal and State Regulators

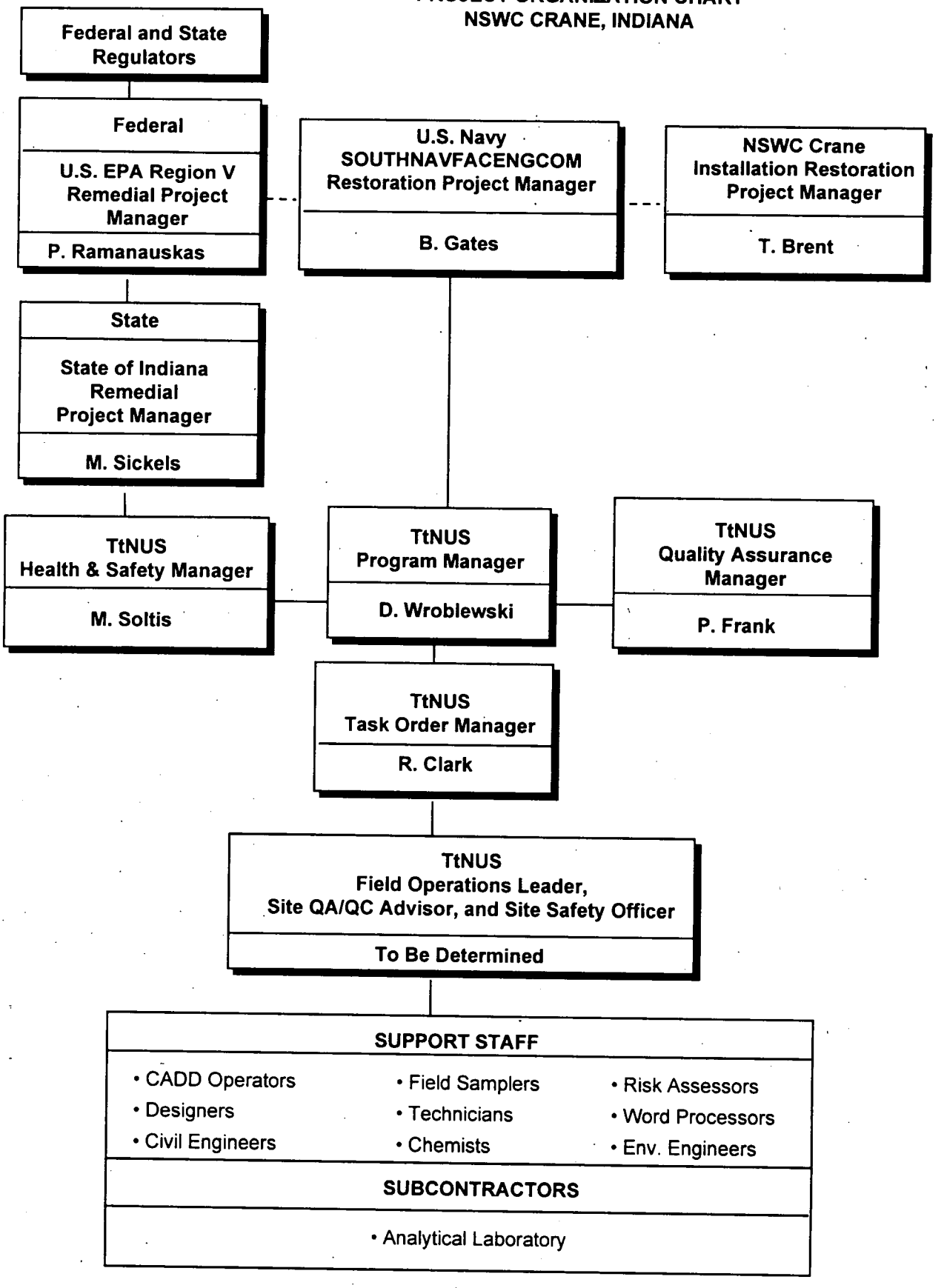
This ground water investigation is being conducted under a RCRA permit administered jointly by the U.S. EPA Region V and the State of Indiana. As such, all aspects of the project will require approval by both agencies. The U.S. EPA representative for the project is Mr. Peter Ramanauskas. The state representative for the project is Mr. Mike Sickels.

Program Manager

The TtNUS CLEAN Program Manager, Debbie Wroblewski, provides operations, technical, and administrative leadership, and oversees and supports quality policies. The Program Manager assigns project Task Order Managers (TOMs) and oversees their performance. The Program Manager also ensures the availability of technical and clerical resources for program operations, and maintains consistency in procedures and projects amongst Contract Task Order (CTO) assignments. In these matters, the Program Manager is assisted by the TOM.

FIGURE 1-5

PROJECT ORGANIZATION CHART
NSWC CRANE, INDIANA



Task Order Manager

The TtNUS TOM, Roger Clark, Ph.D., has overall responsibility for the project (i.e., scope, schedule, budget) and technical management, and is responsible for identifying and ensuring the availability of qualified on-site personnel. Additional responsibilities of the TOM are as follows:

- Ensuring timely resolution of project-related technical, quality, safety, and waste management issues.
- Functioning as primary interface with the contract RPM, field personnel, laboratory point-of-contact, and subcontractors.
- Monitoring and evaluating subcontractor performance.
- Coordinating and overseeing work performed by technical staff (including risk assessment and modeling).
- Coordinating and overseeing the maintenance of all project records.
- Coordinating and overseeing the review of project deliverables.
- Preparing and issuing final deliverables to the client.
- Approving the implementation of corrective actions.
- Performs final data assessment with assistance from the data validation coordinator

Health and Safety Manager

The TtNUS Health and Safety Manager (HSM), Matt Soltis, is responsible for the following:

- Implementing the Site Security and Health and Safety Plan (HASP).
- Overseeing the development and review of the Site Security and HASP.
- Assigning the Site Safety Officer (SSO) and supervising his or her performance.
- Conducting health and safety audits.

- Preparing health and safety reports for management.

1.5.2 Quality Assurance Responsibilities

This section identifies the quality assurance responsibilities for the NSWC Crane RFI. Responsibilities of U.S. EPA Region V regulators, TtNUS personnel, and the analytical laboratory are discussed.

U.S. EPA Region V Quality Assurance Manager

The U.S. EPA Region V Quality Assurance Manager is responsible for reviewing and approving the QAPP and providing overall quality assurance support and review throughout the RFI process.

TtNUS Quality Assurance and Quality Control Manager

The TtNUS Quality Assurance Manager (QAM), Paul Frank, is responsible for the following activities:

- Developing, maintaining, and monitoring quality assurance policies and procedures.
- Providing training to TtNUS staff in quality assurance and quality control (QA/QC) policies and procedures.
- Conducting systems and performance audits to monitor compliance with environmental regulations, contractual requirements, project plan requirements, and corporate policies and procedures.
- Conducting audits of project records.
- Ensuring external audits of subcontracted laboratories, and monitoring subcontractor quality controls and records.
- Assisting in the development of corrective action plans, and ensuring correction of nonconformances reported in internal or external audits.
- Overseeing the implementation of the QAPP.
- Overseeing and reviewing the development and revision of the QAPP.

- Overseeing the TtNUS Site QA/QC Advisor.

- Preparing quality assurance reports for management.

- Mr. Andy Kendrick (TtNUS) will be responsible for conducting all internal field audits.

1.5.3 Laboratory Responsibilities

The subcontract analytical laboratory will be responsible for analyzing all RFI samples in accordance with the specified analytical methods and for reporting data in accordance with the requirements outlined under the Contract Laboratory Program. The analytical laboratory will properly dispose of unused samples and report the receipt of any broken sample bottles or other problems relative to samples (e.g., headspace in volatile organic vials) to the TOM.

1.5.4 Field Responsibilities

The TtNUS field investigation team will be organized according to the activities planned. Field team members will be selected based on the type and extent of effort required. All team members will be appropriately skilled and trained for the tasks they are assigned to perform. The team will consist of a combination of the following personnel:

- Field Operations Leader (FOL) (The FOL may also fill other roles)
- Site QA/QC Advisor
- SSO
- Field hydrogeologist/geologists

The proposed NSWC Crane field activities will be performed by TtNUS personnel and subcontractors, and overseen by the TtNUS FOL. The FOL's responsibilities are discussed below.

The FOL is responsible for coordinating all on-site personnel and providing technical assistance when required. The FOL or designee will coordinate and be present during all sampling activities and will ensure the availability and maintenance of all sampling materials and equipment. The FOL is responsible for the completion of all sampling, field, and chain-of-custody documentation, will assume custody of all samples, and will ensure the proper handling and shipping of samples. Other duties of the FOL include the following:

- Functions as communications link between field crew members, the Site QA/QC Advisor, SSO, and TOM.
- Oversees the mobilization and demobilization of all field equipment.
- Resolves logistical, weather, personnel, and equipment problems.
- Maintains (or ensures the maintenance of) the site logbook.
- Initiates field change requests when necessary.

The FOL (or his assistant) will act as the Site QA/QC Advisor, who is responsible for ensuring adherence to all QA/QC guidelines as defined in the Field Sampling Plan (FSP) and QAPP. Strict adherence to these procedures is critical to the collection of acceptable and representative data. The following summarizes the Site QA/QC Advisor's responsibilities:

- Ensures that field duplicates and field quality control blanks are collected at the proper frequency.
- Ensures that additional volumes of sample are supplied to the analytical laboratory at the proper frequency to accommodate laboratory QA/QC analyses.
- Ensures that measuring and test equipment are calibrated, used, and maintained in accordance with applicable procedures.
- Acts as liaison between site personnel, laboratory personnel, and the QAM.
- Manages bottleware shipments, and oversees field preservation and in-field filtration activities.

The FOL will also serve as the SSO. The duties of the SSO are detailed in the Site Security and HASP. The SSO has stop-work authority that can be executed upon the determination of an imminent safety hazard.

To accomplish these duties effectively, the FOL may be assisted by a yet-to-be-determined TtNUS field geologist or hydrogeologist.

1.6 COMMUNITY RELATIONS

Six progress and review meetings are scheduled between the Navy, TtNUS staff, and members of the Restoration Advisory Board (RAB). The RAB will include representatives from regulatory agencies such

as the U.S. EPA, State Environmental Protection Branch, State Fish and Wildlife Commission, and the community. The first meeting will present the Revised Draft Work Plan, the second through fifth meetings will present the quarterly sampling results, and the sixth meeting will present the Draft Final Report.

1.7 DELIVERABLES AND DATA MANAGEMENT

Four interim reports will be issued approximately 2 months after the completion of each of the first four sampling rounds. These reports will briefly discuss the on-site field activities, observations, and sampling methodologies and discuss the sampling results. The results from the fifth and final sampling round will be incorporated into the draft final report. The draft final report will include a discussion and tables regarding all sampling results from this investigation.

Historical analytical data regarding the sludge samples and ground water chemistry data will be maintained in a database. The database will be used to identify data trends and anomalous data results and will be used to compare the results to state and federal standards. The database will also be used to identify anomalous results that are statistically significant relative to background concentrations.

1.8 PROJECT SCHEDULE

The first round of sampling will be conducted within 10 weeks of final Work Plan approval. Subsequent rounds will be collected at 3-month intervals following the first sampling event. Assuming final written approval of the Work Plan is received by September 1, 1999, the first sampling event would be completed by November 8, 1999. Subsequent rounds 2,3,4,and 5 would be conducted in February, May, August, and November of 2000, respectively.

2.0 SAMPLING AND ANALYSIS PLAN

This Sampling and Analysis Plan discusses general sampling operations and procedures for the proposed sampling events to be performed at the Landfarm. Details of the sampling events, including sampling locations, sample analytes, and rationale, are provided.

2.1 FIELD OPERATIONS SUMMARY

The purpose of this field investigation is to acquire ground water chemistry data at seven ground water monitoring wells installed around the perimeter of the Landfarm. These monitoring wells were installed by USACEWES during a previous investigation. USACEWES indicated that the surveyed elevations of the wells are suspect; therefore, the relative elevations of the seven wells will be resurveyed during the upcoming field sampling operations. For this investigation, seven ground water monitoring wells will be sampled quarterly. A total of five quarterly sampling events will be performed during this investigation. Ground water samples will be analyzed for the parameters listed in Table 2-1 during the first two sampling rounds. The results of the first two sampling rounds will allow TtNUS to identify the potential contaminants of concern for this site. The remaining three quarterly sampling events will be focused on the potential contaminants of concern. For purposes of discussion, it is assumed that samples collected during all five sampling events will be analyzed for the full list of parameters.

2.2 MOBILIZATION / DEMOBILIZATION

Following approval of the Work Plan, TtNUS will begin mobilization activities. All field team members will review the Sampling and Analysis Plan, the HASP, and the QAPP prior to mobilization. In addition, a field team orientation meeting will be held to familiarize personnel with the scope of the field activities.

The FOL will coordinate the mobilization activities for this project. The equipment required for the field activities will be mobilized from TtNUS's Pittsburgh office to the site by the FOL and the field crew. The Landfarm is in a fairly remote area of the Base. As a result, electricity is not available on-site. Therefore, power for electric pumps and all other electric-powered equipment, if required, will be supplied from portable gas-powered generators. It is presently anticipated that no portable gas-powered equipment will be used during field operations, but if conditions arise that require such equipment, an application for their use will be made to the NSWC Crane Fire Department. The FOL and crew will demobilize from the site upon completion of the field operations and transport all field equipment back to the Pittsburgh office.

TABLE 2-1

QUARTERLY CHEMICAL ANALYSIS SUMMARY
SWMU 30 (LANDFARM) INVESTIGATION
NSWC CRANE, INDIANA

Analytical Parameter	Analytical Method	Aqueous Samples	Field Duplicate Samples	Source Water Blanks	Equipment Rinsate Blanks	Matrix Spike/ Duplicate	Trip Blanks
Volatile Organics	SW-846 8260B	7	1	1	1	1/1	1
Mercury, Total and Dissolved	SW-846 7470A	7	1	1	1	1/1	0
Appendix IX Metals (except mercury), Dissolved	SW-846 6020	7	1	1	1	1/1	0
Appendix IX Metals (except mercury), Total	SW-846 6020	7	1	1	1	1/1	0
Cyanide	SW-846 9012A	7	1	1	1	1/1	0
Nitrate/Nitrite	EPA 353.2	7	1	1	1	1/1	0
Hexavalent Chromium	Field Hach Test Kit Method 8023	7	1	1	1	NA	0
Ammonia	EPA 350.1	7	1	1	1	1/1	0
Phosphorus, Total	EPA 365.2	7	1	1	1	1/1	0
Phosphorus, Dissolved	EPA 365.2	7	1	1	1	1/1	0
Total Kjeldahl Nitrogen	EPA 351.2	7	1	1	1	1/1	0

Notes: Specific analytes are listed in Table 1-2 of the QAPP. The total number of aqueous samples is based on sampling the seven existing wells. One field duplicate will be collected per round.

Matrix spike (MS) and duplicate samples will be collected at a frequency of 1 per 20 samples. For inorganics, an MS and a sample duplicate will be collected, and for organics, an MS and a matrix spike duplicate (MSD) will be collected. MS/MSDs and sample duplicates are not applicable for field analyses (NA).

2.3 GROUND WATER SAMPLING

The objective of this section is to provide guidance for the proper sampling equipment and techniques for ground water sample collection. All ground water sampling shall be conducted in accordance with TtNUS SOP SA-1.1 (Appendix C).

2.3.1 Water-Level Measurements and Ground Water Monitoring Well Evaluations

Water-level measurements and total well depth soundings will be obtained at each of the seven monitoring well locations at this site. All water-level measurements will be taken within a 24-hour period of consistent weather conditions. The sequence for measuring water levels at this site will proceed from ground water monitoring well locations 30-3, to 30-1 (background) to 30-2 (cross-gradient), to 30-4, 30-5, 30-6, and 30-7 (downgradient); this sequence will minimize the potential for cross-well contamination. All water-level measurements will be taken using an electronic water-level meter. Water-level elevations will be recorded to within a 0.01-foot accuracy from a marked reference point on the well riser pipe. Water levels will be recorded on a TtNUS water-level form. A blank water-level form is provided in Appendix B for reference purposes.

The condition of each ground water monitoring well will be evaluated prior to the initiation of well development and sampling. This evaluation will examine for the presence of a silted screen, a damaged surface casing, or a damaged concrete surface pad. The condition of well tags and locks will be recorded. Water levels will be measured and monitoring wells will be evaluated in accordance with procedures outlined in TtNUS SOP GH-1.2 (Appendix C).

A photoionization detector (PID) or flame ionization detector (FID) will be used to monitor the presence of organic vapors in the well annulus. The organic vapor monitor will be calibrated daily in accordance with procedures outlined in the manufacturer's operations manual. Organic vapor monitor calibration records will be recorded daily on TtNUS equipment calibrations sheets as per TtNUS SOP SA-6.3 (Appendixes B and C).

2.3.2 Monitoring Well Development and Purging

Older monitoring wells that have not been recently purged require development to maintain or improve their original specific capacity. Regaining high specific capacity is critical in approximating natural aquifer flow conditions and the subsequent collection of representative ground water samples. Therefore, the seven existing wells at the site will be developed prior to purging and sampling.

Each of the seven ground water monitoring wells will be developed and purged in accordance with procedures outlined in TtNUS SOP SA-1.1 (Appendix C). Each monitoring well will be developed during the first scheduled sampling round using a surge block and submersible pump. The purpose of the well development is to remove sediments from inside the casing and within the sand filter that surrounds the screen. Each monitoring well will be developed (pumped) for at least 1 hour to help remove sediment around the well screen and within the sand filter (SOP SA-1.1). Development will continue until all visual sediments have been removed. Water-level elevation data will be collected periodically during the well development process to ensure that the well is not pumped dry. In the event that the well runs dry, field personnel will allow sufficient recharge time and continue with other scheduled activities. Specific capacity data will be collected at the end of the development process. Specific capacity data are useful in determining the potential yield of a well and for estimating the hydraulic properties of the aquifer.

Each of the existing wells consist of a 5- to 10-foot screened section within an approximately 15-foot-thick artificial sand pack. The sandstone aquifer exhibits semiconfined to confined conditions and should provide sufficient water to develop, purge, and sample the wells. In order to remove the fine-grained material that has likely accumulated in the stagnant wells, surging and overpumping is the recommended method of development. Overpumping is not an ideal method for initial development of unconsolidated aquifers but is an effective development method for a relatively stable sandstone aquifer.

Overpumping is the process where a well is pumped at a higher rate than it will be subsequently purged and sampled. The objective is to remove fine-grained material at higher flow rates so that it will not be entrained in the sample collected at lower flow rates. Although it is anticipated that this aquifer can sustain a sufficient yield, if the well does pump dry during the 1-hour period, the well will be allowed to recharge back to a level greater than or equal to the height of the screened section and pumped a second time. The total pumping time is anticipated to equal 1 hour per well. Prior to and upon completion of development, the sediment thickness, if any, within the well will be measured and recorded on the development form.

Monitoring well development will be completed at least 24 hours before well purging and sampling. The intent of this delay is to provide time for the well and surrounding sand pack and aquifer material to sufficiently equilibrate to natural conditions.

The sequence for developing and purging ground water monitoring wells at this site will proceed from locations 30-3 and 30-1 (background), to 30-2 (cross-gradient), to 30-4, 30-5, 30-6, and 30-7 (downgradient) to minimize the potential for cross-well contamination.

Ground water monitoring well purging will be performed prior to each sampling event. The water-level elevation and total well depth will be measured prior to the start of ground water monitoring well purging. These measurements will be used to calculate the volume of water each ground water monitoring well contains for estimating purging requirements. To calculate the volume of water present, the following geometric formula for determining the volume of a cylinder will be used:

$$\text{volume} = \Pi r^2 hC$$

or

$$\text{volume} = (0.163)(h) \text{ for a 2-inch monitoring well}$$

Where: $\Pi = 3.14$ (dimensionless)
r = The radius of the well (feet)
h = The length of the water column in the well (feet)
C = 7.48 gallons per cubic foot
volume = gallons

Teflon-lined PVC bladder pumps will be used to sample each well. A minimum of one well volume will be purged from each ground water monitoring well using the low flow (0.14 L/min to 0.35 L/min) bladder pump prior to sampling. Measurements of pH, temperature, dissolved oxygen, turbidity, and conductivity will be taken at the start of purging and every 5 to 10 minutes thereafter. Purging will continue until these parameters stabilize or a maximum of six well volumes are removed. All development and purging fluids removed from the wells will be placed in labeled containers. Stabilization is achieved and sampling can begin when a minimum of one casing volume has been removed and three consecutive readings, taken at 5- to 10-minute intervals, are within the following limits:

- pH ± 0.1 standard unit
- Specific conductance ± 3 percent
- Temperature ± 3 percent
- Turbidity less than 10 Nephelometric Turbidity Units

2.3.3 Sampling

Ground water samples will be collected using low flow sampling techniques as described in SOP SA 1.1 (Teflon-lined PVC bladder pumps). All samples will be transferred directly from the bladder pump to the sample container. Samples for VOC analysis will always be collected first, making sure that zero head

space is present in the sample container once filled. Filtered ground water samples will be collected and analyzed for the presence of dissolved metals. The field-filtered samples will be processed using a 0.45 micron particulate filter. Unfiltered ground water samples will be collected and analyzed for total metals and all other parameters. Sample nomenclature and rationale are presented in Table 2-2. Bottle and sample preservation requirements are presented in Table 2-3.

One field duplicate sample will be collected per quarterly sampling event. The purpose of the field duplicate sample is to examine the variability of the samples. One trip blank will be collected per shipment of VOC samples. The purpose of the trip blank is to examine the potential for cross contamination of samples during shipping. One equipment rinsate blank will be collected for each of the sampling rounds. The equipment rinsate blank will be collected from a bladder pump to examine its cleanliness before it is installed in a monitoring well. The common purpose of the equipment rinsate blank is to examine the effectiveness of the decontamination procedures. One source water blank will be collected per quarterly sampling event for the purpose of evaluating contamination in water used for decontamination activities. Quality control samples are further discussed in Section 3.4.

Sampling chain-of-custody requirements are addressed in Section 3.3. All pertinent field data will be recorded on a sample log sheet and in the field log book. A TtNUS sample log sheet is included in Appendix B.

2.4 FIELD MEASUREMENTS

Field measurements recorded during field and sampling operations include air quality within the well casing, ground water temperature, pH, turbidity, and specific conductance. The following instruments are anticipated to be used during the field activities:

- FID or PID
- Temperature probe
- Specific conductance meter
- pH meter
- Dissolved oxygen meter
- HACH Test Kit for hexavalent chrome
- Turbidity meter
- Electronic water-level meter
- Oxidation reduction potential (ORP) flow-through cell

TABLE 2-2

SUMMARY OF SAMPLING ACTIVITIES
NSWC CRANE, INDIANA
PAGE 1 OF 5

GROUND WATER SAMPLES FOR FIRST SAMPLING ROUND

Sample I.D.	Sample Location (See Figure 1-3)	Well Completion Depth	Analytical Parameters	Sample Rationale
030-GW-001-01	30-1	45.5 feet	Parameter List as per Table 2-1	Upgradient Sampling Location
030-GW-002-01	30-2	50.3 feet	Parameter List as per Table 2-1	Cross-Gradient Sampling Location
030-GW-003-01	30-3	51.6 feet	Parameter List as per Table 2-1	Upgradient Sampling Location
030-GW-004-01	30-4	39.8 feet	Parameter List as per Table 2-1	Downgradient Sampling Location
030-GW-005-01	30-5	42.5 feet	Parameter List as per Table 2-1	Downgradient Sampling Location
030-GW-006-01	30-6	42.8 feet	Parameter List as per Table 2-1	Downgradient Sampling Location
030-GW-007-01	30-7	42.8 feet	Parameter List as per Table 2-1	Downgradient Sampling Location
030-IW-001-01	n/a	n/a	Parameter List as per Table 2-1	IDW Sample

QA/QC SAMPLES FOR FIRST SAMPLING ROUND

DUP-GW-001-01	Duplicate Sample	n/a	Parameter List as per Table 2-1	Examine Sample Variability
030-TB-001-01	Trip Blank	n/a	VOC List as per Table 2-1	Examine for VOC Cross-Contamination During Shipping
030-FB-001-01	Field Blank	n/a	Parameter List as per Table 2-1	Examine Water used for Equipment Decontamination
030-RB-001-01	Equipment Rinsate Blank	n/a	Parameter List as per Table 2-1	Determine Effectiveness of Equipment Decontamination

TABLE 2-2

SUMMARY OF SAMPLING ACTIVITIES
NSWC CRANE, INDIANA
PAGE 2 OF 5

GROUNDWATER SAMPLES FOR SECOND SAMPLING ROUND

Sample I.D.	Sample Location (See Figure 1-3)	Well Completion Depth	Analytical Parameters	Sample Rationale
030-GW-001-02	30-1	45.5 feet	Parameter List as per Table 2-1	Upgradient Sampling Location
030-GW-002-02	30-2	50.3 feet	Parameter List as per Table 2-1	Cross-Gradient Sampling Location
030-GW-003-02	30-3	51.6 feet	Parameter List as per Table 2-1	Upgradient Sampling Location
030-GW-004-02	30-4	39.8 feet	Parameter List as per Table 2-1	Downgradient Sampling Location
030-GW-005-02	30-5	42.5 feet	Parameter List as per Table 2-1	Downgradient Sampling Location
030-GW-006-02	30-6	42.8 feet	Parameter List as per Table 2-1	Downgradient Sampling Location
030-GW-007-02	30-7	42.8 feet	Parameter List as per Table 2-1	Downgradient Sampling Location
030-IW-001-02	n/a	n/a	Parameter List as per Table 2-1	IDW Sample

QA/QC SAMPLES FOR SECOND SAMPLING ROUND

DUP-GW-001-02	Duplicate Sample	n/a	Parameter List as per Table 2-1	Examine Sample Variability
030-TB-001-02	Trip Blank	n/a	VOC List as per Table 2-1	Examine for VOC Cross-Contamination During Shipping
030-FB-001-02	Field Blank	n/a	Parameter List as per Table 2-1	Examine Water used for Equipment Decontamination
030-RB-001-02	Equipment Rinstate Blank	n/a	Parameter List as per Table 2-1	Determine Effectiveness of Equipment Decontamination

TABLE 2-2

SUMMARY OF SAMPLING ACTIVITIES
NSWC CRANE, INDIANA
PAGE 3 OF 5

GROUND WATER SAMPLES FOR THIRD SAMPLING ROUND - (QUARTERLY SAMPLING)

Sample I.D.	Sample Location (See Figure 1-3)	Well Completion Depth	Analytical Parameters	Sample Rationale
030-GW-001-03	30-1	45.5 feet	Focused Parameter List	Upgradient Sampling Location
030-GW-002-03	30-2	50.3 feet	Focused Parameter List	Cross-Gradient Sampling Location
030-GW-003-03	30-3	51.6 feet	Focused Parameter List	Upgradient Sampling Location
030-GW-004-03	30-4	39.8 feet	Focused Parameter List	Downgradient Sampling Location
030-GW-005-03	30-5	42.5 feet	Focused Parameter List	Downgradient Sampling Location
030-GW-006-03	30-6	42.8 feet	Focused Parameter List	Downgradient Sampling Location
030-GW-007-03	30-7	42.8 feet	Focused Parameter List	Downgradient Sampling Location
030-IW-001-02	n/a	n/a	Parameter List as per Table 2-1	IDW Sample

QA/QC SAMPLES FOR THIRD SAMPLING ROUND

DUP-GW-001-03	Duplicate Sample	n/a	Focused Parameter List	Examine Sample Variability
030-TB-001-03	Trip Blank	n/a	VOC List as per Table 2-1	Examine for VOC Cross-Contamination During Shipping
030-FB-001-03	Field Blank	n/a	Parameter List as per Table 2-1	Examine Water used for Equipment Decontamination
030-RB-001-03	Equipment Rinstate Blank	n/a	Parameter List as per Table 2-1	Determine Effectiveness of Equipment Decontamination

TABLE 2-2

SUMMARY OF SAMPLING ACTIVITIES
NSWC CRANE, INDIANA
PAGE 4 OF 5

GROUND WATER SAMPLES FOR FOURTH SAMPLING ROUND - (QUARTERLY SAMPLING)

Sample I.D.	Sample Location (See Figure 1-3)	Well Completion Depth	Analytical Parameters	Sample Rationale
030-GW-001-04	30-1	45.5 feet	Focused Parameter List	Upgradient Sampling Location
030-GW-002-04	30-2	50.3 feet	Focused Parameter List	Cross-Gradient Sampling Location
030-GW-003-04	30-3	51.6 feet	Focused Parameter List	Upgradient Sampling Location
030-GW-004-04	30-4	39.8 feet	Focused Parameter List	Downgradient Sampling Location
030-GW-005-04	30-5	42.5 feet	Focused Parameter List	Downgradient Sampling Location
030-GW-006-04	30-6	42.8 feet	Focused Parameter List	Downgradient Sampling Location
030-GW-007-04	30-7	42.8 feet	Focused Parameter List	Downgradient Sampling Location
030-IW-001-04	n/a	n/a	Parameter List as per Table 2-1	IDW Sample

QA/QC SAMPLES FOR FOURTH SAMPLING ROUND

DUP-GW-001-04	Duplicate Sample	n/a	Focused Parameter List	Examine Sample Variability
030-TB-001-04	Trip Blank	n/a	VOC List as per Table 2-1	Examine for VOC Cross-Contamination During Shipping
030-FB-001-04	Field Blank	n/a	Parameter List as per Table 2-1	Examine Water used for Equipment Decontamination
030-RB-001-04	Equipment Rinstate Blank	n/a	Parameter List as per Table 2-1	Determine Effectiveness of Equipment Decontamination

TABLE 2-2

SUMMARY OF SAMPLING ACTIVITIES
NSWC CRANE, INDIANA
PAGE 5 OF 5

GROUNDWATER SAMPLES FOR FIFTH SAMPLING ROUND - (QUARTERLY SAMPLING)

Sample I.D.	Sample Location (See Figure 1-3)	Well Completion Depth	Analytical Parameters	Sample Rationale
030-GW-001-05	30-1	45.5 feet	Focused Parameter List	Upgradient Sampling Location
030-GW-002-05	30-2	50.3 feet	Focused Parameter List	Cross-Gradient Sampling Location
030-GW-003-05	30-3	51.6 feet	Focused Parameer List	Upgradient Sampling Location
030-GW-004-05	30-4	39.8 feet	Focused Parameter List	Downgradient Sampling Location
030-GW-005-05	30-5	42.5 feet	Focused Parameter List	Downgradient Sampling Location
030-GW-006-05	30-6	42.8 feet	Focused Parameter List	Downgradient Sampling Location
030-GW-007-05	30-7	42.8 feet	Focused Parameter List	Downgradient Sampling Location
030-IW-001-05	n/a	n/a	Parameter List as per Table 2-1	IDW Sample

QA/QC SAMPLES FOR FIFTH SAMPLING ROUND

DUP-GW-001-05	Duplicate Sample	n/a	Focused Parameter List	Examine Sample Variability
030-TB-001-05	Trip Blank	n/a	VOC List as per Table 2-1	Examine for VOC Cross-Contamination During Shipping
030-FB-001-05	Field Blank	n/a	Parameter List as per Table 2-1	Examine for VOC Cross-Contamination During Shipping
030-FB-001-05	Equipment Rinstate Blank	n/a	Parameter List as per Table 2-1	Determine Effectiveness of Equipment Decontamination

Notes:

IDW – Investigation Derived Waste
n/a – not applicable
Matrix spike (MS) and duplicate samples will be collected at a frequency of 1 per 20 samples. For inorganics, an MS and a sample duplicate will be collected, and for organics, an MS and a matrix spike duplicate (MSD) will be collected. MS/MSDs and sample duplicates are not applicable for field analyses (NA). See Table 2-3 for specific numbers of vials/bottles required.

TABLE 2-3

SUMMARY OF SAMPLE ANALYSES, CONTAINER TYPES AND VOLUMES, PRESERVATION REQUIREMENTS, AND HOLDING TIMES NSWC CRANE, INDIANA

Parameter	Sample Container	Container Volume	Preservation[1]	Maximum Holding Time[2]	Analytical Methodology
Volatile Organic Compounds	Glass, black phenolic plastic screw cap, Teflon-lined septum	(3) 40 mL[3]	Cool to 4°C, dark, zero headspace, HCl to pH < 2	14 days	SW-846[4] Method 8260B[5]
Explosives	Amber glass, Teflon-lined cap	(2) 1000 mL[6]	Cool to 4°C, dark	Extraction within 7 days; analysis within 40 days of extraction	SW-846 Method 8330
Metals (total and dissolved)	Polyethylene bottle, plastic cap, plastic liner	1000 mL	HNO_3 to pH < 2	Within 180 days; mercury within 28 days	SW-846 Method 6020; mercury - SW-846 Method 7470A
Ammonia	Polyethylene bottle, plastic cap, plastic liner	1000 mL[7]	Cool to 4°C, H_2SO_4 to pH <2	28 days	EPA Method [15] 350.1
Cyanide	Polyethylene bottle, plastic cap, plastic liner	500 mL	Cool to 4°C, NaOH to pH > 12	14 days	SW-846 Method 9012A
Nitrate/Nitrate	Polyethylene bottle, plastic cap, plastic liner	1000 mL[7]	Cool to 4°C, H_2SO_4 to pH <2	28 days	EPA Method 353.2
Phosphorus (total and dissolved)	Polyethylene bottle, plastic cap, plastic liner	1000 mL[7]	Cool to 4°C, H_2SO_4 to pH <2	28 days	EPA Method 365.2
Total Kjeldahl Nitrogen (TKN)	Polyethylene bottle, plastic cap, plastic liner	1000 mL[7]	Cool to 4°C, H_2SO_4 to pH <2	28 days	EPA Method 351.2

1 HCl = Hydrochloric acid, H_2SO_4 = Sulfuric Acid, NaOH = Sodium Hydroxide, HNO_3 = Nitric Acid.
2 All holding times are from date of collection.
3 Four additional 40-mL vials are required for samples designated for matrix spike/matrix spike duplicate analysis (minimum 1 per 20 samples).
4 U.S. EPA, 1986. Test Methods for Evaluating Solid Wastes, Physical/Chemical Methods. SW-846, 3rd ed, including updates.
5 Analysis will be performed using a 25-mL sample volume in order to achieve lower quantitation limits for ground water samples.
6 Two additional 1000-mL bottles are required for samples designated for matrix spike/matrix spike duplicate analysis (minimum 1 per 20 samples).
7 Ammonia, nitrate/nitrite, TKN, and total phosphorus samples will be provided for analysis in the same 1000-mL bottle.

mL = milliliter
< = less than
°C = degrees centigrade

Field instruments are calibrated in accordance with the manufacturer's procedures and the following:

- The pH meters are calibrated daily. At least two buffer solutions that bracket the expected pH shall be used (e.g. 4.0 and 7.0).
- Specific conductance meters are calibrated prior to each day's use.
- Thermometers are calibrated monthly at the TtNUS equipment warehouse in Pittsburgh.
- Turbidity meters are calibrated daily using a standard within the expected range of sample turbidities.
- FIDs and PIDs are calibrated daily as per TtNUS SOPs ME-12 and ME-15.
- The dissolved oxygen and ORP probes will be calibrated before each sampling event.

Horiba® water quality meters are commonly used in place of separate temperature, conductivity, pH, and turbidity meters. This device can record six separate parameters, and the calibration is checked prior to each day's use.

All instrument calibration is recorded on an Equipment Calibration Log Sheet (Appendix B). During calibration, a maintenance check is performed on each piece of equipment. If damaged or defective parts are identified during the maintenance check, and it is determined that the damage could impact the instrument's performance, the instrument is removed from service until the defective parts can be repaired or the instrument replaced.

2.5 DECONTAMINATION

Teflon-lined PVC bladder pumps will be used at this site for well purging and sample collection. All non-dedicated equipment required for field sampling activities will be decontaminated prior to and during sampling activities. Any decontamination water that is generated will be handled in the same manner as purge water, as described in Section 2.6.

For any non-dedicated ground water sampling equipment, decontamination will be performed both prior to field sampling and between samples. The following decontamination steps will be conducted.

- Potable water rinse
- Liquinox detergent wash
- Potable water rinse
- Deionized water rinse
- Air dry

Field measurement equipment that comes into contact with ground water will be decontaminated using a deionized water rinse. This equipment is anticipated to include a Horiba® (or equivalent) water quality meter and a water level meter. The sequence of sampling presented in Section 2.3.1 will be followed to minimize cross-contamination.

2.6 INVESTIGATION DERIVED WASTE

It is anticipated that this investigation will generate three types of potentially contaminated residues:

- Personal protective equipment (PPE)
- Development and purge fluids
- Equipment decontamination fluids

PPE - All PPE will be double bagged and placed in trash receptacles at the facility.

Development and Purge Fluids - All development and purge fluids will be collected and stored on-site using a 300- and/or 2,100-gallon plastic holding tank, respectively, until appropriate IDW analyses are completed. Providing that IDW analyses are acceptable as described below, development and purge fluids will be discharged to the NSWC-permitted sanitary sewer system. IDW analyses from the first round of sampling will be deemed suitable for use on subsequent sampling rounds. The 2,100-gallon tank will be situated in a semi-permanent location near an access to the wastewater treatment plant. Purge and development fluids will be collected on-site using a 300-gallon portable holding tank attached to a truck or a smaller polyethylene container, as appropriate. The fluids will be transported to the main 2,100-gallon holding tank daily and transferred from the portable 300-gallon tank using a submersible pump. The main fluid storage tank (2,100-gallon) will be sealed and labeled with contents, date, and origin(s) of waste. An IDW sample will be collected from the 300-gallon tank at the completion of each sampling round and analyzed for the parameters listed in Table 2-1. The IDW sample will be identified using the sample nomenclature examples provided in Table 2-2 and Section 3.1. NSWC Crane will use the analytical results to determine if the IDW fluids can be treated on-site via the wastewater treatment facility or if they must be treated or disposed of off site. Progress of the laboratory analyses will be

monitored by the TOM to confirm that the IDW fluids are not stored on-site for over 90 days. The laboratory turnaround time for the IDW sample analyses is 30 days, which should be sufficient time to identify a potential problem before corrective action is required. NSWC Crane will take possession of the containers and contents after receipt of the analytical results.

3.0 SAMPLE HANDLING PROCEDURES

Sample handling includes field-related considerations regarding the selection of sample containers, preservatives, allowable holding times, and analyses requested. In addition, sample identification, packaging, and shipping will be addressed.

The EPA User's Guide to the Contract Laboratory Program (EPA, December, 1988) and the Federal Register (EPA, October 26, 1984) were used to determine the container types, holding times, and sample preservation methods.

3.1 SAMPLE IDENTIFICATION SYSTEM

Each sample collected will be assigned a unique sample tracking number as per TtNUS SOP CT-0.4 (Appendix C). The sample tracking number consists of a four-segment alpha-numeric code that identifies the site, the sampling location, the sample media, and the sample number that corresponds to a depth. All other pertinent information regarding sample identification will be recorded in the field log books. Examples of the sample codes to be used for this project are provided in Table 2-2.

3.2 SAMPLE PACKAGING AND SHIPPING

Samples will be packaged and shipped in accordance with TtNUS SOP SA-6.1 (Appendix C). The FOL will be responsible for ensuring the completion of the following forms:

- Sample Labels
- Chain-of-Custody Forms
- Chain-of-Custody Seals for Coolers
- Shipping Labels for Coolers
- Express Mail Air Bills

3.3 SAMPLE CUSTODY

Custody of samples must be maintained and documented at all times as per TtNUS SOP SA 6.1. Chain-of-custody begins with the collection of samples in the field. A sample chain-of-custody record is provided in Appendix B.

3.4 QUALITY CONTROL SAMPLES

Quality control (QC) samples will be collected or generated during environmental sampling activities. QC samples include field duplicates, source water blanks, and equipment rinsate blanks. Trip blanks will be provided by the analytical laboratory chosen to support this project. Table 2-2 of this document presents the types and numbers of required QC samples. The four types of field QC samples are defined as follows:

Field Duplicates. Field duplicates are obtained during a single act of sampling and are used to assess the overall precision of the sampling and analysis program. Ten percent of all samples for each media shall be field duplicates, or a minimum of one per sampling event. Field duplicates shall be analyzed for the same parameters in the laboratory as their environmental sample counterparts.

Equipment Rinsate Blanks. Equipment rinsate blanks are obtained under representative field conditions by running analyte-free water through sample collection equipment after decontamination and placing it in the appropriate sample containers for analysis. Rinsate blanks will be collected for non-dedicated equipment for all sampling rounds.

Source Water Blanks. Source water blanks are obtained by sampling the waters used for decontamination during the field investigation. Samples consist of analyte-free water used for decontaminating sampling equipment. Source water blanks will be used to determine if the analyte-free water or the laboratory bottles are contributing to sample contamination. Source water blanks will be collected for each type of water used for decontamination and will be submitted at a frequency of one per sampling event. Source water blanks, as applicable, will be analyzed for the entire suite of parameters under investigation. It is anticipated that one source water blank will be collected for each sampling round.

Trip Blanks. Trip blanks are included when analyzing for VOCs and will be prepared and provided by the subcontractor laboratory. Trip blanks will remain with the sample containers at all times and are thus subjected to the same field conditions as the field samples. One trip blank will be included in every shipping or sample collection cooler that contains samples to be analyzed for VOCs, regardless of sample matrix.

Matrix Spikes/Duplicates. Matrix spikes are investigative samples analyzed to provide information about the effect of the sample matrix on the digestion and measurement methodology. All matrix spikes for organic analysis are performed in duplicate. Matrix spike (MS) and duplicate samples will be collected at a frequency of 1 per 20 samples. For inorganics; an MS and a sample duplicate will be collected. For

organics, an MS and a matrix spike duplicate (MSD) will be collected. MS/MSDs and sample duplicates are not applicable for field analyses.

3.5 RECORD KEEPING

In addition to chain-of-custody records, certain standard forms will be completed for sample description and documentation. These forms shall include sample log sheets, boring logs, daily activity records, and logbooks. A bound, weatherproof notebook shall be maintained by each sampling event leader. All information related to sampling or field activities will be recorded in the field notebook. This information will include, but is not limited to, sampling time, weather conditions, unusual events, field measurements, an descriptions of photographs.

A bound, weatherproof logbook shall be maintained by the FOL. This book will contain a summary of each day's activities and will reference the field notebooks when applicable. At completion of the field activities, the FOL shall submit to the TOM all field records, data, field notebooks, logbooks, chain-of-custody receipts, sample log sheets, and daily activity logs. Requirements for field documentation are outlined in TtNUS SOP SA-6.3 (Appendix C).

REFERENCES

Brown & Root (B&R) Environmental, August 1996. Work Plan for Solid Waste Management Unit 30; Naval Surface Warfare Center (NSWC); Crane, Indiana.

B&R Environmental, November 1996. Draft Work Plan for Phase II Groundwater RCRA Facility Investigation; Solid Waste Mangement Unit 30 (Landfarm); Naval Surface Warfare Center (NSWC); Crane, Indiana.

U.S. Army Corps of Engineers, Waterways Experiment Station, 1995. Work Plan for RCRA Facility Investigation, Groundwater Release Assessment, SWMU 30/00 Landfarm/Sludge Application Site, Crane Division, Naval Surface Warfare Center, Crane Indiana, Vicksburg, Mississippi.

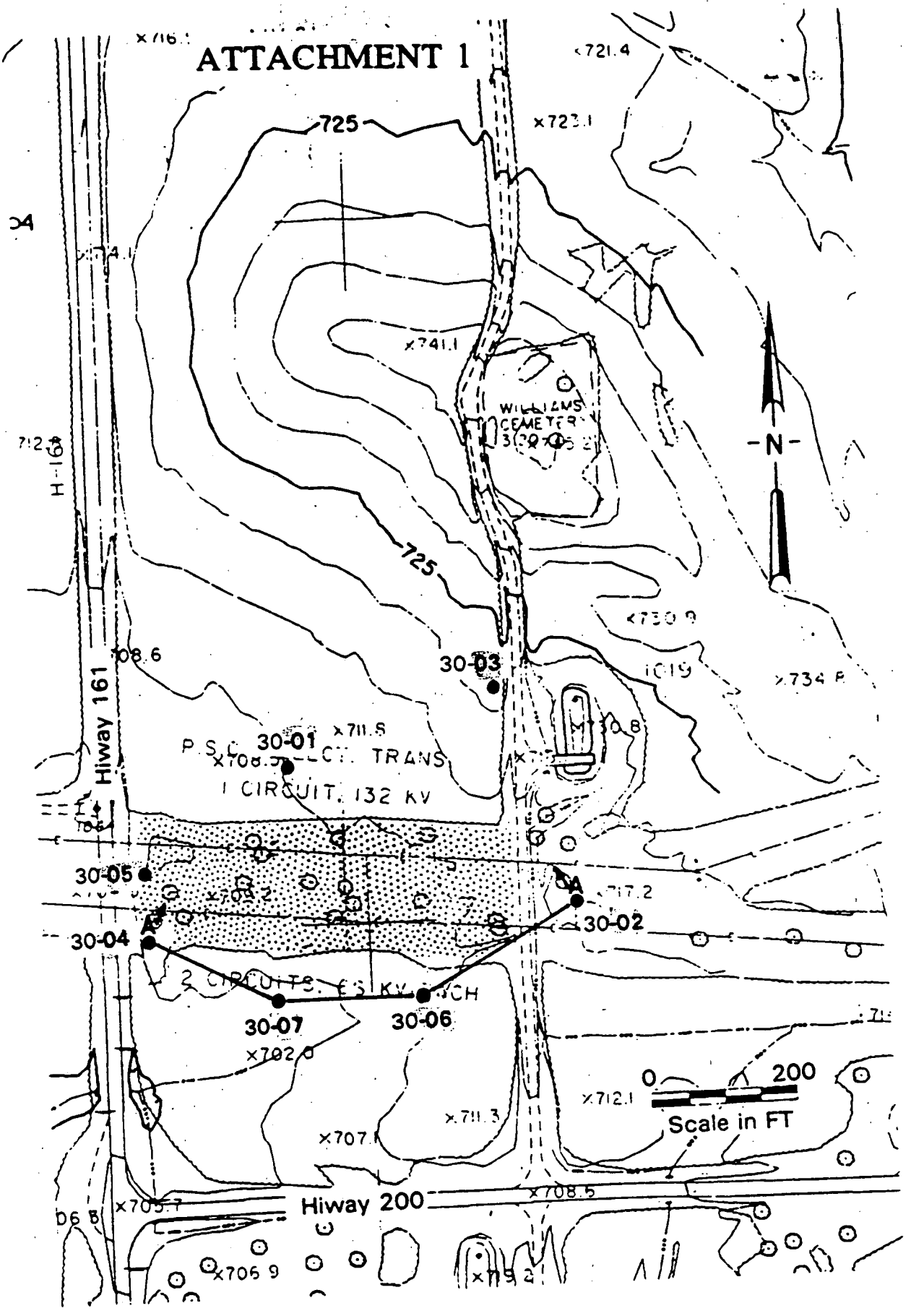
U.S. Environmental Protection Agency (U.S. EPA), October 1984. Federal Register.

U.S. Environmental Protection Agency (U.S. EPA), December 1988. EPA's Users Guide to the Contract Laboratory Program.

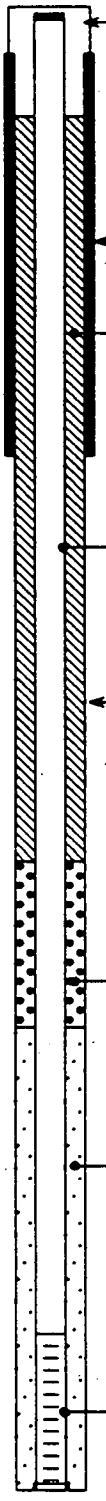
APPENDIX A

GEOLOGIC LOGS AND WELL CONSTRUCTION DIAGRAMS

ATTACHMENT 1



WELL CONSTRUCTION



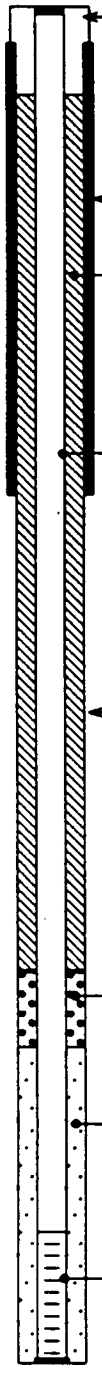
DEPTH (feet)	SAMPLE NUMBER	BLOWS/FT.	PID (ppm)	SAMPLES	SOIL CLASS	GRAPHIC LOG	MATERIALS DESCRIPTION
	1 2				ML-CL		OVERBURDEN (0-12 FEET) SILTY CLAY TO CLAYEY SILT LIGHT COLORED, PROBABLY LOESS, MIXED WITH RESIDUAL SOIL
5 10							
							INTERBEDDED SHALE AND SANDSTONE (12-18.6) SHALE IS GRAY TO BLACK INTERBEDDED WITH QUARTZ SANDSTONE AND BLACK GRAY RHYTHMITE SHALE
15							
							SANDSTONE (18.6-45.5) RED-BROWN MICACEOUS QUARTZITE, MEDIUM GRAINED, FRIABLE
20 25							
							▽ Water Level 9-23-93
30							
							SANDSTONE IS MORE COMPETENT, BUT LOST SOME WATER BETWEEN 31.8 AND 37.8 FEET
35							
							SAND WITH WATER ENTERING (FRACTURE ZONE) WATER ENTERS BELOW 37.8 (ARTESIAN)
40 45							
							TD=45.5 DATE DRILLED 9-17-93
50							Revised by Brown and Root Environmental June, 1996

U.S. Army Corps of Eng.
Waterways Experiment Station

LOG OF BORING 30-01

CLIENT NAME Naval Surface Warfare Center LOCATION Crane, Indiana
SURFACE ELEVATION 710.51 (TOC) Feet DRILLING METHOD [illegible] Auger/HQ Wireline
TOTAL DEPTH OF HOLE 45.5 Feet GEOLOGIST W.L. Murphy

WELL CONSTRUCTION



DEPTH (feet)	SAMPLE NUMBER	BLOWS/FT.	PID (ppm)	SAMPLES	SOIL CLASS	GRAPHIC LOG	MATERIALS DESCRIPTION
	1 2				ML-CL		OVERBURDEN (0-16.6 FEET) MOTTLED BROWN TO OCHRE SILTY CLAY TO SILT. RESIDUAL SOIL. OCHRE COLOR W/ROCK FRAG. BELOW 10 FEET SOIL IS LESS COHESIVE AND DRY. REFUSAL AT 16.6
5							
10							
15							
20							SANDSTONE, LIMONITE STAINED. (16.6-22.8). MANY BEDDING PLANE BREAKS. FRIABLE. (RQD=0) LOST LOT OF WATER AT 21.5-22.5 PROBABLY THROUGH THE JOINTS. CORE PIECES LESS THAN 0.1 FEET
25							SANDSTONE. (22.8-26.6) FRAGMENTED AND JOINTED BECOMES HARDER AT 33.8-35.0
30							
35							∇ Water Level 9-23-93
40							
45							SANDSTONE IS CLEAN, WHITE FROM 43.6-46.4 BECOMES BROWN AT 47-47.5. LIGHT BROWN. FRIABLE. SOFT TO BOTTOM OF HOLE (RQD=45) WATER ENTERING THIS ZONE (AQUIFER)
50							TD=50.3 DATE DRILLED 9-21-93
55							Revised by Brown and Root Environmental June, 1998
60							

U.S. Army Corps of Eng.
Waterways Experiment Station

LOG OF BORING 30-02

CLIENT NAME Naval Surface Warfare Center LOCATION Crane, Indiana
SURFACE ELEVATION 716.17 (TOC) Feet DRILLING METHOD 7 in. Auger/HQ Wireline
TOTAL DEPTH OF HOLE 50.3 Feet GEOLOGIST W.L. Murphy

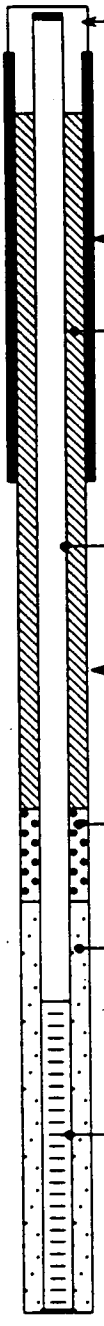
WELL CONSTRUCTION	DEPTH (feet)	SAMPLE NUMBER	BLOWS/FT.	PID (ppm)	SAMPLES	SOIL CLASS	GRAPHIC LOG	MATERIALS DESCRIPTION
3-in Diam. Steel Protective Casing 2.4 Feet Stick-up; 6-in. PVC Surface Casing; Cement/ Bentonite Seal	5, 10					ML-CL		OVERBURDEN (0-12.2 FEET): 0-1.5 SILT (ML) TO (CL) PROBABLY LOESS WITH LIMONITE STAINS. REWORKED AND RESIDUAL SOIL. REFUSAL AT 10.2
2-in. Schedule 40 PVC Casing; 8in. Borehole	15, 20, 25							INTERBEDDED BLACK SHALE, SILTSTONE WITH SOFT CLAY AND IRONSTONE. CLAY IS PROBABLY WEATHERED SHALE. GRADATIONAL CHANGE TO FLASER BEDDED SANDSTONE AT 26.5
33.5 TO 37.5 Bentonite Pellet Seal; 37.5 TO 51.6 Sand Pack; 41.4 TO 51.4 0.02 Slotted PVC Well Screen	30, 35, 40, 45, 50							FLASER BEDDED SANDSTONE W BLACK SHALE INTERBEDS TO 35.0. (=35.0) SANDSTONE, TAN FINE TO MEDIUM GRAINED QUARTZ SANDSTONE W. SOME BLACK MINERAL GRAINS. BECOMES COARSER BELOW 39.4 ▽ water Level 9-15-93. SANDSTONE AS ABOVE STREAKED W/BROWN COLOR FROM 50.5 TO 57.5 AND IRON STAINED SOFT AND FRIABLE (AQUIFER)
	55, 60							TD=51.6 DATE DRILLED 9-24-93. Revised by Brown and Root Environmental June, 1996

U.S. Army Corps of Eng.
Waterways Experiment Station

LOG OF BORING 30-03

CLIENT NAME Naval Surface Warfare Center LOCATION Crane, Indiana
SURFACE ELEVATION 719.1' (TOC) Feet DRILLING METHOD Hollow Auger/HQ Wireline
TOTAL DEPTH OF HOLE 51.6 Feet GEOLOGIST W.L. Murphy

WELL CONSTRUCTION



DEPTH (feet)	SAMPLE NUMBER	BLOWS/FT.	PID (ppm)	SAMPLES	SOIL CLASS	GRAPHIC LOG	MATERIALS DESCRIPTION
5, 10					ML-CL		OVERBURDEN (0-12.9 FEET) SILT TO SILTY CLAY, PROBABLY LOESS TAN AND MOTTLED, GRADES WITH DEPTH TO REWORKED LOESS AND ROCK FRAGMENTS. REFUSAL AT 12.9 FEET.
15, 20, 25							INTERBEDDEDBLACK SHALE, OCHRE CLAY TO TAN SHALE AND SANDSTONE. TRACE OF LIGNITE RQD=0. CORE IS VERY BROKEN
30, 35, 40							SANDSTONE (27.2-39.8) FINE TO MEDIUM GRAINED, MORE FRIABLE BELOW 31.9 FEET. ▽ water Level 9-28-93 SANDSTONE IS AS ABOVE WITH A COLOR CHANGE TO WHITE AT 38.9. MEDIUM GRAINED, FRIABLE WITH GOOD RQD (95%). (AQUIFER)
							TD=39.8 DATE DRILLED 9-27-93
45, 50							Revised by Brown and Root Environmental June, 1996

U.S. Army Corps of Eng.
Waterways Experiment Station

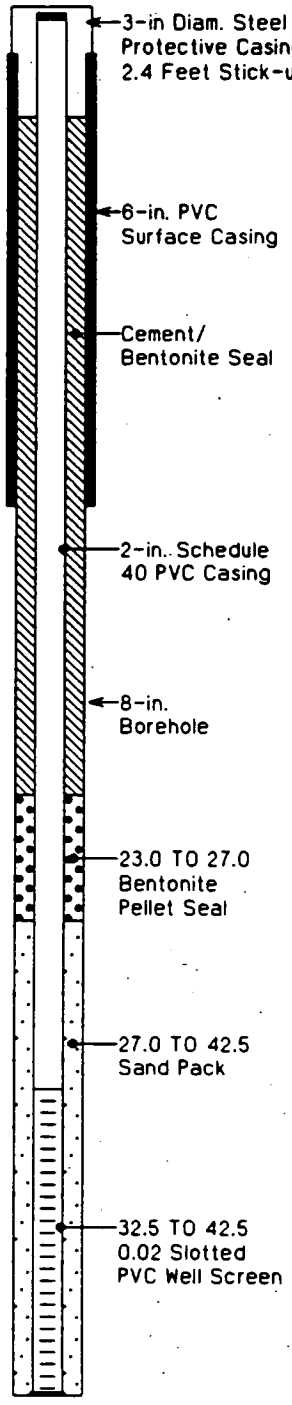
LOG OF BORING 30-04

CLIENT NAME Naval Surface Warfare Center LOCATION Crane, Indiana

SURFACE ELEVATION 705.95 (TOC) Feet DRILLING METHOD 7 in. Auger HQ Wire

TOTAL DEPTH OF HOLE 39.8 Feet GEOLOGIST W.L. Murphy

WELL CONSTRUCTION



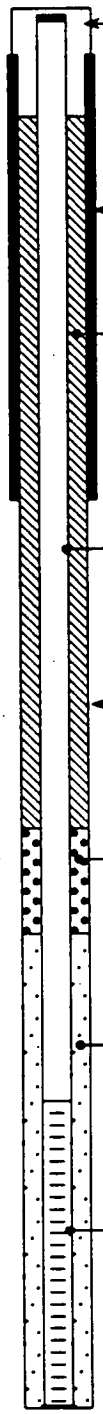
DEPTH (feet)	SAMPLE NUMBER	BLOWS/FT.	PID (ppm)	SAMPLES	SOIL CLASS	GRAPHIC LOG	MATERIALS DESCRIPTION
5, 10	1				ML-CL		OVERBURDEN (0-13.6 FEET) LOESS. (ML/CL), PROBABLY REWORKED WITH RESIDUAL SOIL. PEBBLE SIZE ROCK FRAGMENTS BELOW 5 FEET. REFUSAL AT 13.6
15, 20							INTERBEDDED TAN OCHRE SOFT CLAY AND HARD SANDSTONE (13.6-22.7) RQD VERY LOW WITH SOME VERY BROKEN ZONES 22.7 TO 24 INTERBEDDED SHALE AND SANDSTONE
25, 30, 35, 40							▽ Water Level 9-29-93 SANDSTONE, FINE TO COARSE GRAINED, MORE FRIABLE WITH DEPTH, VERY FEW BLACK SHALE LAMINAE. RQD=50% SANDSTONE IS MASSIVE (BEDDING ONLY APPARENT AT BREAKS) RQD=100%, EXCEPT BOTTOM 3 FEET (AQUIFER)
45, 50							TD=42.5 DATE DRILLED 9-29-93 Revised by Brown and Root Environmental June, 1996

U.S. Army Corps of Eng.
Waterways Experiment Station

LOG OF BORING 30-05

CLIENT NAME Naval Surface Warfare Center LOCATION Crane, Indiana
SURFACE ELEVATION 707.28 (TOC) Feet DRILLING METHOD 7 in. Auger/HG [illegible]
TOTAL DEPTH OF HOLE 42.5 Feet GEOLOGIST W.L. Murphy

WELL CONSTRUCTION



DEPTH (feet)	SAMPLE NUMBER	BLOWS/FT.	PID (ppm)	SAMPLES	SOIL CLASS	GRAPHIC LOG	MATERIALS DESCRIPTION
5, 10	1				ML-CL		OVERBURDEN (0-13.4 FEET). REWORKED LOESS (ML-CL) WITH ROCK FRAGMENTS, LITTLE PLASTICITY, TAN TO OCHRE SILT WITH MINOR CLAY. LOESS REWORKED WITH RESIDUAL SOIL. TOP OF ROCK AT 13.4
15, 20							INTERBEDDED BLACK SHALE, SOFT OCHRE TO GRAY CLAY, HARD RED-BROWN IRONSTONE, AND BROWN SANDSTONE (13.4 TO 23.3), RQD=0. 23.3 TO 25.7 INTERBEDDED BLACK SHALE, COAL AND SANDSTONE, WAVY OR FLASER BEDDED.
25, 30, 35, 40							SANDSTONE (25.7-42.8) ▽ Water Level 9-30-93 SANDSTONE, BROWN FRIABLE, MEDIUM GRAINED WITH ABRUPT CHANGE TO WHITE TO LIGHT GRAY AT 29.8 BECOMES FINE-GRAINED AND CALCAREOUS, RQD=85% SHARP CONTACT AT 34.5 TO BROWN SANDSTONE, FRI NON-CALCAREOUS WITH OCCASIONAL STRINGERS.
45, 50							TD=42.8 DATE DRILLED 9-29-93

Revised by Brown and Root Environmental June 1996

U.S. Army Corps of Eng.
Waterways Experiment Station

LOG OF BORING 30-06

CLIENT NAME Naval Surface Warfare Center LOCATION Crane, Indiana
SURFACE ELEVATION 707.82 (TOC) Feet DRILLING METHOD 7 in. Auger/HQ Wireline
TOTAL DEPTH OF HOLE 42.8 Feet GEOLOGIST W.L. Murphy

WELL CONSTRUCTION

DEPTH (feet)	SAMPLE NUMBER	BLOWS/FT.	PID (ppm)	SAMPLES	SOIL CLASS	GRAPHIC LOG	MATERIALS DESCRIPTION
					ML-CH		OVERBURDEN. 0-10.1 FEET) (ML-CH)
5, 10							INTERBEDDED CLAY, SHALE AND SANDSTONE FROM 10.1 TO 23.5
15, 20, 25, 30, 35, 40							SANDSTONE 23.5-42.8) ∇ Water Level 10-9-93
45, 50							TD=42.8 DATE DRILLED 10-6-93

Revised by Brown and Root Environmental June, 1996

U.S. Army Corps of Eng.
Waterways Experiment Station

LOG OF BORING 30-07

CLIENT NAME Naval Surface Warfare Center LOCATION Crane, Indiana
SURFACE ELEVATION 705.03 (TOC) Feet DRILLING METHOD 7 in. Auger/HQ Wire
TOTAL DEPTH OF HOLE 45.5 Feet GEOLOGIST W.L. Murphy

DRILLING LOG (Cont Sheet) | ELEVATION TOP OF HOLE | **Hole No.** Landfarm 30-01

PROJECT: LANDFARM Wells (Crane) | INSTALLATION: Well installation | SHEET ___ OF ___ SHEETS

ELEVATION a	DEPTH b	LEGEND c	CLASSIFICATION OF MATERIALS (Description) d	% CORE RECOVERY e	BOX OR SAMPLE NO. f	REMARKS (Drilling time, water loss, depth of weathering, etc., if significant) g
						All depths shown are from Top of Ground (TOG)
			Protective 3" steel casing and cap cemented into PVC casing	30-01		
						2A' STICKUP
			GROUND			0
			6" PVC Surface Casing			OVERBURDEN ML-CL
						10
			12.0			INTERBEDDED SHALE & SANDSTONE
			Grout (Neat cement/bentonite)			
			18.6			20
			25.1		9/23/93	
			Bentonite Pellets			9/21/93
			30.5			30 SANDSTONE
			33.5 Sand			AQUIFER (Water entering this zone)
						40
			TD 45.5 T.O.G.			
						50

DRILLING LOG (Cont Sheet) ELEVATION TOP OF HOLE

Hole No. Longterm 30-02

PROJECT | INSTALLATION | SHEET OF SHEETS

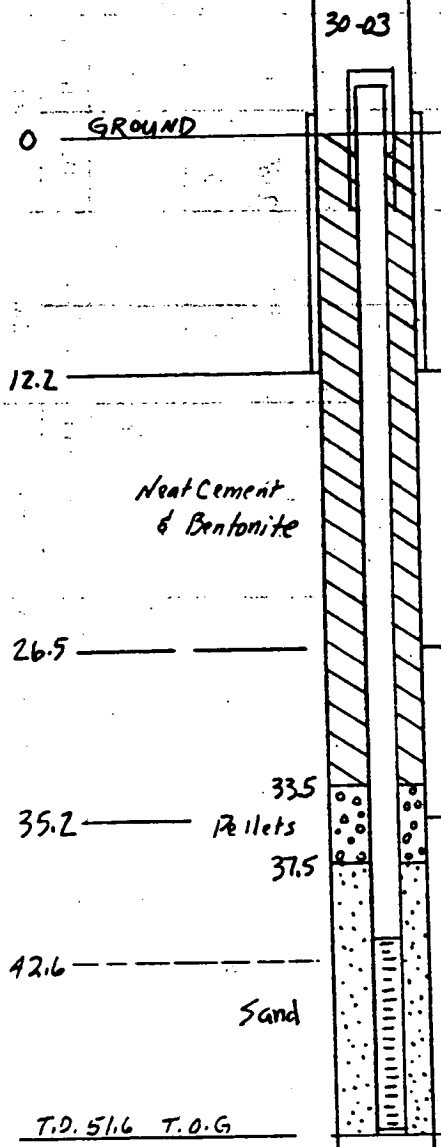
ELEVATION a	DEPTH b	LEGEND c	CLASSIFICATION OF MATERIALS (Description) d	% CORE RECOVERY e	BOX OR SAMPLE NO. f	REMARKS (Drilling time, water loss, depth of weathering, etc., if significant) g
				30-02		2.4' stickup
			GROUND (TOG)			
						OVERBURDEN
			6" PVC Surface Casing			CL-ML
						SANDSTONE
					9/29/93 ▽	
			35' Bentonite 38'			
			Sand		45.2 SCRN	AQUIFER (Water entering this zone)
			TD 50.3 TOG		50.2	

DEPTH, FT TOG: 0, 10, 20, 30, 40, 50

DRILLING LOG (Cont Sheet) | ELEVATION TOP OF HOLE | **Hole No.** 30-03

PROJECT | INSTALLATION | SHEET OF SHEETS

ELEVATION a	DEPTH b	LEGEND c	CLASSIFICATION OF MATERIALS (Description) d	% CORE RECOVERY e	BOX OR SAMPLE NO. f	REMARKS (Drilling time, water loss, depth of weathering, etc., if significant) g



DRILLING LOG (Cont Sheet) ELEVATION TOP OF HOLE ... **Hole No.** Landfarm 30-04

PROJECT | INSTALLATION | SHEET OF SHEETS

ELEVATION a	DEPTH b	LEGEND c	CLASSIFICATION OF MATERIALS (Description) d	% CORE RECOVERY e	BOX OR SAMPLE NO. f	REMARKS (Drilling time, water loss, depth of weathering, etc., if significant) g
			30-04			
			0 GROUND			0
						OVERBURDEN
						ML-CL
						10
			12.9'			
						INTERBEDDED
					08/28/93 ▽	SHALE &
						20 SANDSTONE
			23.5 Bentonite 26.5			
			?29' - - - - - - ?		29.7	- - - - - - - ?
						AQUIFER →
			Sand			30
						SANDSTONE
			TD 39.8 T.O.G.		39.7	40
						50

DRILLING LOG (Cont Sheet)

ELEVATION TOP OF HOLE

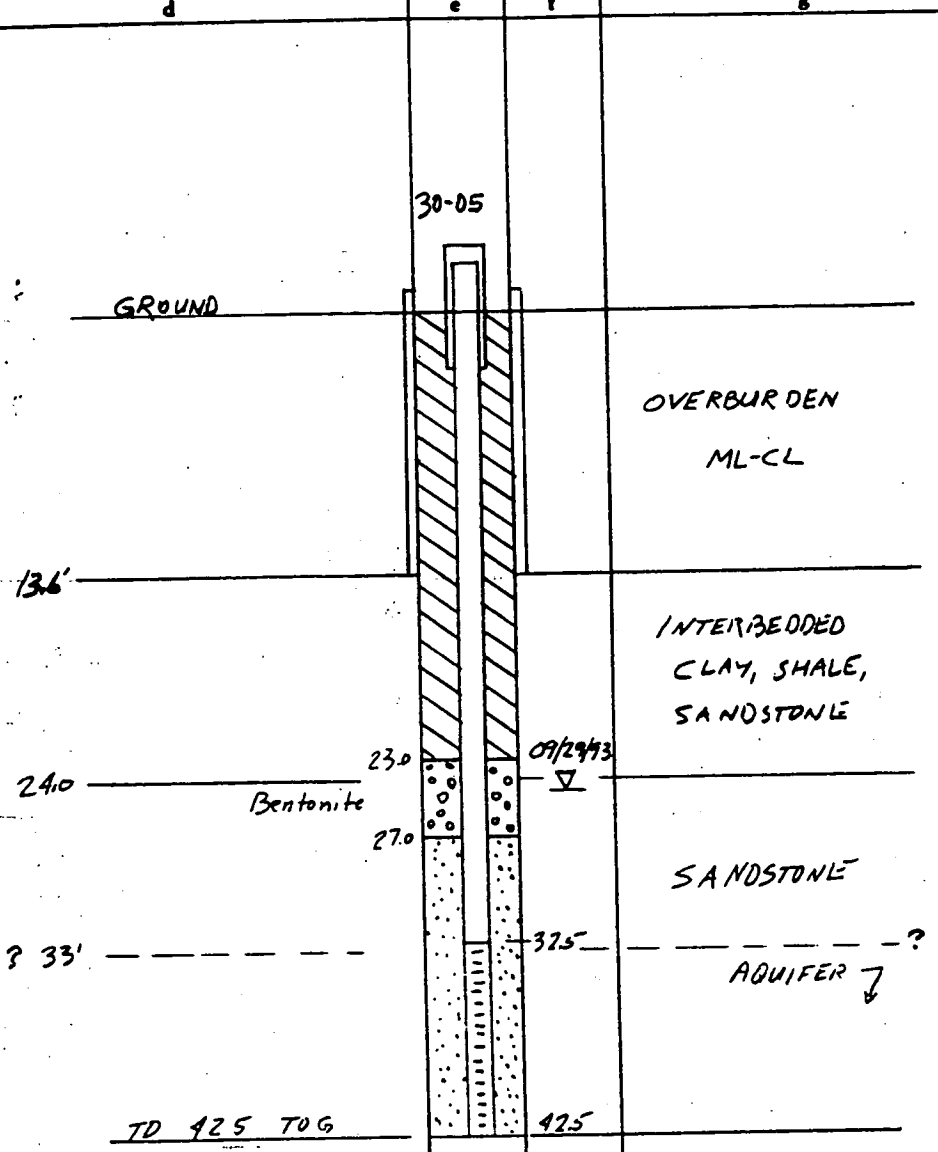
Hole No. 30-05

PROJECT

INSTALLATION

SHEET OF SHEETS

ELEVATION a	DEPTH b	LEGEND c	CLASSIFICATION OF MATERIALS (Description) d	% CORE RECOVERY e	BOX OR SAMPLE NO. f	REMARKS (Drilling time, water loss, depth of weathering, etc., if significant) g



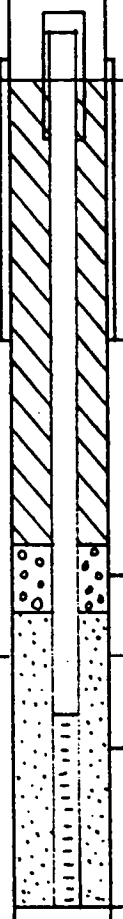
DEPTH, FT (TOG)

0 10 20 30 40 50

ILLING LOG (Cont Sheet) | ELEVATION TOP OF HOLE | Hole No. 30-06

OJECT | INSTALLATION | SHEET OF SHEETS

EVATION a	DEPTH b	LEGEND c	CLASSIFICATION OF MATERIALS (Description) d	% CORE RECOVERY e	BOX OR SAMPLE NO. f	REMARKS (Drilling time, water loss, depth of weathering, etc., if significant) g
			0' GROUND			
						OVERBURDEN ML-CL
			13.4			
						INTERBEDDED SHALE, CLAY AND SANDSTONE
			24.0		9/30/93 ▽	
			25.7 Bentonite 27.4			
						SANDSTONE, BROWN
			29.8			
						SANDSTONE, WHITE, CALC.
			34.5 Sand			
						(AQUIFER) SANDSTONE, BROWN
			TD 42.8 T.O.G.			



DEPTH, FT: 0, 10, 20, 30, 40, 50

Depths are from T.O.G.

DRILLING LOG (Cont Sheet) | ELEVATION TOP OF HOLE | **Hole No. 30-07**

PROJECT | INSTALLATION: Landfarm | SHEET OF SHEETS

ELEVATION a	DEPTH b	LEGEND c	CLASSIFICATION OF MATERIALS (Description) d	% CORE RECOVERY e	BOX OR SAMPLE NO. f	REMARKS (Drilling time, water loss, depth of weathering, etc., if significant) g
			0 GROUND			0
						OVERBURDEN CH-ML
			10.1			10
						INTERBEDDED CLAY, SHALE AND SANDSTONE
						20
			22.0			
			23.5			
			Bentonite		10/9/93	
			28.1			SANDSTONE
						30
			Sand			
						40
			T.D. 42.8 T.D.G.			

ATTACHMENT 2

BORING LOG
FIELD DATA

Project: Crane Landfarm Wells Site: Landfarm Date: 9/17/93
Location: NSWCC Job No.:
Drill Rig: Failing 1500 Inspector: W.L. Murphy Operator: E. Brown, V. Mixon Surface El: ~ Boring No.: Landfarm 30-01

SAMPLE NUMBER	DATE TAKEN	STRATUM FROM	STRATUM TO	DRIVE FROM	DRIVE TO	SAMPLE FROM	SAMPLE TO	TYPE OF SAMPLER			CLASSIFICATION AND REMARKS
1		0		0.0		0.0	0.4	Drove 3" Split	Jar #1		Silty clay to clayey silt (CL/ML).
2					2.00	1.4	2.0	spoon (jars)	Jar # 2		Light colored, ochre below 0.5'. Probably
			12.0	6" Flt	Auger to	13.8'					loess, mixed w/ residual soil at depth.
		12.0		13.8	14.4	13.8	14.4	Drove SS w/ 300 lb hammer			Gray to black, hard shale. Dry. Sampled
											w/ SS, discarded sample. Could drive only
				14.4	20.5	14.4	20.5 —	HQ Core barrel	RUN 1 6.1	PULL 3.8	0.6' w/ SS: v. hd. At 14.4', set up
				Coring	w/ water			(10')	Lost	8.3	mud pan for drill water (no mud), cored
											remainder of hole w/ 10' core barrel.
											HQ Wire/water. 12.0 - 18.6' is interbedded
											red-brn ironstone (quartz sandstone)
											and black-gray rhythmite shale
			18.6								(wavy-bdd).
		18.6									18.6' to 20.5 is SANDSTONE (see description
											below)

WES FORM 910 Sheet 1 of 3 Sheets

BORING LOG
FIELD DATA

Project _____ Site _____ Date 9/18/93
Location _____ Job No. _____
Drill Rig _____ Inspector _____ Operator _____ Surface El _____ Boring No Landfarm 30 -01

SAMPLE NUMBER	DATE TAKEN	STRATUM FROM	STRATUM TO	DRIVE FROM	DRIVE TO	SAMPLE FROM	SAMPLE TO	TYPE OF SAMPLER			CLASSIFICATION AND REMARKS
Drove 6" PVC casing to 14.9 ft (thru 0.8) Water did not come up overnight. Rockbit to 20.7', Flushed w/ water to clean out hole. Bailed hole again, water level at 18.3'. Can't tell if water coming up. Went back in w/ core barrel for 5' run											
				20.7	25.8			HQ Core barrel	RUN 2 5.1	PULL 4.85	20.7-25.8 SANDSTONE as above. Red-brown
									Core loss	0.25	micaceous, quartzitic. Med. grained, friable,
	Bailed-out hole - Not making water. - Back in w/ core barrel.										fairly soft to drill. Rounded, well sorted, mostly
											quartz w/ mica, some black mineral (carbonaceous)
											grains especially on bedding planes. Limonite (?)
											staining is heavy, prominent along bedding. Prominent
											thin bedding, some x-bdng.
				25.8	31.8			HQ Core barrel	RUN 6.0	PULL 6.0	25.8-31.8 SANDSTONE as above. (Competent
	Bailed - Not making water								100% core		rock, few breaks.
				31.8	37.8	31.8	37.6	"	RUN 6.0	PULL 5.8	31.8-37.8 SANDSTONE as above. Lost
	Bailed - Not making water. Water taped at 37.0'								100% core 0.2' in hole.		a little water this run.
	on 9/18 @ 1530 - Let stand over weekend.										
	Water did not come up over weekend										

WES FORM JAN 74 819 EDITION OF NOV 1971 MAY BE USED

Sheet 2 of 3 Sheets

41.5
37.6

BORING LOG
FIELD DATA

Project _____ Site _____ Date 9/20/93
Location _____ Job No. _____
Drill Rig _____ Inspector _____ Operator _____ Surface El _____ Boring No. LANDFARM 30 - 01

SAMPLE NUMBER	DATE TAKEN	STRATUM FROM	STRATUM TO	DRIVE FROM	DRIVE TO	SAMPLE FROM	SAMPLE TO	TYPE OF SAMPLER			CLASSIFICATION AND REMARKS
	0820 - Monday	-									
			45.5	37.8	46.1	37.6	45.5	HQ Wireline Core	Run 5 8.3	Pull 7.90	37.8 - 45.5 SANDSTONE as above.
		Hole	taped	at	45.5			left	no core 0.6 in	loss hole	Interval of chocolate brown color at
											40.8 to 41.5' depth (TOG). Sandstone
	Bailed hole to bottom. Water came up 8' in 5 minute (making water).										is otherwise lighter in color than above, pure
	Set 2" (0.020" sld) PVC screen - bottom plug at 45.5'. Added										qtz sandstone, v. friable in bottom foot.
	med-grained sand by hand, in a slurry, up to 30.5' depth.										
	Set bentonite pellets to 25.1' depth (T.O.G.) @ 1215, 5/20/93.										Water enters below 37.8' (artesian)
	Grouted to surface w/ 2 bags Portland Cement thickened with bentonite mud.										
	2.4' stickup of 2" PVC casing.										
	Water stood at 28.1' TOG (30.5 TOC) on 9/21/93										

WES FORM JAN 74 819 EDITION OF NOV 1971 MAY BE USED

Sheet 3 of 3 Sheets

BORING LOG
FIELD DATA

Project LANDFARM MONITORING WELLS Site LANDFARM, CRANE Date 9/21/93
Location Job No.
Drill Rig Failing 15ee Inspector MURPHY Operator BROWN/MIXON Surface El ~714 Boring No. 30-02

SAMPLE NUMBER	DATE TAKEN	STRATUM FROM	STRATUM TO	DRIVE FROM	DRIVE TO	SAMPLE FROM	SAMPLE TO	TYPE OF SAMPLER			CLASSIFICATION AND REMARKS
1	9/21	0.0		0		0.2	0.6	3" Split spn	JAR	#1	Mottled brown to ochre silty clay (CL)
2	9/21				1.5	1.1	1.5	"	JAR	#2	to silt (ML) (loess). Some plasticity (moldable)
			16.6	0.0	16.6	NONE		6" Flight auger			from cuttings. - Residual soil, ochre color, CL
											w/rock frags (pebble size). Plastic, cohesive.
											Below ~10', soil is less cohesive, more
											like ML, dry. Refusal at 16.6' RQD
	Pushed 6" PVC Casing to 16.6' w/ 1.1' stick-up. Sealed bottom of casing										
	w/ "Hole Plug" (bentonite), also top.										
	9/22	16.6		16.6	22.8			HQ Core barrel	RUN 1 6.2	PULL 9.6	16.6-22.8 SANDSTONE, limonite stained,
		Coring w/ water				16.6	18.8 -	discarded rest	Lost 1.6'		like boring 30-01. Many bedding plane breaks
								Taped 22.8			friable, core pieces < 0.8' (RQD = 0) Lost lot
											of water at ~21.5' to 22.8, probably thru
											jts. Kept only top 2.2' of core.
				22.8	33.0				RUN 2 10.2	PULL 9.2	SANDSTONE as above. Fragmented &
						25.3	27.1 -	discarded rest	Lost	1.0'	jtd 22.8 to 26.6, better below. Many bdng
											jnts. Kept 25.3-27.1 of core.

WES FORM JAN 74 819 EDITION OF NOV 1971 MAY BE USED

Sheet 1 of 2 Sheets

BORING LOG
FIELD DATA

Project _____ Site _____ Date 9/23/93
Location _____ Job No. _____
Drill Rig _____ Inspector _____ Operator _____ Surface El _____ Boring No. 30-02

SAMPLE NUMBER	DATE TAKEN	STRATUM FROM	STRATUM TO	DRIVE FROM	DRIVE TO	SAMPLE FROM	SAMPLE TO	TYPE OF SAMPLER			CLASSIFICATION AND REMARKS
	9/22			33.0	43.0			HQ Core	RUN 3 10.0	PULL 9.5	Taped at 42.9 SANDSTONE as above
								Taped 42.9	0.1 in 0.4	hole Lost	Harden at 33.8'-35.0'
		W.L	at 30.1	TOC	@ 1345	, 9/22					
		W.L	at 36.0	TOC	@ 0725	, 9/23					
	9/23		50.3	43.0	50.3			HQ Core	RUN 4 7.3	PULL 7.0	SANDSTONE as above. Clean, white from
			Hole	Taped	at 503	TOG		Taped 50.3	Lost	0.4	43.6 - 46.4. Chocolate brown @ 47.0 - 47.5,
											Light brown, friable, soft to bottom.
		Making	water								
	Set	5' screen at		50.2	to 45.2	Sand	pack to	38'. Bentonite pellets to			35'. Grouted to surface.
			2.4'	stickup	on riser						
			W.L.	at	35.2 TOG	on	9/24				
					35.0	on	9/27				
					34.4	on	9/29				

WES FORM JAN 74 819 EDITION OF NOV 1971 MAY BE USED

Sheet 2 of ___ Sheets

BORING LOG
FIELD DATA

Project [illegible] Site [illegible] Date 9/24/93
Location [illegible] Job No. [illegible]
Drill Rig [illegible] Inspector [illegible] Operator [illegible] Surface El [illegible] Boring No. [illegible]

SAMPLE NUMBER	DATE TAKEN	STRATUM FROM	STRATUM TO	DRIVE FROM	DRIVE TO	SAMPLE FROM	SAMPLE TO	TYPE OF SAMPLER			CLASSIFICATION AND REMARKS
1	9/24	0.0		0	1.5	1.[illegible]	1.5	3" Split spoon	JAR	#1	0-1.5 [illegible] (ML) to CL. Probably loess w/ limonite stain
			12.2	0	12.2	None		7" ID Auger			Loess, reworked, and residual soil, as above [illegible] 30-01 and -02. Dry. Refusal at 12.2 TOG
			Set 6" PVC casing to 12.2' TOG, sealed at bottom w/ bentonite								
	9/24	12.2		12.4	22.4	12.4	22.2	HQ Core barrel	RUN 1 10.0	PULL 9.8	12.2-22.2 Interbedded [illegible] black shale, ochre siltstone and soil tan clay, w/ occasional v. hd red-brn ironstone (cemented sandstone). Clay is probably weathered shale. 1.0' core loss.
		Coring w/ water						Tagged 22.2'	0.2' in 1.0' lost	hole	
			Kept all of Run 1 (Box 1) for stratigraphy								
			26.5	22.4	32.6	25.5	26.8	HQ Core barrel	RUN 2 10.2	PULL 10.1	22.4-26.5 Interbdd shale, ss as above to 26.5. Gradational change to flaser-bedded SANDSTONE w/ blacks shale interbeds. Kept 25.5-26.3 [illegible]
								Tagged 32.6'	0.3'	Lost	

Sheet 1 of 2 Sheets

WES FORM JAN 74 819 EDITION OF NOV 1971 MAY BE USED

BORING LOG
FIELD DATA

Project _____ Site _____ Date 9/25
Location _____ Job No. _____
Drill Rig _____ Inspector _____ Operator _____ Surface El _____ Boring No. 30-03

SAMPLE NUMBER	DATE TAKEN	STRATUM FROM	STRATUM TO	DRIVE FROM	DRIVE TO	SAMPLE FROM	SAMPLE TO	TYPE OF SAMPLER			CLASSIFICATION AND REMARKS
				32.6	42.6	41.4	42.3	HQ Core barrel	RUN 3 10.0	PULL 9.9	32.6-35.2 Flaser bedded sandstone with
			35.2					Taped 42.6	Lost	0.1	black shale interbeds. Below 35.2 is all
					Kept	41.4-42.3, discarded rest.					SANDSTONE, friable, tan, fine-med grained,
				Bailed hole, not making water							quartzitic, micaceous w/ some black min-
											eral grains, but 95% quartz, as in Borings
											30-01, 30-02. 0.5' red ironstone at 35.5-36.0'
											SANDSTONE is coarser, more friable below 39.4.
		35.2	51.6	42.6	51.6	42.6	51.6	HQ Core barrel	RUN 4 9.0	PULL 8.8	42.6-51.6 SANDSTONE as above. Streaked
								Taped 51.6	Lost	0.2'	w/ chocolate brown color 52.5-57.5 (severe
											iron oxide staining). Soft, friable.
		Bailed hole, Water at				45.5 @ 1628		9/24			
						47.1 @ 1631		9/24			
						39.7 @ 0737		9/25			
		Set 10' PVC Screen at 51.4 to 41.4.						Filter sand to	37.5',		bentonite pellets to 33.5.

WES FORM 819 EDITION OF NOV 1971 MAY BE USED

Sheet 2 of 2 Sheets

BORING LOG
FIELD DATA

Project: Landfarm Monitoring Wells Site: Landfarm, NSWC Date: 9/27/93
Location: SW Corner (Down slope) Job No.:
Drill Rig: Failing 500 Inspector: MURPHY Operator: BROWN/MIXON Surface El: ~703 Boring No.: 30-04

SAMPLE NUMBER	DATE TAKEN	STRATUM FROM	STRATUM TO	DRIVE FROM	DRIVE TO	SAMPLE FROM	SAMPLE TO	TYPE OF SAMPLER			CLASSIFICATION AND REMARKS
1	9/27	0.0		0.0	1.5	0.4	0.8	3" Split spoon	Jar	#1	Silt (ML) to silty clay (CL), probably loess. Tan, mottled.
			12.9	0.0	12.9			7" Fill Auger			0.0-12.9 Loess (ML) to silty clay (CL), grades w/ depth to reworked loess and rock frags (residual soil). Refusal at 12.9'
		Set 6" PVC Casing to 12.9' in 1 1/2' of bentonite at bottom, filled annulus of hole around casing to surface w/ bentonite.									
		12.9		12.9	23.1	12.9 and 22.7	14.3 and 23.1	HQ Core barrel Taped 23.1'	Run 1 10.2 Lost 3.6'	Pull 6.6	12.9-16.8 Interbedded black shale, ochre clay, tan shale, brown sandstone and hd red-brn [illegible] in holes 30-03, 30-02, 30-01. 0.1' black lignite coal at bottom of run. Core broken. RQD ~ Zero. Lost 3.6' total. Kept 12.9-14.3 and 22.7-23.1 in box, discarded rest
		Coring w/ water									
			27.2	23.1	33.1	23.1 and 32.0	25.2 32.7	HQ Core barrel Taped 32.7	Run 2 10.0 No Core 0.2' in hole	Pull 7.8 Loss	23.1-27.2 Interbdd brown SANDSTONE and black to gray shale (not well bdd). SANDSTONE is fine to med. grained, more friable below 31.9'. Kept 24.1-25.2 and 32.0-32.7, RQD better this run. (All SANDSTONE below 27.2)
		27.2									
		Bailed hole, water coming in									

WES FORM JAN 74 819 EDITION OF NOV 1971 MAY BE USED

Pull = Auger [illegible]

Sheet 1 of 2 Sheets

6.9

BORING LOG
FIELD DATA

Project _____ Site _____ Date _____
Location _____ Job No. _____
Drill Rig _____ Inspector _____ Operator _____ Surface El _____ Boring No. 30-04

SAMPLE NUMBER	DATE TAKEN	STRATUM FROM	STRATUM TO	DRIVE FROM	DRIVE TO	SAMPLE FROM	SAMPLE TO	TYPE OF SAMPLER			CLASSIFICATION AND REMARKS
			39.8	33.1	39.8	32.9	39.8	HQ Core barrel	Run 3 6.7	Pull 6.7	33.1-39.8 SANDSTONE as above. Below
								TAPED 39.8	0.2 Coreloss		38.9' color changes gradually to white. Sand-
											stone is medium-grained, friable. RQD is
			Bailed hole, let sit overnight								about 75% this run. A few bedding jts. Kept
											all of Run 3. Pull interval was 6.5', recov.
		Water at 22.2' TOG 0900, 09/28/93									6.7, 0.2' core loss.
	Set 10' wellscreen at 39.7 to 29.7. Added filter sand to								26.5',		
	added bentonite pellets to 23.5'										

ES FORM JAN 74 819 EDITION OF NOV 1971 MAY BE USED

Sheet 2 of 2 Sheets

BORING LOG
FIELD DATA

Project Landfarm Monitoring Wells Site Landfarm, NSWC Crane Date 9/28/93
Location West Side, adjacent Hiway 161 Job No.
Drill Rig Failing 1500 Inspector MURPHY Operator BROWN/MIXON Surface El ~ 704 Boring No. 30-05

SAMPLE NUMBER	DATE TAKEN	STRATUM FROM	STRATUM TO	DRIVE FROM	DRIVE TO	SAMPLE FROM	SAMPLE TO	TYPE OF SAMPLER			CLASSIFICATION AND REMARKS
1	9/28	0.0		0.0	1.5	0.5	0.8	3" Splitspoon	Jar #1		Loess (?) ML-CL, plastic, ochre to tan color.
			13.6	0.0	13.6	None		7" Flt Auger			0.0-13.6 Loess (?) ML-CL, probably reworked with residual soil. Pebble-sized rock frags below ~5'. Plastic. Rock at 13.6'
		Set 6" PVC casing to 13.6' in 1' bentonite (Hole Plug). Filled annulus w/ bentonite to surface.									
		13.6		13.6	23.1	13.6	22.7	HQ Core barrel	RUN 1 9.5	PULL 3.7	13.6-22.7 Interbdd tan-ochre soft, plastic clay and hd, fine-grnd red-brn SANDSTONE (ironstone). 5.4' of core lost in several badly broken zones. RQD v. low.
				Coring w/ water				Taped 22.7	0.4 in hole Lost 5.4'		
				23.1	33.1	22.7	32.5	"	RUN 2 10.0	PULL 9.6	22.7-24.0 Interbdd black shale and brown SANDSTONE as above. 24.0-32.5 is brn SANDSTONE, fine-med, coarser and more friable w/ depth. Very few black shale laminae. Core broken: RQD ~ 50%.
			24.0					Taped 32.5	0.6 in hole Lost 4.2'		
		24.0									
				33.1	42.6	32.5	42.5	"	RUN 3 9.5	PULL 9.9	32.5-42.5 Brn med-grnd SANDSTONE as above. Massive (bdding only apparent at breaks) RQD 100% except in bottom 4'. Much more
								Taped 42.5	0.1 in hole Lost 0.1'		

WES FORM JAN 74 819 EDITION OF NOV 1971 MAY BE USED

PULL = Amount Recov.
RUN = " cored.

Sheet 1 of 2 Sheets

BORING LOG
FIELD DATA

Project _____ Site _____ Date _____
Location _____ Job No. _____
Drill Rig _____ Inspector _____ Operator _____ Surface El _____ Boring No. 30-05

SAMPLE NUMBER	DATE TAKEN	STRATUM FROM	STRATUM TO	DRIVE FROM	DRIVE TO	SAMPLE FROM	SAMPLE TO	TYPE OF SAMPLER			CLASSIFICATION AND REMARKS
			42.5								friable in bottom 2', and coarser grained.
		Bailed hole, water came up to ~28' @1730, 9/28/93									
		Set screen at 42.5 to 32.5 (10'). Brought sand									
		up to 27.0 TOG, bentonite to 23.0' TOG									
		Water at 24.7' T.O.G. 0745, 09/29/93									

WES FORM 810 EDITION OF NOV 1971 MAY BE USED

Sheet 2 of 2 Sheets

BORING LOG
FIELD DATA

Project: Landfarm Monitoring Wells Site: Landfarm, NSWC Crane Date: 9/29/83
Location: South side, east Job No.:
Drill Rig: Failing Inspector: Murphy Operator: Brown/Mixon Surface El: ~708 Boring No.: 30-06

SAMPLE NUMBER	DATE TAKEN	STRATUM FROM	STRATUM TO	DRIVE FROM	DRIVE TO	SAMPLE FROM	SAMPLE TO	TYPE OF SAMPLER			CLASSIFICATION AND REMARKS
1	9/29	0.0		0.0	1.5	0.5	0.8	3" Splitspoon	Jar	#1	Reworked loess (?) ML-CL w/ rock frags. Little plasticity
			13.4	0.0	13.4	None		7" FH Auger			0.0-13.4 Tan to ochre silt ML w/ minor clay CL, loess (?) reworked w/ residual soil, w/ rock frags. Top of rock 13.4'
		Set 6" PVC Casing to 13.4' in 1' bentonite (Hole Plug). Filled annular of casing w/ bentonite to surface.									
		13.4		13.4	23.4	13.4	23.3	HQ Core barrel	Run 1 10.0	Pull 6.7	13.4-23.3 Interbdd black SHALE, soft ochre to gray clay, hard red-brn ironstone, and brown SANDSTONE as in other borings. RQD=0
								Taped 23.3	0.1 in Lost	hole 3.2'	Recov. 6.7' of 9.9', lost 3.2' in several zones.
				23.4	33.4	23.3	33.2	"	Run 2 10.0	Pull 9.2	23.3-25.7 Interbdd black SHALE, COAL and SANDSTONE, wavy or flaser bedded. 24.7-25.7 is
								Taped 33.2	0.2 in Lost	hole 0.7'	all gray SHALE and black SHALE, flaser bedded. Abrupt
			25.7								change to SANDSTONE at 25.7
		25.7	29.8								25.7-29.8 Brown, friable, med, gtzitic SANDSTONE
		29.8									29.8-33.2 Abrupt change to white to light gray, dense, fine grained, calcareous gtzitic SANDSTONE.
											Core loss probably in upper bed. Core in good condition

WES FORM JAN 74 819 EDITION OF NOV 1971 MAY BE USED

Run = Amount cored
Pull = Amount recov.

Sheet 1 of 2 Sheets

BORING LOG
FIELD DATA

Project _____ Site _____ Date _____
Location _____ Job No. _____
Drill Rig _____ Inspector _____ Operator _____ Surface El _____ Boring No. 30-06

SAMPLE NUMBER	DATE TAKEN	STRATUM FROM	STRATUM TO	DRIVE FROM	DRIVE TO	SAMPLE FROM	SAMPLE TO	TYPE OF SAMPLER			CLASSIFICATION AND REMARKS
											below top foot, RQD ~ 85%.
			34.5	33.4	43.4	33.2	42.8	HQ core	RUN 3 10.0	PULL 8.5	33.2-34.5 White, dense, calc. SANDSTONE
								Tapped 42.8	0.6 in Lost	hole 1.1'	as above. Sharp contact at 34.5 to brown
											SANDSTONE.
		34.5	42.8								34.5-42.8 Brown friable non-calc SAND-
											STONE as above 29.8, w/ occasional chocolate
		Bailed hole, water entering slowly.									brown stringers. One 0.15' gray shale bed
		Water at 26.6' T.O.G. 0700, 9/30/93									at 37.6'. Pulled from 33.2 to 42.8' (9.6'),
		Set screen 42.8-32.8' Brought sand up to 27.4' TOG.									recov. 8.5', lost 1.1'. Core is in short lengths
		Bentonite pellets to 24.0'									below 35.7 (RQD below 35.7 = 0).

WES FORM JAN 74 819 EDITION OF NOV 1971 MAY BE USED

Sheet 2 of _____ Sheets

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NOTE:

Boring No. Landfarm 30-07

Boring Log Field Data - Not available

Per Bill Murphy (Corps of Engineers Waterways Experiment Station)

APPENDIX B

FIELD FORMS

Note: Brown and Root Environmental was purchased on January 1, 1998 and became Tetra Tech NUS (TtNUS).

- Well Development Form
- Groundwater Level Measurement Sheet
- Groundwater Sample Log Sheet
- Equipment Calibration Log
- Chain-of-Custody Record

Brown & Root Environmental

WELL DEVELOPMENT SHEET

PROJECT NAME: _____ SITE/LOCATION: _____
PROJECT NUMBER: _____ MEASURING DEVICE: _____
PERSONNEL: _____ ADJUSTMENT FACTOR: _____
WEATHER: _____ DATE: _____
REMARKS: _____ WELL NO.: _____

☐ Domestic Well ☐ Monitoring Well
☐ Other Well _____

Static Water Level: _____
Total Depth: _____
One Casing Volume: _____

Method: _____
Start Time: _____
Complete Time: _____

Approx. Volume	Time	pH	Temp. (oC)	Turbidity	Color	Specific Conductance	Dissolved Oxygen

NOTE: All measurements to nearest 0.01 foot measured from top of well riser pipe unless otherwise noted.

Additional Comments: _____

Signature: _____ Page: _____ of: _____

GROUNDWATER LEVEL MEASUREMENT SHEET

Page ___ of ___

PROJECT NAME: _____ LOCATION: _____
PROJECT NUMBER: _____ MEASURING DEVICE: _____
PERSONNEL: _____ ADJUSTMENT FACTOR: _____
DATE: _____ REMARKS: _____
WEATHER CONDITIONS: _____

Well or Piezometer Number	Time	Elevation of Reference Point (Feet)*	Water Level Indicator Reading (Feet)*	Groundwater Elevation (Feet)*	Comments

*Measurements to nearest 0.01 foot.

Signature(s): _____

GROUNDWATER
SAMPLE LOG SHEET

Page ____ of ____

Project Site Name: _____

Project No.: _____

- ☐ Domestic Well Data
- ☐ Monitoring Well Data
- ☐ Other Well Type: _____
- ☐ QA Sample Type: _____

Sample ID No.: _____

Sample Location: _____

Sampled By: _____

C.O.C. No.: _____

Sampling Data

Date: _____ Time: _____ Method: _____	pH	S.C.	Temp. (°C)	Turbidity	Color	TBD	TBD	TBD

Purge Data

Date: _____ Method: _____	Volume	pH	S.C.	Temp.(°C)	Turbidity	Color	TBD	TBD
	Initial							
Monitor Reading (ppm):	1							
Well Casing Dia. & Material Type:	2							
	3							
Total Well Depth (TD):	4							
Static Water Level (WL):	5							
TD-WL (ft.) =								
One Casing Volume: (gal/L)								
Start Purge (hrs.):								
End Purge (hrs.):								
Total Purge Time (min):								
Total Amount Purged (gal/L):								

Analysis	Preservative	Container Requirements	Collected (✓)

Observations/Notes:

Circle if Applicable:

MS/MSD	Duplicate ID No:

Signature(s):

TBD: To Be Determined

Brown & Root Environmental

EQUIPMENT CALIBRATION LOG

INSTRUMENT NAME / MODEL : _____

JOB NAME : _____

MANUFACTURER : _____

JOB NUMBER : _____

CALIBRATION DATE	INITIAL SETTINGS	STANDARDS USED	PROCEDURE	ADJUSTMENTS MADE	FINAL SETTINGS	SIGNATURE	COMMENTS

CHAIN OF CUSTODY RECORD

PROJECT NO.:	SITE NAME:
SAMPLERS (SIGNATURE):	

STATION NO.	DATE	TIME	COMP	GRAB	STATION LOCATION	NO. OF CON-TAINERS							REMARKS

RELINQUISHED BY (SIGNATURE):	DATE / TIME:	RECEIVED BY(SIGNATURE):	RELINQUISHED BY (SIGNATURE):	DATE / TIME:	RECEIVED BY(SIGNATURE):
RELINQUISHED BY (SIGNATURE):	DATE / TIME:	RECEIVED BY (SIGNATURE):	RELINQUISHED BY (SIGNATURE):	DATE / TIME:	RECEIVED BY(SIGNATURE):
RELINQUISHED BY (SIGNATURE):	DATE / TIME:	RECEIVED FOR LABORATORY BY (SIGNATURE):	DATE / TIME:	REMARKS:	

Order No. 70440 (0693)

APPENDIX C

STANDARD OPERATING PROCEDURES

Note: Brown and Root Environmental was purchased on January 1, 1998 and became Tetra Tech NUS (TtNUS).

CT-04	Sample Nomenclature
GH-1.2	Evaluation of Existing Monitoring Wells and Water Level Measurement
GH-2.5	Water Level Measurement and Groundwater Contour Mapping
GH-2.8	Groundwater Monitoring Point Installation
SA-1.1	Groundwater Sample Acquisition and Onsite Water Quality Testing
SA-6.1	Non-Radiological Sampling Handling
SA-6.3	Field Documentation
SA-7.1	Decontamination of Field Equipment and Waste Handling
ME-12	Photovac 2020 Photoionization Air Monitor
ME-15	Photovac Micro-FID Handheld Flame Ionization Detector

TABLE OF CONTENTS

SECTION		PAGE
1.0	PURPOSE	2
2.0	SCOPE	2
3.0	GLOSSARY	2
4.0	RESPONSIBILITIES	2
5.0	PROCEDURES	3
5.1	Introduction	3
5.2	Sample Number Field Requirements	3
5.3	Example Sample Field Designations	4
5.4	Example Sample Numbers	6

1.0 PURPOSE

The purpose of this document is to specify a consistent sample nomenclature system that will facilitate subsequent data management in a cost-effective manner. The sample nomenclature system has been devised such that the following objectives can be attained:

- Sorting of data by matrix.
- Sorting of data by depth.
- Maintenance of consistency (field, laboratory, and data base sample numbers).
- Accommodation of all project-specific requirements on a global basis.
- Accommodation of laboratory sample number length constraints (10 characters).

2.0 SCOPE

The methods described in this procedure shall be used consistently for all projects requiring electronic data handling managed by personnel located in the Northeast Region of Brown & Root Environmental (Pittsburgh, Wayne, Holt, and Wilmington) and for any large contracts managed by the Northeast Region (e.g., NORTHDIV CLEAN, SOUTHDIV CLEAN, ARCS I, ARCS III, etc.). Smaller projects (as determined by Project Manager) are outside the scope of this SOP.

3.0 GLOSSARY

None.

4.0 RESPONSIBILITIES

Program Manager - It shall be the responsibility of the Program Manager (or designee) to inform contract-specific Project Managers of the existence and requirements of this Standard Operating Procedure.

Project Manager - It shall be the responsibility of the Project Manager to determine the applicability of this Standard Operating Procedure based on: (1) program-specific requirements, and (2) project size and objectives. It shall be the responsibility of the Project Manager (or designee) to ensure that the sample nomenclature is thoroughly specified in the relevant project planning document (e.g., sampling and analysis plan) and is consistent with this Standard Operating Procedure if relevant. It shall be the responsibility of the project manager to ensure that the Field Operations Leader is familiar with the sample nomenclature system.

Field Operations Leader - It shall be the responsibility of the Field Operations Leader to ensure that all field technicians or sampling personnel are thoroughly familiar with this Standard Operating Procedure and the project-specific sample nomenclature system. It shall be the responsibility of the Field Operations Leader to ensure that the sample nomenclature system is used during all project-specific sampling efforts.

5.0 PROCEDURES

5.1 Introduction

The sample numbering system consists of 12 distinct alpha-numeric characters, only 10 of which will be provided to the laboratory on the sample labels and chain-of-custody forms. The sample number provided to the lab shall be as follows where "A" indicates "alpha," "N" indicates "numeric," and "E" indicates "either"):

$$\overline{E}\ \overline{E}\ \overline{E}\ \overline{A}\ \overline{A}\ \overline{E}\ \overline{E}\ \overline{E}\ \overline{N}\ \overline{N}$$

Once the analytical results are received from the laboratory the sample number will be revised by a subroutine such that the sample number is more user friendly (i.e., dashes will be inserted). The sample number will then appear as follows:

$$\overline{E}\ \overline{E}\ \overline{E}\ -\ \overline{A}\ \overline{A}\ -\ \overline{E}\ \overline{E}\ \overline{E}\ -\ \overline{N}\ \overline{N}$$

If multiple sampling events occur (or are planned) for a given matrix, a subroutine within the database will be used to append two additional characters such that the sample number will appear as follows:

$$\overline{E}\ \overline{E}\ \overline{E}\ -\ \overline{A}\ \overline{A}\ -\ \overline{E}\ \overline{E}\ \overline{E}\ -\ \overline{N}\ \overline{N}\ -\ \overline{N}\ \overline{N}$$

Site Type Location Depth Round

5.2 Sample Number Field Requirements

The various fields in the sample number will include the following:

- Site Identifier
- Sample Type
- Sample Location
- Sample Depth Indicator
- Sampling Round

The site identifier must be a three-character field (numeric characters, alpha characters, or a mixture of alpha and numeric characters may be used). A site number is necessary since many facilities/sites have multiple individual sites, SWMUs, operable units, etc.

The sample type must be a two-character alpha field. Suggested codes are provided in Section 5.3 of this SOP.

The sample location must be a three-character field (alpha, numeric, or a mixture).

The depth field must be provided for all samples, regardless if it is strictly applicable (as discussed in Section 5.3).

The sampling round is optional, but, if provided, must be two numeric characters.

5.3 Example Sample Field Designations

Examples of each of the fields are as follows:

Site Number - Examples of site numbers/designations are as follows:

A01 - Area of Concern Number 1
125 - Solid Waste Management Unit Number 125
000 - Base or Facility Wide Sample (e.g., upgradient well)
BBG - Base Background

The examples cited are only suggestions. Each Project Manager (or designee) must designate appropriate (and consistent) site designations for their individual project.

Sample Type - Examples of sample types are as follows:

AS - Air Sample
BS - Biota Sample (See Note)
CP - Composite Sample
CS - Chip Sample
DS - Drum Sample
DU - Dust Sample
FP - Free Product
ID - Investigation Derived Waste Sample
LT - Leachate Sample
MW - Monitoring Well
OF - Outfall Sample
RW - Residential Well Sample
SB - Soil Boring Sample
SD - Sediment Sample
SC - Scrape Sample
SG - Soil Gas Sample
SP - Seep Sample
SS - Surface Soil Sample
SU - Subsurface Soil Sample
SW - Surface Water Sample
TP - Test Pit Sample
TW - Temporary Well Sample
WC - Well Construction Material Sample
WI - Wipe Sample
WP - Well Point Sample
WS - Waste/Sludge Sample

Note: The biota sample designation may be contingent upon the type of biota sampled (e.g., BL - Lobster; BF - Finfish; BC - Clam; BO - Oyster). Numerous other examples can be cited but will be site-specific.

This field will also be used to designate field Quality Control Samples, as follows:

TB - Trip Blank
FB - Field Blank
RB - Rinsate Blank (Equipment Blank)
BB - Bottle Blank
AB - Ambient Condition Blank

Field quality control samples should be numbered sequentially (e.g., RB-001; FB-010, etc.).

Filtered/unfiltered surface water or groundwater samples shall be handled in an separate manner, as subsequently discussed.

Location - Examples of the location field are as follows:

A01 - Grid node A1
001 - Monitoring Well 1

It is important that consistency be maintained with respect to the use of the characters "0" and O. Data base subroutines will not sort correctly if a mixture are used (e.g, AO1 and A02).

Depth - Formerly, depth specifications were indicated with a four digit field (e.g., 0002 - 0 to 2 feet). While this is effective for depth sorting, it is difficult to include this level of detail in a 10-character lab number (FormMaster limitations). In addition, this approach will not accommodate non-integer depths (e.g., 2.5 feet to 4.5 feet).

Based on such potential problems, the following approach shall be used: Sample depths will simply represent the horizon from which the sample was obtained: For example, if ten split-spoon samples are collected from a boring, they will be numbered 01 through 10. The sample log sheet will be used to record the specific depth of the sample, and this information will be entered in a separator field in the data base.

Similar nomenclature will be used for depth-specific surface water and sediment samples, etc. If no depth information is required (e.g., groundwater samples), the field must still be filled (e.g., Ø, Ø).

This field will also be used for the designation of filtered and unfiltered samples. An unfiltered groundwater sample shall be designated as U0, if and only if, a corresponding filtered sample is collected. Such as sample shall be designated as F0.

Sampling Round - The sampling round field is straightforward. It can range from 01 to 99.

5.4 Example Sample Numbers

Examples of complete sample numbers (field/data base versus laboratory) are as follows:

Field/Data Base ID	Lab ID	Description
101-SB-A01-01	101SBA0101	The first sample (e.g., 0 to 2 feet) from soil boring A01 (grid) at Site 101.
101-SB-A01-02	101SBA0102	The second sample from boring A01 (could be the next depth interval or a duplicate of 101-SB-A01-01).
125-MW-001-01-01	125MW00101	A groundwater sample from monitoring well MW001 (first sampling round)
125-MW-001-02-01	125MW00102	A duplicate groundwater sample from monitoring well MW001 (first sampling round)
130-MW-003-U1-01	130MW003U1	An unfiltered groundwater sample from monitoring well MW003 (first sampling round)
130-MW-003-F1-01	130MW003F1	A filtered groundwater sample from monitoring well MW003 (first sampling round)
137-RB-001-00-01	137RB00100	The first rinsate blank collected at site 137.
137-TB-004-00-02	137TB00400	The fourth trip blank collected during the second sampling event at Site 137.
155-SW-003-01-01	155SW00301	A surface water sample collected from the surface of a pond at Site 155.
155-SW-003-02-01	155SW00302	A surface water sample collected from the bottom of the water column in a pond at Site 155.

TABLE OF CONTENTS

SECTION **PAGE**

1.0 PURPOSE 2

2.0 SCOPE 2

3.0 GLOSSARY 2

4.0 RESPONSIBILITIES 2

5.0 PROCEDURES 2

5.1 Preliminary Evaluation 3
5.2 Field Inspection 3
5.3 Water Level (Hydraulic Head) Measurements 4
5.3.1 General 4
5.3.2 Water Level Measuring Techniques 5
5.3.3 Methods 5
5.3.4 Water Level Measuring Devices 6
5.3.5 Data Recording 8
5.3.6 Specific Quality Control Procedures for Water Level Measuring Devices 8
5.4 Health and Safety Considerations 8

6.0 RECORDS 8

ATTACHMENTS

A GROUNDWATER LEVEL MEASUREMENT SHEET 9

1.0 PURPOSE

The purpose of this procedure is to provide reference information regarding the proper methods for evaluating existing monitoring wells, and determining water level measurements.

2.0 SCOPE

The procedures described herein are applicable to all existing monitoring wells and, for the most part, are independent of construction materials and methods.

3.0 GLOSSARY

Hydraulic Head - The height to which water will rise in a well.

Water Table - A surface in an unconfined aquifer where groundwater pressure is equal to atmospheric pressure (i.e., the pressure head is zero).

4.0 RESPONSIBILITIES

Site Geologist/Hydrogeologist - Has overall responsibility for obtaining water level measurements and developing groundwater contour maps. The site geologist/hydrogeologist (in concurrence with the Project Manager) shall specify the reference point from which water levels are measured (usually a specific point on the upper edge of the inner well casing), the number of data points needed and which wells shall be used for a contour map, and how many complete sets of water levels are required to adequately define groundwater flow directions (e.g., if there are seasonal variations).

Field Personnel - Must have a basic familiarity with the equipment and procedures involved in obtaining water levels, and must be aware of any project-specific requirements.

5.0 PROCEDURES

Accurate, valid and useful groundwater monitoring requires that four important conditions be met:

- Proper characterization of site hydrogeology.
- Proper design of the groundwater monitoring program, including adequate numbers of wells installed at appropriate locations and depths.
- Satisfactory methods of groundwater sampling and analysis to meet the data quality objectives (DQOs).
- The assurance that specific monitoring well samples are representative of water quality conditions in the monitored interval.

To insure that these conditions are met, adequate descriptions of subsurface geology, well construction methods and well testing results must be available. The following steps will help to insure that the required data are available to permit an evaluation of the utility of existing monitoring wells for collecting additional samples.

5.1 Preliminary Evaluation

A necessary first step in evaluating existing monitoring well data is the study and review of the original work plan for monitoring well installation (if available). This helps to familiarize the site geologist/hydrogeologist with site-specific conditions, and will promote an understanding of the original purpose of the monitoring wells.

The next step of the evaluation should involve a review of all available information concerning borehole drilling and well construction. This will allow interpretation of groundwater flow conditions and area geology, and will help to establish consistency between hydraulic properties of the well and physical features of the well or formation. The physical features which should be identified and detailed, if available, include:

- The well identification number, permit number and location by referenced coordinates, the distance from prominent site features, or the location of the well on a map.
- The installation dates, drilling methods, well development methods, and contractors.
- The depth to bedrock -- where rock cores were not taken, auger refusal, drive casing refusal or penetration test results (blow counts for split-barrel sampling) may be used to estimate bedrock interface.
- The soil profile and stratigraphy.
- The borehole depth and diameter.
- The elevation of the top of the protective casing, the top of the well riser, and the ground surface.
- The total depth of the well.
- The type of well materials, screen type, slot size, and length, and the elevation/depths of the screen, interval, and/or monitored interval.
- The elevation/depths of the tops and bottom of the filter pack and well seals and the type and size.

5.2 Field Inspection

During the onsite inspection of existing monitoring wells, features to be noted include:

- The condition of the protective casing, cap and lock.
- The condition of the cement seal surrounding the protective casing.
- The presence of depressions or standing water around the casing.
- The presence of any electrical cable and its connections.

If the protective casing, cap and lock have been damaged or the cement collar appears deteriorated, or if there are any depressions around the well casing capable of holding water, surface water may have

infiltrated into the well. This may invalidate previous sampling results since the time when leakage started is unknown.

The routine physical inspection must be followed by a more detailed investigation to identify other potential routes of contamination or sampling equipment malfunction. Any of these occurrences may invalidate previously-collected water quality data. If the monitoring well is to be used in the future, considerations shown in the steps described above should be rectified to rehabilitate the well. After disconnecting any wires, cables or electrical sources, remove the lock and open the cap. Check for the presence of organic vapors with a photoionization detector (PID) or flame-ionization detector (FID) and combustible gas meter to determine the appropriate worker safety level. The following information should be noted:

- Cap function.
- Physical characteristics and composition of the inner casing or riser, including inner diameter and annular space.
- Presence of grout between the riser and outer protective casing and the existence of drain holes in the protective casing.
- Presence of a riser cap, method of attachment to casing, and venting of the riser.
- Presence of dedicated sampling equipment; if possible, remove such equipment and inspect size, materials of construction and condition.

The final step of the field inspection is to confirm previous hydraulic or physical property data and to obtain data not previously available. This includes the determination of static water levels, total well depth and well obstruction. This may be accomplished using a weighted tape measure which can also be used to check for sediment (the weight will advance slowly if sediment is present, and the presence of sediment on the weight upon removal should be noted). If sediment is present, the well be should be redeveloped before sampling.

Lastly, as a final step, the location, condition and expected water quality of the wells should be reviewed in light of their usefulness for the intended purpose of the investigation.

5.3 Water Level (Hydraulic Head) Measurements

5.3.1 General

Groundwater level measurements can be made in monitoring wells, private or public water wells, piezometers, open boreholes, or test pits (after stabilization). Groundwater measurements should generally not be made in boreholes with drilling rods or auger flights present. If groundwater sampling activities are to occur, groundwater level measurements shall take place prior to well evacuation or sampling.

All groundwater level measurements shall be made to the nearest 0.01 foot, and recorded in the site geologist/hydrogeologist's field notebook or on the Groundwater Level Measurement Sheet (Attachment A), along with the date and time of the reading. The total depth of the well shall be measured and recorded, if not already known. Weather changes that occur over the period of time during which water levels are being taken, such as precipitation and barometric pressure changes, should be noted.

In measuring groundwater levels, there shall be a clearly-established reference point of known elevation, which is normally identified by a mark on the upper edge of the inner well casing. The reference point shall be noted in the field notebook. To be useful, the reference point should be tied in with an established USGS benchmark or other properly surveyed elevation datum. An arbitrary datum could be used for an isolated group of wells, if necessary.

Cascading water within a borehole or steel well casings can cause false readings with some types of sounding devices (chalked line, electrical). Oil layers may also cause problems in determining the true water level in a well. Special devices (interface probes) are available for measuring the thickness of oil layers and true depth to groundwater, if required.

Water level readings shall be taken regularly, as required by the site geologist/hydrogeologist. Monitoring wells or open-cased boreholes that are subject to tidal fluctuations should be read in conjunction with a tidal chart (or preferably in conjunction with readings of a tide staff or tide level recorder installed in the adjacent water body); the frequency of such readings shall be established by the site hydrogeologist. All water level measurements at a site used to develop a groundwater contour map shall be made in the shortest practical time to minimize affects due weather changes, and at least during the same day.

5.3.2 Water Level Measuring Techniques

There are several methods for determining standing or changing water levels in boreholes and monitoring wells. Certain methods have particular advantages and disadvantages depending upon well conditions. A general description of these methods is presented, along with a listing of various advantages and disadvantages of each technique. An effective technique shall be selected for the particular site conditions by the site geologist/hydrogeologist.

In most instances, preparation of accurate potentiometric surface maps require that static water level measurements be obtained to a precision of 0.01 feet. To obtain such measurements in individual accessible wells, chalked tape or electrical water level indicator methods have been found best, and thus are the most often utilized. Other, less precise methods, such as the popper or bell sound, or bailer line methods, may be appropriate for developing preliminary estimates of hydraulic conditions. When a large number of (or continuous) readings are required, time-consuming individual readings are not usually feasible. In such cases, it is best to use the float recorder or pressure transducer methods. When conditions in the well limit readings (i.e., turbulence in the water surface or limited access through small diameter tubing), less precise, but appropriate methods such as the air line or capillary tubing methods can be used (see subseuqent SOP section for discussion of these devices).

5.3.3 Methods

Water levels can be measured by several different techniques, but the same steps shall be followed in each case. The proper sequence is as follows:

1. Check operation of recording equipment above ground. Prior to opening the well, don personal protective equipment, as required.

2. Record all information specified below in the geologist/hydrogeologist's field notebook or on the Groundwater Level Measurement Sheet (Attachment A):

   - Well number.

- Water level (to the nearest 0.01 foot; 0.3 cm). Water levels shall be taken from the surveyed reference mark on the top edge of the inner well casing.

- Time and day of the measurement.

Water level measuring devices with permanently marked intervals shall be used when possible. If water level measuring devices marked by metal or plastic bands clamped at intervals along the measuring line are used, the spacing and accuracy of these bands shall be checked frequently as they may loosen and slide up or down the line, resulting in inaccurate reference points (see Section 5.3.6).

5.3.4 Water Level Measuring Devices

Chalked Steel Tape

The water level is measured by chalking a weighted steel tape and lowering it a known distance (to any convenient whole foot mark) into the well or borehole. The water level is determined by subtracting the wetted chalked mark from the total length lowered into the hole.

The tape shall be withdrawn quickly from the well because water has a tendency to rise up the chalk due to capillary action. A water finding paste may be used in place of chalk. The paste is spread on the tape the same way as the chalk, and turns red upon contacting water.

Disadvantages to this method include the following: depths are limited by the inconvenience of using heavier weights to properly tension longer tape lengths; ineffective if borehole/well wall is wet or inflow is occurring above the static water level; chalking the tape is time-consuming; difficult to use during periods of precipitation.

Electric Water Level Indicators

These devices consist of a spool of small-diameter cable and a weighted probe attached to the end. When the probe comes in contact with the water, an electrical circuit is closed and a meter, light, and/or buzzer attached to the spool will signal the contact.

There are a number of commercial electric sounders available, none of which is entirely reliable under all conditions likely to occur in a contaminated monitoring well. In conditions where there is oil on the water, groundwater with high specific conductance, water cascading into the well, steel well casing, or a turbulent water surface in the well, measuring with an electric sounder may be difficult.

For accurate readings, the probe shall be lowered slowly into the well. The electric tape is marked at the measuring point where contact with the water surface was indicated. The distance from the mark to the nearest tape band is measured using an engineer's folding ruler or steel tape, and added to the band reading to obtain the depth to water. If the band is not a permanent marking band, spacing shall be checked periodically as described in Section 5.3.6.

Popper or Bell Sounder

A bell- or cup-shaped weight that is hollow on the bottom is attached to a measuring tape and lowered into the well. A "plopping" or "popping" sound is made when the weight strikes the surface of the water. An accurate reading can be determined by lifting and lowering the weight in short strokes, and reading the tape when the weight strikes the water. This method is not sufficiently accurate to obtain water levels to 0.01 feet, and thus is more appropriate for obtaining only approximate water levels quickly.

Float Recorder

A float or an electromechanically actuated water-seeking probe may be used to detect vertical changes of the water surface in the hole. A paper-covered recording chart drum is rotated by the up and down motion of the float via a pulley and reduction gear mechanism, while a clock drive moves a recording pen horizontally across the chart. To ensure continuous records, the recorder shall be inspected, maintained, and adjusted periodically. This type of device is useful for continuously measuring periodic water level fluctuations, such as tidal fluctuations or influences of pumping wells.

Air Line

An air line is especially useful in pumped wells where water turbulence may preclude the use of other devices. A small-diameter weighted tube of known length is installed from the surface to a depth below the lowest water level expected. Compressed air (from a compressor, bottled air, or air pump) is used to purge the water from the tube, until air begins to escape the lower end of the tube, and is seen (or heard) to be bubbling up through the water in the well. The pressure needed to purge the water from the air line multiplied by 2.307 (feet of water for 1 psi) equals the length in feet of submerged air line. The depth to water below the center of the pressure gauge can be calculated by subtracting the length of air line below the water surface from the total length of the air line.

The disadvantages to this method include the need for an air supply and lower level of accuracy (unless a very accurate air pressure gauge is used, this method cannot be used to obtain water level readings to the nearest 0.01 ft).

Capillary Tubing

In small diameter piezometer tubing, water levels are determined by using a capillary tube. Colored or clear water is placed in a small "U"-shaped loop in one end of the tube (the rest of the tube contains air). The other end of the capillary tube is lowered down the piezometer tubing until the water in the loop moves, indicating that the water level has been reached. The point is then measured from the bottom of the capillary tube or recorded if the capillary tube is calibrated. This is the best method for very small diameter tubing monitoring systems such as Barcad and other multilevel systems. Unless the capillary tube is calibrated, two people may be required to measure the length of capillary tubing used to reach the groundwater. Since the piezometer tubing and capillary tubing usually are somewhat coiled when installed, it is difficult to accurately measure absolute water level elevations using this method. However, the method is useful in accurately measuring differences or changes in water levels (i.e., during pumping tests).

Pressure Transducer

Pressure transducers can be lowered into a well or borehole to measure the pressure of water and therefore the water elevation above the transducer. The transducer is wired into a recorder at the surface to record changes in water level with time. The recorder digitizes the information and can provide a printout or transfer the information to a computer for evaluation (using a well drawdown/recovery model). The pressure transducer should be initially calibrated with another water level measurement technique to ensure accuracy. This technique is very useful for hydraulic conductivity testing in highly permeable material where repeated, accurate water level measurements are required in a very short period of time. A sensitive transducer element is required to measure water levels to 0.01 foot accuracy.

Borehole Geophysics

Approximate water levels can be determined during geophysical logging of the borehole (although this is not the primary purpose for geophysical logging and such logging is not cost effective if used only for this purpose). Several logging techniques will indicate water level. Commonly-used logs which will indicate saturated/unsaturated conditions include the spontaneous potential (SP) log and the neutron log.

5.3.5 Data Recording

Water level measurements, time, data, and weather conditions shall be recorded in the geologist/hydrogeologist's field notebook or on the Groundwater Level Measurement Sheet. All water level measurements shall be measured from a known reference point. The reference point is generally a marked point on the upper edge of the inner well casing that has been surveyed for an elevation. The exact reference point shall be marked with permanent ink on the casing since the top of the casing may not be entirely level. It is important to note changes in weather conditions because changes in the barometric pressure may affect the water level within the well.

5.3.6 Specific Quality Control Procedures for Water Level Measuring Devices

All groundwater level measurement devices must be cleaned before and after each use to prevent cross contamination of wells. Manufacturer's instructions for cleaning the device shall be strictly followed. Some devices used to measure groundwater levels may need to be calibrated. These devices shall be calibrated to 0.01 foot accuracy and any adjustments/corrections shall be recorded in the field logbook/notebook. After the corrections/adjustments are made to the measuring device and entered in the field logbook/notebook, the corrected readings shall be entered onto the Groundwater Level Measuring Sheet (Attachment A). Elevations will be entered on the sheet when they become available.

5.4 Health and Safety Considerations

Groundwater contaminated by volatile organic compounds may release toxic vapors into the air space inside the well pipe. The release of this air when the well is initially opened is a health/safety hazard which must be considered. Initial monitoring of the well headspace and breathing zone concentrations using a PID (e.g., HNu) or FID (e.g., OVA) and combustible gas meters shall be performed to determine required levels of protection.

6.0 RECORDS

A record of all field procedures, tests and observations must be recorded in the site logbook or designated field notebook. Entries in the log/notebook should include the individuals participating in the field effort, and the date and time. The use of annotated sketches may help to supplement the evaluation.

ATTACHMENT A
EXAMPLE GROUNDWATER LEVEL MEASUREMENT SHEET

GROUNDWATER LEVEL MEASUREMENT SHEET

Page ___ of ___

PROJECT NAME: _____ LOCATION: _____
PROJECT NUMBER: _____ MEASURING DEVICE: _____
PERSONNEL: _____ ADJUSTMENT FACTOR: _____
DATE: _____ REMARKS: _____
WEATHER CONDITIONS: _____

Well or Piezometer Number	Time	Elevation of Reference Point (Feet)*	Water Level Indicator Reading (Feet)*	Groundwater Elevation (Feet)*	Comments

*Measurements to nearest 0.01 foot.

Signature(s): _____

TABLE OF CONTENTS

SECTION		PAGE
1.0	PURPOSE	2
2.0	SCOPE	2
3.0	GLOSSARY	2
4.0	RESPONSIBILITIES	3
5.0	PROCEDURES	3
5.1	Potentiometric Surface Mapping	3
5.1.1	Selection of Wells	3
5.1.2	Construction of Equipotential Lines	4
5.1.3	Determination of Groundwater-Flow Direction	4
5.2	Groundwater Flow Considerations	4
5.3	Determination of Flow Rate	5
6.0	REFERENCES	7

ATTACHMENTS

A	GENERALIZED POROSITY AND HYDRAULIC CONDUCTIVITY VALUES FOR GEOLOGIC MATERIALS	8

1.0 PURPOSE

The purpose of this procedure is to provide a basic understanding of developing contour maps and the approaches used to identify and quantify the direction and rate of groundwater flow and contaminant plume movement.

2.0 SCOPE

This procedure provides only a general overview of the field techniques, mathematical and physical relationships and data handling procedures used for determining groundwater flow direction and rate. The references identified herein can provide a more complete explanation of particular methods cited, as well as a more comprehensive discussion on the interpretation of hydrogeologic data.

3.0 GLOSSARY

Aquifer - A geologic formation capable of transmitting usable quantities of groundwater to a well or other discharge point.

Aquitard - A geologic formation which retards the flow of groundwater due to its low permeability.

Confined Aquifer - An aquifer that is overlain and underlain by zones of lower permeability (aquitards). If the aquifer is "artesian," the potentiometric head of the aquifer at a given point is higher than the top of the zone comprising the aquifer at that point.

Equipotential Line - A line connecting points of equal elevation of the water table or potentiometric surface. Equipotential lines on the water table are also called water table contour lines.

Flow Line - A flow line indicates the direction of groundwater movement within the saturated zone. Flow lines are drawn perpendicular to equipotential lines.

Flow Net - A diagram of groundwater flow showing flow lines and equipotential lines.

Hydraulic Conductivity (K) - A parameter relating the volume of fluid flowing through a cross-sectional area of a saturated permeable medium to the driving force for the flow (hydraulic gradient). The hydraulic conductivity is a function both of the aquifer characteristics (porosity and interconnection of pores) and of the characteristics of the fluid (density and viscosity) passing through the aquifer. Thus, an aquifer will have a different hydraulic conductivity for water compared to pure-phase organic liquids.

Hydraulic Gradient (i) - The rate of change of hydraulic head per unit distance of flow at a given point and in the downgradient direction.

Hydraulic Head - The height to which water will rise inside a well casing, equal to the elevation head plus the pressure head. In a well screened across the water table, hydraulic head equals the elevation head, as the pressure head equals 0. In wells screened below the water table in an unconfined aquifer or screened at any interval within a confined aquifer, the head is the sum of the elevation of the aquifer (the elevation head) and the fluid pressure of the water confined in the aquifer (the pressure head).

Permeability - The capacity of a porous medium to transmit water. The degree of permeability depends on the size and shape of the pores, and the extent of their interconnections.

Porosity - Percentage of the total volume of a rock or soil which is occupied by voids (pore space).

Potentiometric (piezometric) Surface - A hypothetical surface that coincides with the static level of the water in aquifer (i.e., the maximum elevation to which water will rise in a well or piezometer penetrating the aquifer). The term "potentiometric surface" is usually applied to confined aquifers, although the water table is the potentiometric surface of an unconfined aquifer.

Unconfined Aquifer - An aquifer in which the water table forms the upper boundary.

Water Table - The surface in the groundwater system at which the fluid pressure is equal to atmospheric pressure (i.e., the net pressure head is zero) and below which all strata are saturated with water.

4.0 RESPONSIBILITIES

Project Hydrogeologist - The project hydrogeologist has overall responsibility for obtaining water level measurements and developing groundwater contour maps. The hydrogeologist (with the concurrence of the Project Manager) shall specify the reference point from which water levels are measured (usually a specific point on the upper edge of the inner well casing), the number of data points needed and which wells shall be used for a contour map, and how many complete sets of water levels are required to adequately define groundwater flow directions (e.g., if there are seasonal variations).

Field Personnel - All supporting field personnel must have a basic familiarity with the equipment and procedures involved in obtaining water levels, and must be aware of any project-specific requirements.

5.0 PROCEDURES

5.1 Potentiometric Surface Mapping

5.1.1 Selection of Wells

All wells used to prepare a flow net in a plan or map view should represent the same hydrogeologic unit, be it aquifer or aquitard. All water level measurements used shall be collected on the same day, preferably within 2-3 hours. This is especially important when working in an area where groundwater levels are tidally influenced or influenced by pumping.

Before mapping, review the recorded water levels and monitoring-well construction data, site geology and topographic setting must be reviewed to ascertain that the wells are completed in the same hydrogeologic unit and to determine if strong vertical hydraulic gradients may be present. Such conditions will be manifested by a pronounced correlation between well depth and water level, or by a difference in water level between two wells located near each other but set to different depths or having different screen lengths. Professional judgment of the hydrogeologist is important in this determination. If vertical gradients are significant, the data to be used must be limited vertically, and only wells finished in a chosen vertical zone of the hydrogeologic unit can be used.

At least three wells must be used to provide an estimation of the direction of groundwater flow; information from many more wells are needed to provide an accurate contour map. Generally, shallow systems require data from more wells than deep systems for accurate contour mapping.

5.1.2 Water Level Measurements

After selection of the wells to be used for mapping, the next step in determining the direction of groundwater flow is to obtain water level elevations from the selected points. In addition, any other

readily available wells/surface water bodies should also be measured to ensure that sufficient data are available for interpretation purposes.

Elevations are obtained from measurements of the depth to water in a monitoring well or piezometer taken from the top of the well casing (see SOP GH-1.2) and then referencing the elevation of the casing to a chosen and consistent datum point, usually mean sea level. Subtracting the depth to water from the casing elevation provides the elevation of the potentiometric surface. Elevations of points and areas of groundwater discharge or recharge such as springs, seeps, streams, rivers, and lakes also need to be determined, typically through staff gauge measurements. Comparison of these elevations, which represent hydraulic heads, will reveal the direction of flow because groundwater flows from areas of high head to areas of low head.

5.1.3 Construction of Equipotential Lines

Graphical methods available for depicting the flow of groundwater include the use of equipotential lines and flow lines to construct potentiometric surface maps and vertical flow nets. If the hydrogeologic system consists of a water table aquifer and one or more confined aquifers, separate contour maps should be prepared for each aquifer system. Water table maps should be developed using water level measurements obtained from monitoring wells screened at the unsaturated-saturated interface. Water level measurements collected from monitoring wells screened in the deeper portions of an unconfined aquifer should generally be contoured as a separate potentiometric surface map. Surface water discharge or recharge features are contoured in the water table system. Vertical flow nets should be constructed using a cross section aligned parallel to the direction of groundwater flow. All water level measurements along this cross section, both deep and shallow, are used in developing equipotential lines and flow lines for the flow net.

To construct equipotential lines, water elevations in the chosen wells are plotted on a site map. Other hydrogeologic features associated with the zone of interest -- such as seeps, wetlands, and surface-water bodies -- should also be plotted along with their elevations.

The data should then be contoured, using mathematically valid and generally accepted techniques. Linear interpolation is the most commonly used technique. However, quadratic interpolation or any technique of trend-surface analysis or data smoothing is acceptable. Computer-generated contour maps may be useful for large data sets. Contour lines shall be drawn as smooth, continuous lines which never cross one another.

Inspect the contour map, noting known features, such as pumping wells and site topography. The contour lines must be adjusted utilizing the professional judgment of the hydrogeologist in accordance with these features. Closed contours should be avoided unless a known sink exists. Groundwater mounding is common under landfills and lagoons; if the data imply this, the feature must be evident in the contour plot.

5.1.4 Determination of Groundwater-Flow Direction

Flow lines shall be drawn so that they are perpendicular to equipotential lines. Flow lines will begin at high head elevations and end at low head elevations. Closed highs will be the source of additional flow lines. Closed depressions (i.e., wells) will be the termination of some flow lines. Care must be used in areas with significant vertical gradients to avoid erroneous conclusions concerning gradients and flow directions.

5.2 Groundwater Flow Considerations

Groundwater movement is an integral part of the hydrologic cycle. Recharge to the shallow groundwater environment generally occurs by infiltration of precipitation through an upper unsaturated soil zone. Movement is downward under the force of gravity until the water reaches the saturated zone of the water table aquifer. Once water is part of the water table aquifer, movement is controlled by differences in hydraulic head with movement from areas of high head to areas of low head. Areas of low head include natural discharge areas such as springs, lakes, rivers, and, ultimately, the ocean. These features can be considered as outcrops of the water table. Points of low head also are created by pumping wells.

Local head differences and consequent vertical flow patterns within an aquifer can be detected by well clusters. A well cluster consists of several adjacent wells, generally installed within a few feet of each other, and screened at different depths. Variations in water levels in these closely spaced wells indicates the vertical component of groundwater flow within an aquifer, provided that the wells are all screened within the same aquifer.

The number, location, and extent of geologic units and their properties with regard to aquifer or aquitard characteristics must be understood to properly interpret water level data gathered from the monitoring system. This firm understanding of the hydrogeologic system must be developed through a program of soil borings and interpretation of subsurface geology. The adequacy of the positions and depths of borings used to define relevant subsurface hydrogeologic conditions must also be assessed. The location of surface water discharge or recharge points must be considered. Surface water features influence the system, as flow is most likely toward them (if they are discharge points) or away from them (if they are recharge points). Man-made discharge or recharge features such as pumping or injection wells, ditches, and trenches can also affect the flow of groundwater.

5.3 Determination of Flow Rate

Darcy's Law states that the quantity of water flowing through a geologic material is dependent upon the permeability of the material, the hydraulic gradient, and the cross sectional area through which the water flows. This relation is expressed in the equation:

$$Q = KiA$$

where:

- Q = volume of water flowing through the cross sectional area of the formation (L^3/T).
- K = hydraulic conductivity (L/T).
- i = hydraulic gradient (L/L. i.e., dimensionless).
- A = cross sectional area of formation being considered (L^2).

The relation is similar to one used in stream flow measurements where:

$$Q = VA$$

where:

- Q = discharge from the cross sectional area of a stream or pipe (L^3/T).
- V = average velocity of flowing water (L/T).
- A = cross sectional area through which water flows (L^2).

The velocity of water movement in a geologic formation depends on the specific formation properties and the head differences across the formation. This relation is defined in the equation:

$$V = \frac{K_i}{n}$$

where:

V = average linear velocity of groundwater through the formation (L/T)
K = hydraulic conductivity (L/T)
i = hydraulic gradient (dimensionless)
n = porosity (expressed as a fraction).

Values of porosity for several geologic materials are given in Attachment A. More accurate and specific values of porosity can be obtained by laboratory analysis of a formation sample.

Hydraulic conductivity is related to the permeability of the formation and depends on the size and interconnection of the pore spaces. In isotropic and homogeneous formations, the hydraulic conductivity will be the same vertically and horizontally. However, in anisotropic formations, horizontal and vertical conductivity can be markedly different and the vertical hydraulic conductivity can be up to several orders of magnitude lower than the horizontal hydraulic conductivity. For example, an unfractured shale has a very high porosity, but because of its layered nature, the pore spaces are not connected vertically. Consequently, unfractured shale often has a moderate horizontal hydraulic conductivity but a very low vertical hydraulic conductivity.

Generally, hydraulic conductivity is high for sands, gravels, and limestone containing large solution cavities and low for clays and tightly fractured rock. Attachment A gives values of hydraulic conductivity for several geologic materials. More accurate values can be obtained during field testing of aquifers or from laboratory measurements on undisturbed cores. However, results from field testing usually indicate higher hydraulic conductivities than laboratory testing because full-scale field testing includes the effects of the formational macrostructure (i.e., secondary permeability due to jointing or fractures) which is not reflected in the testing of a small sample in the laboratory.

The hydraulic gradient, i, is determined from field measurements of hydraulic head obtained from water level measuring points. Do not measure gradient from well to well; measure across equipotential lines that are based on the well (and other) data. Once a potentiometric surface map has been generated using the hydraulic head data, the hydraulic gradient can be calculated using the following formula:

$$i = \frac{dh}{dl}$$

where:

dh = change in head (L)
dl = distance between equipotential lines (L)

The hydraulic gradient along any flow line can be calculated from a potentiometric surface map by dividing the contour interval by the length of the flow line between contour lines.

When chemical solutes are traveling in groundwater, as in cases of groundwater contamination, the calculated groundwater velocity may predict flow rates in excess of what is actually observed. This difference in chemical versus water velocities may be due to attenuation or biodegradation of the chemical species in the aquifer. Attenuation is most often caused by adsorption of the chemical contaminant onto the formation grains or matrix. The result is that the chemical does not appear at the downgradient sampling point as quickly as the velocity calculation predicts. An equation to correct for this attenuation is:

$$V_c = V_w/(1 + K_d P_b/n)$$

where:

V_c = velocity of the chemical solute flow (L/T)
V_w = velocity of groundwater flow (L/T)
P_b = formation mass bulk density (M/L^3)
n = formation porosity (expressed as a fraction)
K_d = distribution coefficient = (L^3/M)

The K_d is equal to the mass of solute per unit mass of solid phase divided by the concentration of solute in solution. The term in the denominator is known as the retardation factor.

Density differences between water and contaminants can also cause velocity determination errors. Light hydrocarbons such as gasoline are less dense than water and consequently float on the water table. These contaminants can migrate along the water table surface at rates faster or slower than the rate of groundwater movement, depending on specific conditions, and may also volatilize into unsaturated soil pore spaces. On the other hand, contaminants denser than water such as heavy hydrocarbons (e.g., coal tar) or chlorinated compounds (e.g., TCE, PCE) tend to sink to the bottom of an aquifer if present in concentrations exceeding their solubility limit. Here, the contamination may move at faster or slower rates than the overlying groundwater or may actually move in a direction opposite to that of the groundwater, depending on the geologic characteristics of the aquifer base and direction of dip of the underlying aquitard.

Other factors involving the physicochemical interaction between the chemical and the groundwater, such as dilution (mixing contaminated water or chemicals with additional quantities of groundwater) and dispersion (molecular diffusion of the chemical throughout the groundwater regime), can also affect the observed rates of travel of contaminants in groundwater. In addition to such physicochemical characteristics, all of the aquifer and aquitard properties and groundwater flow characteristics described above must be known so that adequate and accurate estimations of the extent and severity of groundwater contamination can be developed.

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ATTACHMENT A

GENERALIZED POROSITY AND HYDRAULIC CONDUCTIVITY VALUES FOR GEOLOGIC MATERIALS

Material	Porosity Range (%)	Hydraulic Conductivity Range	
		cm/sec	ft/day
Gravel	30-40	10^{-1} to 10^{-2}	280 to 2.8×10^{5}
Coarse sand (clean)	30-40	10^{-1} to 1	280 to 2,800
Medium sand (clean)	35-45	10^{-2} to 10^{-1}	28 to 280
Fine sand (clean)	40-50	5×10^{-4} to 10^{-2}	1.4 to 28
Silty sand	25-40	10^{-5} to 10^{-2}	0.03 to 280
Glacial Till	Variable	10^{-10} to 10^{-4}	3×10^{-7} to 0.3
Unweathered Clay/Shale	45-55 (clay)	10^{-7} to 10^{-4}	3×10^{-4} to 0.3 (horizontal)
		10^{-10} to 10^{-6}	3×10^{-7} to 3×10^{-3} (vertical)
Karst Limestone	---	10^{-4} to 10^{-1}	0.3 to 2,800
Fractured Igneous/ Metamorphic Rocks	---	10^{-6} to 10^{-1}	3×10^{-3} to 280
Sandstone	5-30	10^{-8} to 10^{-4}	3×10^{-5} to 0.3

Source: References 1 and 2

TABLE OF CONTENTS

SECTION		PAGE
1.0	PURPOSE	2
2.0	SCOPE	2
3.0	GLOSSARY	2
4.0	RESPONSIBILITIES	2
5.0	PROCEDURES	3
5.1	Equipment/Items Needed	3
5.2	Well Design	3
5.2.1	Well Depth, Diameter, and Monitored Interval	3
5.2.2	Riser Pipe and Screen Materials	5
5.2.3	Annular Materials	6
5.2.4	Protective Casing	7
5.3	Monitoring Well Installation	7
5.3.1	Monitoring Wells in Unconsolidated Sediments	7
5.3.2	Confining Layer Monitoring Wells	8
5.3.3	Bedrock Monitoring Wells	8
5.3.4	Drive Points	8
5.3.5	Innovative Monitoring Well Installation Techniques	8
5.4	Well Development Methods	9
5.4.1	Overpumping and Backwashing	9
5.4.2	Surging with a Surge Plunger	9
5.4.3	Compressed Air	10
5.4.4	High Velocity Jetting	10
6.0	REFERENCES	10
7.0	RECORDS	10

ATTACHMENTS

A	RELATIVE COMPATIBILITY OF RIGID WELL-CASING MATERIAL (PERCENT) / RELATIVE COMPATIBILITY OF SEMI-RIGID OR ELASTOMERIC MATERIALS (PERCENT)	12
B	COMPARISON OF STAINLESS STEEL AND PVC FOR MONITORING WELL CONSTRUCTION	13

1.0 PURPOSE

This procedure provides general guidance and information pertaining to proper monitoring well design, installation, and development.

2.0 SCOPE

This procedure is applicable to the construction of permanent monitoring wells. The methods described herein may be modified by project-specific requirements for monitoring well construction. In addition, many regulatory agencies have specific regulations pertaining to monitoring well construction and permitting. These requirements must be determined during the project planning phases of the investigation, and any required permits must be obtained before field work begins. Innovative monitoring well installation techniques, which typically are not used, will be discussed only generally in this procedure.

3.0 GLOSSARY

Monitoring Well - A well which is properly screened (if screening is necessary, e.g., open borehole), cased, and sealed which is capable of providing a groundwater level and groundwater sample representative of the zone being monitored.

Piezometer - A pipe or tube inserted into the water bearing zone, typically open to water flow at the bottom and to the atmosphere at the top, and used to measure water level elevations. Piezometers may range in size from 1/2-inch-diameter plastic tubes to well points or monitoring wells.

Potentiometric Surface - The surface representative of the level to which water will rise in a well cased to the screened aquifer.

Well Point (Drive Point) - A screened or perforated tube (Typically 1-1/4 or 2 inches in diameter) with a solid, conical, hardened point at one end, which is attached to a riser pipe and driven into the ground with a sledge hammer, drop weight, or mechanical vibrator. Well points may be used for groundwater injection and recovery, as piezometers (i.e., to measure water levels) or to provide groundwater samples for water quality data.

4.0 RESPONSIBILITIES

Driller - The driller provides adequate and operable equipment, sufficient quantities of materials, and an experienced and efficient labor force capable of performing all phases of proper monitoring well installation and construction. The driller may also be responsible for obtaining, in advance, any required permits for monitoring well installation and construction.

Rig Geologist - The rig geologist supervises and documents well installation and construction performed by the driller, and insures that well construction is adequate to provide representative groundwater data from the monitored interval. Geotechnical engineers, field technicians, or other suitable trained personnel may also serve in this capacity.

5.0 PROCEDURES

5.1 Equipment/Items Needed

Below is a list of items that may be needed when installing a monitoring well:

- Health and safety equipment as required by the Site Safety Officer.
- Well drilling and installation equipment with associated materials (typically supplied by the driller).
- Hydrogeologic equipment (weighted engineer's tape, water level indicator, retractable engineers rule, electronic calculator, clipboard, mirror and flashlight - for observing downhole activities, paint and ink marker for marking monitoring wells, sample jars, well installation forms, and a field notebook).
- Drive point installations tools (sledge hammer, drop hammer, or mechanical vibrator; tripod, pipe wrenches, drive points, riser pipe, and end caps).

5.2 Well Design

The objectives for each monitoring well and its intended use must be clearly defined before the monitoring system is designed. Within the monitoring system, different monitoring wells may serve different purposes and, therefore, require different types of construction. During all phases of the well design, attention must be given to clearly documenting the basis for design decisions, the details of well construction, and the materials to be used. The objectives for installing the monitoring wells may include:

- Determining groundwater flow directions and velocities.
- Sampling or monitoring for trace contaminants.
- Determining aquifer characteristics (e.g., hydraulic conductivity).

Siting of monitoring wells shall be performed after a preliminary estimation of the groundwater flow direction. In most cases, groundwater flow and potential well locations can be determined through the review of geologic data and the site terrain. In addition, data from production wells or other monitoring wells in the area may be used to determine the groundwater flow direction. If these methods cannot be used, piezometers, which are relatively inexpensive to install, may have to be installed in a preliminary investigative phase to determine groundwater flow direction.

5.2.1 Well Depth, Diameter, and Monitored Interval

The well depth, diameter, and monitored interval must be tailored to the specific monitoring needs of each investigation. Specification of these items generally depends on the purpose of the monitoring system and the characteristics of the hydrogeologic system being monitored. Wells of different depth, diameter, and monitored interval can be employed in the same groundwater monitoring system. For instance, varying the monitored interval in several wells, at the same location (cluster wells) can help to determine the vertical gradient and the levels at which contaminants are present. Conversely, a fully penetrating well is usually not used to quantify or vertically locate a contaminant plume, since groundwater samples collected in wells that are screened over the full thickness of the water-bearing zone will be representative of average conditions across the entire monitored interval. However, fully penetrating wells can be used to establish the existence of contamination in the water-bearing zone. The

well diameter desired depends upon the hydraulic characteristics of the water-bearing zone, sampling requirements, drilling method and cost.

The decision concerning the monitored interval and well depth is based on the following (and possibly other) information:

- The vertical location of the contaminant source in relation to the water-bearing zone.
- The depth, thickness and uniformity of the water-bearing zone.
- The anticipated depth, thickness, and characteristics (e.g., density relative to water) of the contaminant plume.
- Fluctuation in groundwater levels (due to pumping, tidal influences, or natural recharge/discharge events).
- The presence and location of contaminants encountered during drilling.
- Whether the purpose of the installation is for determining existence or non-existence of contamination or if a particular stratigraphic zone is being investigated.
- The analysis of borehole geophysical logs.

In most situations where groundwater flow lines are horizontal, depending on the purpose of the well and the site conditions, monitored intervals are 20 feet or less. Shorter screen lengths (1 to 2 feet) are usually required where flow lines are not horizontal, (i.e., if the wells are to be used for accurate measurement of the potentiometric head at a specific point).

Many factors influence the diameter of a monitoring well. The diameter of the monitoring well depends on the application. In determining well diameter, the following needs must be considered:

- Adequate water volume for sampling.
- Drilling methodology.
- Type of sampling device to be used.
- Costs.

Standard monitoring well diameters are 2, 4, 6, or 8 inches. However, drive points are typically 1-1/4 or 2 inches in diameter. For monitoring programs which require screened monitoring wells, either a 2-inch or 4-inch-diameter well is preferred. Typically, well diameters greater than 4 inches are used in monitoring programs in which open-hole monitoring wells are required. In the smaller diameter wells, the volume of stagnant water in the well is minimized, and well construction costs are reduced, however, the type of sampling devices that can be used are limited. In specifying well diameter, sampling requirements must be considered (up to a total of 4 gallons of water may be required for a single sample to account for full organic and inorganic analyses, and split samples). The volume of water in the monitoring well available for sampling is dependent on the well diameter as follows:

Casing Inside Diameter (Inch)	Standing Water Depth to Obtain 1 Gallon Water (Feet)	Total Depth of Standing Water for 4 Gallons (Feet)
2	6.13	25
4	1.53	6
6	0.68	3

However, if a specific well recharges quickly after purging, then well diameter may not be an important factor regarding sample volume requirements.

Pumping tests for determining aquifer characteristics may require larger diameter wells; however, in small-diameter wells in-situ permeability tests can be performed during drilling or after well installation is completed.

5.2.2 Riser Pipe and Screen Materials

Well materials are specified by diameter, type of material, and thickness of pipe. Well screens require an additional specification of slot size. Thickness of pipe is referred to as "schedule" for polyvinyl chloride (PVC) casing and is usually Schedule 40 (thinner wall) or 80 (thicker wall). Steel pipe thickness is often referred to as "Strength" and Standard Strength is usually adequate for monitoring well purposes. With larger diameter pipe, the wall thickness must be greater to maintain adequate strength. The required thickness is also dependent on the method of installation; risers for drive points require greater strength than wells installed inside drilled borings.

The selection of well screen and riser materials depends on the method of drilling, the type of subsurface materials the well penetrates, the type of contamination expected, and natural water quality and depth. Cost and the level of accuracy required are also important. The materials generally available are Teflon, stainless steel, PVC galvanized steel, and carbon steel. Each has advantages and limitations (see Attachment A of this guideline for an extensive presentation on this topic). The two most commonly used materials are PVC and stainless steel for wells in which screens are installed. Properties of these two materials are compared in Attachment B. Stainless steel is preferred where trace metals or organic sampling is required; however, costs are high. Teflon materials are extremely expensive, but are relatively inert and provide the least opportunity for water contamination due to well materials. PVC has many advantages, including low cost, excellent availability, light weight, and ease of manipulation; however, there are also some questions about organic chemical sorption and leaching that are currently being researched (see Barcelona et al., 1983). Concern about the use of PVC can be minimized if PVC wells are used strictly for geohydrologic measurements and not for chemical sampling. The crushing strength of PVC may limit the depth of installation, but Schedule 80 materials normally used for wells greater than 50 feet deep may overcome some of the problems associated with depth. However, the smaller inside diameter of Schedule 80 pipe may be an important factor when considering the size of bailers or pumps required for sampling or testing. Due to this problem, the minimum well pipe size recommended for Schedule 80 wells is 4-inch I.D.

Screens and risers may have to be decontaminated before use because oil-based preservatives and oil used during thread cutting and screen manufacturing may contaminate samples. Metal pipe, may corrode and release metal ions or chemically react with organic constituents, but this is considered by some to be less of a problem than the problem associated with PVC material. Galvanized steel is not

recommended where samples may be collected for metal analyses, as zinc and cadmium levels in groundwater samples may become elevated from leaching of the zinc coating.

Threaded, flush-joint casing is most often preferred for monitoring well applications. PVC, Teflon, and steel can all be obtained with threaded joints at slightly more costs. Welded-joint steel casing is also acceptable. Glued PVC may release organic contaminants into the well, and therefore, should not be used if the well is to be sampled for organic constituents.

When the water-bearing zone is in consolidated bedrock, such as limestone or fractured granite, a well screen is often not necessary (the well is simply an open hole in bedrock). Unconsolidated materials, such as sands, clay, and silts require a screen. A screen slot size of 0.010 or 0.020 inch is generally used when a screen is necessary and the screened interval is artificially packed with a fine sand. The slot size controls the quantity of water entering the well and prevents entry of natural materials or sand pack. The screen shall pass no more than 10 percent of the pack material, or in-situ aquifer material. The rig geologist shall specify the combination of screen slot size and sand pack which will be compatible with the water-bearing zone, to maximize groundwater inflow and minimize head losses and movement of fines into the wells. (For example, as a standard procedure, a Morie No. 1 or Ottowa sand may be used with a 0.010-inch slot screen, however, with a 0.020-inch slot screen, the filter pack material must be the material retained on a No. 20 to No. 30 U.S. standard sieve.)

5.2.3 Annular Materials

Materials placed in the annular space between the borehole and riser pipe and screen include a sand pack when necessary, a bentonite seal, and cement-bentonite grout. The sand pack is usually a fine- to medium-grained poorly graded, silica sand and should relate to the grain size of the aquifer sediments. The quantity of sand placed in the annular space is dependent upon the length of the screened interval, but should always extend at least 1 foot above the top of the screen. At least 1 to 3 feet of bentonite pellets or equivalent shall be placed above the sand pack. Cement-bentonite grout (or equivalent) is then placed to extent from the top of the bentonite pellets to the ground surface.

On occasion, and with the concurrence of the involved regulatory agencies, monitoring wells may be packed naturally (i.e., no artificial sand pack installed), and the natural formation material is allowed to collapse around the well screen after the well is installed. This method has been used where the formation material itself is a relatively uniform grain size, or when artificial sand packing is not possible due to borehole collapse.

Bentonite expands by absorbing water and provides a seal between the screened interval and the overlying portion of the annular space and formation. Cement-bentonite grout is placed on top of the bentonite pellets extending to the surface. The grout effectively seals the well and eliminates the possibility for surface infiltration reaching the screened interval. Grouting also replaces material removed during drilling and prevents hole collapse and subsidence around the well. A tremie pipe should be used to introduce grout from the bottom of the hole upward, to prevent bridging, and to provide a better seal. However, in shallow boreholes that don't collapse, it may be more practical to pour the grout from the surface without a tremie pipe.

Grout is a general term which has several different connotations. For all practical purposes within the monitoring well installation industry, grout refers to the solidified material which is installed and occupies the annular space above the bentonite pellet seal. Grout, most of the time, is made up of two assemblages of material, (e.g., cement-bentonite). A cement-bentonite grout normally is a mixture of cement, bentonite, and water at a ratio of one 90-pound bag of Portland Type I cement, plus

3 to 5 pounds of granular or flake-type bentonite, and 6 gallons of water. A neat cement consists of one ninety-pound bag of Portland Type I cement and 6 gallons of water.

In certain cases, the borehole may be drilled to a depth greater than the anticipated well installation depth. For these cases, the well shall be backfilled to the desired depth with bentonite pellets or equivalent. A short (1- to 2-foot) section of capped riser pipe sump is sometimes installed immediately below the screen, as a silt reservoir, when significant post-development silting is anticipated. This will ensure that the entire screen surface remains unobstructed.

5.2.4 Protective Casing

When the well is completed and grouted to the surface, a protective steel casing is often placed over the top of the well. This casing generally has a hinged cap and can be locked to prevent vandalism. A vent hole shall be provided in the cap to allow venting of gases and maintain atmospheric pressure as water levels rise or fall in the well. The protective casing has a larger diameter than the well and is set into the wet cement grout over the well upon completion. In addition, one hole is drilled just above the cement collar through the protective casing which acts as a weep hole for the flow of water which may enter the annulus during well development, purging, or sampling.

A protective casing which is level with the ground surface is used in roadway or parking lot applications where the top of a monitoring well must be below the pavement. The top of the riser pipe is placed 4 to 5 inches below the pavement, and a locking protective casing is cemented in place to 3 inches below the pavement. A large diameter protective sleeve is set into the wet cement around the well with the top set level with the pavement. A manhole-type lid placed over the protective sleeve. The cement should be slightly mounded to direct pooled water away from the well head.

5.3 Monitoring Well Installation

Pertinent data regarding monitoring well installation shall be recorded on log sheets as depicted and discussed in SOP SA-6.3. Attachments to this referenced SOP illustrate terms and physical construction of various types of monitoring wells.

5.3.1 Monitoring Wells in Unconsolidated Sediments

After the borehole is drilled to the desired depth, well installation can begin. The procedure for well installation will partially be dictated by the stability of the formation in which the well is being placed. If the borehole collapses immediately after the drilling tools are withdrawn, then a temporary casing must be installed and well installation will proceed through the center of the temporary casing, and continue as the temporary casing is withdrawn from the borehole. In the case of hollow-stem auger drilling, the augers will act to stabilize the borehole during well installation.

Before the screen and riser pipe are lowered into the borehole, all pipe and screen sections should be measured with an engineer's rule to ensure proper placement. When measuring sections, the threads on one end of the pipe or screen must be excluded while measuring, since the pipe and screen sections are screwed flush together.

After the screen and riser pipe are lowered through the temporary casing, the sand pack can be installed. A weighted tape measure must be used during the installation procedure to carefully monitor installation progress. The sand is poured into the annulus between the riser pipe and temporary casing, as the casing is withdrawn. Sand should always be kept within the temporary casing during withdrawal in order to ensure an adequate sand pack. However, if too much sand is within the temporary casing (greater

than 1 foot above the bottom of the casing) bridging between the temporary casing and riser pipe may occur.

After the sand pack is installed to the desired depth (at least 1 foot above the top of the screen), then the bentonite pellet seal (or equivalent), can be installed in the same manner as the sand pack. At least 1 to 3 feet of bentonite pellets should be installed above the sand pack.

The cement-bentonite grout is then mixed and either poured or tremied into the annulus as the temporary casing or augers are withdrawn. Finally, the protective casing can be installed as detailed in Section 5.2.4.

In stable formations where borehole collapse does not occur, the well can be installed as discussed above, and the use of a temporary casing is not needed. However, centralizers may have to be installed, one above and one below the screen, to assure enough annular space for sand pack placement.

5.3.2 Confining Layer Monitoring Wells

When drilling and installing a well in a confined aquifer, proper well installation techniques must be applied to avoid cross contamination between the unconfined and confined aquifer. Under most conditions, this can be accomplished by installing double-cased wells. This is accomplished by drilling a large-diameter boring through the upper aquifer, 1 to 3 feet into the underlying confining layer, and setting and pressure grouting or tremie grouting the outer casing into the confining layer. The grout material must fill the space between the native material and the outer casing. A smaller diameter boring is then continued through the confining layer for installation of the monitoring well as detailed for overburden monitoring wells (with the exception of not using a temporary casing during installation). Sufficient time (determined by the rig geologist), must be allowed for setting of the grout prior to drilling through the confined layer.

5.3.3 Bedrock Monitoring Wells

When installing bedrock monitoring wells, a large diameter boring is drilled through the overburden and approximately 5 feet into the bedrock. A casing (typically steel) is installed and either pressure grouted or tremie grouted in place. After the grout has cured, a smaller diameter boring is continued through the bedrock to the desired depth. If the boring does not collapse, the well can be left open, and a screen is not necessary. If the boring collapses, then a screen is required and can be installed as detailed for overburden monitoring wells. However, if a screen is to be used, then the casing which is installed through the overburden and into the bedrock does not require grouting and can be installed temporary until final well installation is completed.

5.3.4 Drive Points

Drive points can be installed with either a sledge hammer, drop hammer, or a mechanical vibrator. The screen is threaded and tightened onto the riser pipe with pipe wrenches. The drive point is simply pounded into the subsurface to the desired depth. If a heavy drop hammer is used, then a tripod and pulley setup is required to lift the hammer. Drive points typically cannot be driven to depths exceeding 10 feet.

5.3.5 Innovative Monitoring Well Installation Techniques

Certain innovative sampling devices have proven advantageous. These devices are essentially screened samplers installed in a borehole with only one or two small-diameter tubes extending to the surface.

Manufacturers of these types of samplers claim that four samplers can be installed in a 3-inch-diameter borehole. This reduces drilling costs, decreases the volume of stagnant water, and provides a sampling system that minimizes cross-contamination from sampling equipment. These samplers also perform well when the water table is within 25 feet of the surface (the typical range of suction pumps). Two manufacturers of these samplers are Timco Manufacturing Company, Inc., of Prairie du Sac, Wisconsin, and BARCAD Systems, Inc., of Concord, Massachusetts. Each manufacturer offers various construction materials.

Two additional types of multilevel sampling systems have been developed. Both employ individual screened openings through a small-diameter casing. One of these systems (marketed by Westbay Instruments Ltd. of Vancouver, British Columbia, Canada) uses a screened port and a sampling probe to obtain samples and head measurements or perform permeability tests. This system allows sampling ports at intervals as close as 5 feet, if desired, in boreholes from 3 to 4.8 inches in diameter.

The second system, developed at the University of Waterloo at Waterloo, Ontario, Canada, requires field assembly of the individual sampling ports and tubes that actuate a simple piston pump and force the samples to the surface. Where the depth to ground water is less than 25 feet, the piston pumps are not required. The assembly is made of easily obtained materials; however, the cost of labor to assemble these monitoring systems may not be cost-effective.

5.4 Well Development Methods

The purpose of well development is to stabilize and increase the permeability of the gravel pack around the well screen, and to restore the permeability of the formation which may have been reduced by drilling operations. Wells are typically developed until all fine material and drilling water is removed from the well. Sequential measurements of pH, conductivity and temperature taken during development may yield information (stabilized values) that sufficient development is reached. The selection of the well development method shall be made by the rig geologist and is based on the drilling methods, well construction and installation details, and the characteristics of the formation that the well is screened in. The primary methods of well development are summarized below. A more detailed discussion may be found in Driscoll (1986).

5.4.1 Overpumping and Backwashing

Wells may be developed by alternatively drawing the water level down at a high rate (by pumping or bailing) and then reversing the flow direction (backwashing) so that water is passing from the well into the formation. This back and forth movement of water through the well screen and gravel pack serves to remove fines from the formation immediately adjacent to the well, while preventing bridging (wedging) of sand grains. Backwashing can be accomplished by several methods, including pouring water into the well and then bailing, starting and stopping a pump intermittently to change water levels, or forcing water into the well under pressure through a water-tight fitting ("rawhiding"). Care should be taken when backwashing not to apply too much pressure, which could damage or destroy the well screen.

5.4.2 Surging with a Surge Plunger

A surge plunger (also called a surge block) is approximately the same diameter as the well casing and is used to agitate the water, causing it to move in and out of the screens. This movement of water pulls fine materials into the well, where they may be removed by any of several methods, and prevents bridging of sand particles in the gravel pack. There are two basic types of surge plungers; solid and valved surge plungers. In formations with low yields, a valved surge plunger may be preferred, as solid

plungers tend to force water out of the well at a greater rate than it will flow back in. Valved plungers are designed to produce a greater inflow than outflow of water during surging.

5.4.3 Compressed Air

Compressed air can be used to develop a well by either of two methods: backwashing or surging. Backwashing is done by forcing water out through the screens, using increasing air pressure inside a sealed well, then releasing the pressurized air to allow the water to flow back into the well. Care should be taken when using this method so that the water level does not drop below the top of the screen, thus reducing well yield. Surging, or the "open well" method, consists of alternately releasing large volumes of air suddenly into an open well below the water level to produce a strong surge by virtue of the resistance of water head, friction, and inertia. Pumping of the well is subsequently done using the air lift method.

5.4.4 High Velocity Jetting

In the high velocity jetting method, water is forced at high velocities from a plunger-type device and through the well screen to loosen fine particles from the sand pack and surrounding formation. The jetting tool is slowly rotated and raised and lowered along the length of the well screen to develop the entire screened area. Jetting using a hose lowered into the well may also be effective. The fines washed into the screen during this process can then be bailed or pumped from the well.

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7.0 RECORDS

A critical part of monitoring well installation is recording of significant details and events in the site lobgook or field notebook. The geologist must record the exact depths of significant hydrogeological features screen placement, gravel pack placement, and bentonite placement.

A Monitoring Well Sheet (see Attachments to SOP SA-6.3) shall be completed thus ensuring the uniform recording of data for each installation and rapid identification of missing information. Well depth, length, materials of construction, length and openings of screen, length and type of riser, and depth and type of all backfill materials shall be recorded. Additional information shall include location, installation date, problems encountered, water levels before and after well installation, cross-reference to the geologic boring log, and methods used during the installation and development process. Documentation is very important to prevent problems involving questionable sample validity. Somewhat different information

will need to be recorded.depending on whether the well is completed in overburden, in a confined layer, in bedrock with a cased well, or as an open hole in bedrock.

The quantities of sand, bentonite, and grout placed in the well are also important. The geologist shall calculate the annular space volume and have a general idea of the quantity of material needed to fill the annular space. Volumes of backfill significantly higher than the calculated volume may indicate a problem such as a large cavity, while a smaller backfill volume may indicate a cave-in. Any problems with rig operation or down-time shall be recorded and may affect the driller's final fee.

ATTACHMENT A

RELATIVE COMPATIBILITY OF RIGID WELL CASING MATERIAL (PERCENT)

Potentially-Deteriorating Substance	Type of Casing Material						
	PVC 1	Galvanized Steel	Carbon Steel	Lo-carbon Steel	Stainless Steel 304	Stainless Steel 316	Teflon*
Buffered Weak Acid	100	56	51	59	97	100	100
Weak Acid	98	59	43	47	96	100	100
Mineral Acid/ High Solids Content	100	48	57	60	80	82	100
Aqueous/Organic Mixtures	64	69	73	73	98	100	100
Percent Overall Rating	91	58	56	59	93	96	100

Preliminary Ranking of Rigid Materials:

1 Teflon®
2 Stainless Steel 316
3. Stainless Steel 304
4 PVC 1
5 Lo-Carbon Steel
6 Galvanized Steel
7 Carbon Steel

* Trademark of DuPont

RELATIVE COMPATIBILITY OF SEMI-RIGID OR ELASTOMERIC MATERIALS (PERCENT)

Potentially-Deteriorating Substance	Type of Casing Material								
	PVC Flexible	PP	PE Conv.	PE Linear	PMM	Viton®*	Silicone	Neoprene	Teflon®*
Buffered Weak Acid	97	97	100	97	90	92	87	85	100
Weak Acid	92	90	94	96	78	78	75	75	100
Mineral Acid/ High Solids Content	100	100	100	100	95	100	78	82	100
Aqueous/Organic Mixtures	62	71	40	60	49	78	49	44	100
Percent Overall Rating	88	90	84	88	78	87	72	72	100

Preliminary Ranking of Semi-Rigid or Elastomeric Materials:

1 Teflon®
2 Polypropylene (PP)
3. PVC Flexible/PE Linear
4 Viton®
5 PE Conventional
6 Plexiglas/Lucite (PMM)
7 Silicone/Neoprene

* Trademark of DuPont

Source: Barcelona et al., 1983

ATTACHMENT B

COMPARISON OF STAINLESS STEEL AND PVC FOR MONITORING WELL CONSTRUCTION

Characteristic	Stainless Steel	PVC
Strength	Use in deep wells to prevent compression and closing of screen/riser.	Use when shear and compressive strength are not critical.
Weight	Relatively heavier.	Light-weight; floats in water.
Cost	Relatively expensive.	Relatively inexpensive.
Corrosivity	Deteriorates more rapidly in corrosive water.	Non-corrosive -- may deteriorate in presence of ketones, aromatics, alkyl sulfides, or some chlorinated hydrocarbons.
Ease of Use	Difficult to adjust size or length in the field.	Easy to handle and work with in the field.
Preparation for Use	Should be steam cleaned organics will be subsequently sampled.	Never use glue fittings -- pipes should be threaded or pressure fitted. Should be steam cleaned when used for monitoring wells.
Interaction with Contaminants*	May sorb organic or inorganic substances when oxidized.	May sorb or release organic substances.

* See also Attachment A.

TABLE OF CONTENTS

SECTION **PAGE**

1.0 PURPOSE 2

2.0 SCOPE 2

3.0 GLOSSARY 2

4.0 RESPONSIBILITIES 3

5.0 PROCEDURES 3

5.1 General 3
5.2 Sampling, Monitoring, and Evacuation Equipment 4
5.3 Calculations of Well Volume 5
5.4 Evacuation of Static Water (Purging) 5
5.4.1 General 5
5.4.2 Evacuation Devices 6
5.5 Onsite Water Quality Testing 7
5.5.1 Measurement of pH 7
5.5.2 Measurement of Specific Conductance 9
5.5.3 Measurement of Temperature 11
5.5.4 Measurement of Dissolved Oxygen Concentration 11
5.5.5 Measurement of Oxidation-Reduction Potential 13
5.5.6 Measurement of Turbidity 14
5.6 Sampling 16
5.6.1 Sampling Plan 16
5.6.2 Sampling Methods 16
5.7 Low Flow Purging and Sampling 17
5.7.1 Scope and Application 17
5.7.2 Equipment 18
5.7.3 Purging and Sampling Procedure 19

6.0 REFERENCES 20

ATTACHMENTS

A PURGING EQUIPMENT SELECTION 22
B SPECIFIC CONDUCTANCE OF 1 MOLAR KCl AT VARIOUS TEMPERATURES . . . 25
C VARIATION OF DISSOLVED OXYGEN CONCENTRATION IN WATER AS A FUNCTION OF TEMPERATURE AND SALINITY 26

1.0 PURPOSE

The purpose of this procedure is to provide general reference information regarding the sampling of groundwater wells.

2.0 SCOPE

This procedure provides information on proper sampling equipment, onsite water quality testing, and techniques for groundwater sampling. Review of the information contained herein will facilitate planning of the field sampling effort by describing standard sampling techniques. The techniques described shall be followed whenever applicable, noting that site-specific conditions or project-specific plans may require modifications to methodology.

3.0 GLOSSARY

Conductance - The conductance of a conductor 1 centimeter long and 1 square centimeter in cross-sectional area. For groundwater measurements, a volume of water contained in a 1 cm x 1 cm sample container (the water acts as the conductor). Conductivity and specific conductance are used synonymously.

Electrolytic Cell - An electrochemical cell in which electrical energy is supplied from an external source. This cell functions in much the same way as a galvanic cell, only the current flows in the opposite direction due to the external source of applied voltage. Electrolytic cells are used in dissolved oxygen measurement.

Galvanic Cell - A electrochemical cell in which chemical energy is spontaneously converted to electrical energy. The electrical energy produced is supplied to an external circuit. Galvanic cells are used in dissolved oxygen measurement.

Ohm - Standard unit of electrical resistance (R). Used in specific conductance measurement. A siemen (or umho) is the standard unit of electrical conductance, the inverse of the ohm.

Oxidation-Reduction Potential (ORP) - A measure of the activity ratio of oxidizing and reducing species as determined by the electromotive force developed by a noble metal electrode, immersed in water, as referenced against a standard hydrogen electrode.

pH - The negative logarithm (base 10) of the hydrogen ion activity. The hydrogen ion activity is related to the hydrogen ion concentration, and, in a relatively weak solution, the two are nearly equal. Thus, for all practical purposes, pH is a measure of the hydrogen ion concentration.

pH Paper - Indicator paper that turns different colors depending on the pH of the solution to which it is exposed. Comparison with color standards supplied by the manufacturer will then give an indication of the solution's pH.

Resistance - A measure of the solution's ability to oppose the passage of electrical current. For metals and solutions, resistance is defined by Ohm's Law, $E = IR$, where E is the potential difference, I is the current, and R is the resistance. Used in measurement of specific conductance.

4.0 RESPONSIBILITIES

Project Hydrogeologist - Responsible for selecting and detailing the specific groundwater sampling techniques, onsite water quality testing (type, frequency, and location), and equipment to be used, and providing detailed input in this regard to the project plan documents. The project hydrogeologist is also responsible for properly briefing and overseeing the performance of the site sampling personnel.

Project Geologist - is primarily responsible for the proper acquisition of the groundwater samples. He/she is also responsible for the actual analyses of onsite water quality samples, as well as instrument calibration, care, and maintenance. When appropriate, such responsibilities may be performed by other qualified personnel (e.g., field technicians).

5.0 PROCEDURES

5.1 General

To be useful and accurate, a groundwater sample must be representative of the particular zone of the water being sampled. The physical, chemical, and bacteriological integrity of the sample must be maintained from the time of sampling to the time of analysis in order to keep any changes in water quality parameters to a minimum.

Methods for withdrawing samples from completed wells include the use of pumps, compressed air, bailers, and various types of samplers. The primary considerations in obtaining a representative sample of the groundwater are to avoid collection of stagnant (standing) water in the well and to avoid physical or chemical alteration of the water due to sampling techniques. In a non-pumping well, there will be little or no vertical mixing of water in the well pipe or casing, and stratification will occur. The well water in the screened section will mix with the groundwater due to normal flow patterns, but the well water above the screened section will remain isolated and become stagnant. To safeguard against collecting non-representative stagnant water in a sample, the following approach shall be followed prior to sample acquisition:

1. All monitoring wells shall be purged prior to obtaining a sample. Evacuation of three to five volumes is recommended prior to sampling. In a high-yielding groundwater formation and where there is no stagnant water in the well above the screened section, extensive evacuation prior to sample withdrawal is not as critical.

2. For wells that can be purged dry, the well shall be evacuated and allowed to recover prior to sample acquisition. If the recovery rate is fairly rapid, evacuation of more than one volume of water is required.

3. For high-yielding monitoring wells which cannot be evacuated to dryness, there is no absolute safeguard against contaminating the sample with stagnant water. One of the following techniques shall be used to minimize this possibility:

   - A submersible pump or the intake line of a surface pump or bailer shall be placed just below the water surface when removing the stagnant water and lowered as the water level drops. Three to five volumes of water shall be removed to provide reasonable assurance that all stagnant water has been evacuated. Once this is accomplished, a bailer or other approved device may be used to collect the sample for analysis.

- The intake line of the sampling pump (or the submersible pump itself) shall be placed near the bottom of the screened section, and approximately one casing volume of water shall be pumped from the well at a low purge rate, equal to the well's recovery rate (low flow sampling).

Stratification of contaminants may exist in the aquifer. Concentration gradients as a result of mixing and dispersion processes, layers of variable permeability, and the presence of separate-phase product (i.e, floating hydrocarbons) may cause stratification. Excessive pumping or improper sampling methods can dilute or increase the contaminant concentrations in the recovered sample compared to what is representative of the integrated water column as it naturally occurs at that point, thus the result is the collection of a non-representative sample.

5.2 Sampling, Monitoring, and Evacuation Equipment

Sample containers shall conform with the guidelines expressed in SOP SA-6.1.

The following equipment shall be on hand when sampling ground water wells (reference SOPs SA-6.1 and SA-7.1):

- Sample packaging and shipping equipment - Coolers for sample shipping and cooling, chemical preservatives, appropriate sampling containers and filler, ice, labels and chain-of-custody documents.

- Field tools and instrumentation - Thermometer, pH paper/meter, camera and film (if appropriate), appropriate keys (for locked wells), engineer's rule, water level indicator, specific conductivity meter, and turbidity meter (as applicable).

- Pumps

  - Shallow-well pumps: Centrifugal, pitcher, suction, or peristaltic pumps with droplines, air-lift apparatus (compressor and tubing) where applicable.

  - Deep-well pumps: Submersible pump and electrical power-generating unit, or air-lift apparatus where applicable.

- Other sampling equipment - Bailers and inert line with tripod-pulley assembly (if necessary). Bailers or submersible centrifugal pumps shall be used to obtain samples for volatile organics from shallow and deep groundwater wells.

- Pails - Plastic, graduated.

- Decontamination solutions - Deionized water, laboratory detergents, 10% nitric acid solution (as required), and analytical-grade solvents (e.g., methanol, acetone, hexane), as required.

Ideally, sample withdrawal equipment shall be completely inert, economical, easily cleaned, cleaned prior to use, reusable, able to operate at remote sites in the absence of power sources, and capable of delivering variable rates for well flushing and sample collection.

5.3 Calculations of Well Volume

To insure that the proper volume of water has been removed from the well prior to sampling it is first necessary to know the volume of standing water in the well pipe. This volume can be easily calculated by the following method. Calculations shall be entered in the site logbook or field notebook or on a sample log sheet form (see SOP SA-6.3):

- Obtain all available information on well construction (location, casing, screens, etc.).
- Determine well or casing diameter.
- Measure and record static water level (depth below ground level or top of casing reference point).
- Determine depth of well by sounding using a clean, decontaminated, weighted tape measure.
- Calculate number of linear feet of static water (total depth or length of well pipe minus the depth to static water level).
- Calculate one static well volume in gallons $V = (0.163)(T)(r^2)$

where:	V	=	Static volume of well in gallons.
	T	=	Thickness of water table in the well measured in feet (i.e., linear feet of static water).
	r	=	Inside radius of well casing in inches.
	0.163	=	A constant conversion factor which compensates for the conversion of the casing radius from inches to feet, the conversion of cubic feet to gallons, and pi.

- Per evacuation volumes discussed above, determine the minimum amount to be evacuated before sampling.

5.4 Evacuation of Static Water (Purging)

5.4.1 General

The amount of purging a well shall receive prior to sample collection will depend on the intent of the monitoring program and the hydrogeologic conditions. Programs to determine overall quality of water resources may require long pumping periods to obtain a sample that is representative of a large volume of that aquifer. The pumped volume may be specified prior to sampling so that the sample can be a composite of a known volume of the aquifer. Alternately the well can be pumped until the parameters such as temperature, electrical conductance, pH, and turbidity (as applicable), have stabilized. Onsite measurements of these parameters shall be recorded in the site logbook, field notebook, or on standardized data sheets.

5.4.2 Evacuation Devices

The following discussion is limited to those devices commonly used at hazardous waste sites. Attachment A provides guidance on the proper evacuation device to use for given sampling situations. Note that all of these techniques involve equipment which is portable and readily available.

Bailers

Bailers are the simplest evacuation devices used and have many advantages. They generally consist of a length of pipe with a sealed bottom (bucket-type bailer) or, as is more useful and favored, with a ball check-valve at the bottom. An inert line is used to lower the bailer and retrieve the sample.

Advantages of bailers include:

- Few limitations on size and materials used for bailers.
- No external power source needed.
- Bailers are inexpensive, and can be dedicated and hung in a well to reduce the chances of cross-contamination.
- There is minimal outgassing of volatile organics while the sample is in the bailer.
- Bailers are relatively easy to decontaminate.

Limitations on the use of bailers include the following:

- It is time consuming to remove stagnant water using a bailer.
- Transfer of sample may cause aeration.
- Use of bailers is physically demanding, especially in warm temperatures at protection levels above Level D.

Suction Pumps

There are many different types of inexpensive suction pumps including centrifugal, diaphragm, peristaltic, and pitcher pumps. Centrifugal and diaphragm pumps can be used for well evacuation at a fast pumping rate and for sampling at a low pumping rate. The peristaltic pump is a low volume pump that uses rollers to squeeze a flexible tubing, thereby creating suction. This tubing can be dedicated to a well to prevent cross contamination. The pitcher pump is a common farm hand-pump.

These pumps are all portable, inexpensive and readily available. However, because they are based on suction, their use is restricted to areas with water levels within 20 to 25 feet of the ground surface. A significant limitation is that the vacuum created by these pumps can cause significant loss of dissolved gases and volatile organics.

Air-Lift Samplers

This group of pump samplers uses gas pressure either in the annulus of the well or in a venturi to force the water up a sampling tube. These pumps are also relatively inexpensive. Air (or gas)-lift samplers are more suitable for well development than for sampling because the samples may be aerated, leading to pH changes and subsequent trace metal precipitation, or loss of volatile organics.

Submersible Pumps

Submersible pumps take in water and push the sample up a sample tube to the surface. The power sources for these samplers may be compressed gas or electricity. The operation principles vary and the displacement of the sample can be by an inflatable bladder, sliding piston, gas bubble, or impeller. Pumps are available for 2-inch-diameter wells and larger. These pumps can lift water from considerable depths (several hundred feet).

Limitations of this class of pumps include:

- They may have low delivery rates.
- Many models of these pumps are expensive.
- Compressed gas or electric power is needed.
- Sediment in water may cause clogging of the valves or eroding the impellers with some of these pumps.
- Decontamination of internal components can be difficult and time-consuming.

5.5 Onsite Water Quality Testing

This section describes the procedures and equipment required to measure the following parameters of an aqueous sample in the field:

- pH
- Specific Conductance
- Temperature
- Dissolved Oxygen (DO) Concentration
- Oxidation Reduction Potential
- Certain Dissolved Constituents Using Specific Ion Elements
- Turbidity

This section is applicable for use in an onsite groundwater quality monitoring program to be conducted at a hazardous or nonhazardous site. The procedures and equipment described are applicable to groundwater samples and are not, in general, subject to solution interferences from color, turbidity, and colloidal material or suspended matter.

This section provides general information for measuring the parameters listed above with instruments and techniques in common use. Since instruments from different manufacturers may vary, review of the manufacturer's literature pertaining to the use of a specific instrument is required before use.

5.5.1 Measurement of pH

5.5.1.1 General

Measurement of pH is one of the most important and frequently used tests in water chemistry. Practically every phase of water supply and wastewater treatment such as acid-base neutralization, water softening, and corrosion control is pH dependent. Likewise, the pH of leachate can be correlated with other chemical analyses to determine the probable source of contamination. It is therefore important that reasonably accurate pH measurements be taken.

Two methods are given for pH measurement: the pH meter and pH indicator paper. The indicator paper is used when only a rough estimate of the pH is required, and the pH meter when a more accurate measurement is needed. The response of a pH meter can be affected to a slight degree by high levels of colloidal or suspended solids, but the effect is usually small and generally of little significance. Consequently, specific methods to overcome this interference are not described. The response of pH paper is unaffected by solution interferences from color, turbidity, colloidal or suspended materials unless extremely high levels capable of coating or masking the paper are encountered. In such cases, use of a pH meter is recommended.

5.5.1.2 Principles of Equipment Operation

Use of pH papers for pH measurement relies on a chemical reaction caused by the acidity or basicity of the solution created by the addition of the water sample reacting with the indicator compound on the paper. Various types of pH papers are available, including litmus (for general acidity or basicity determination) and specific pH range hydrion paper.

Use of a pH meter relies on the same principle as other ion-specific electrodes. Measurement relies on establishment of a potential difference across a glass or other type of membrane in response to (in this instance, hydrogen) ion concentration across that membrane. The membrane is conductive to ionic species and, in combination with a standard or reference electrode, a potential difference proportional to the ion concentration is generated and measured.

5.5.1.3 Equipment

The following equipment is needed for taking pH measurements:

- Stand-alone 150 portable pH meter, or combination meter (e.g., Horiba U-10), or combination meter equipped with an in-line sample chamber.
- Combination electrode with polymer body to fit the above meter (alternately a pH electrode and a reference electrode can be used if the pH meter is equipped with suitable electrode inputs).
- Buffer solutions, as specified by the manufacturer.
- pH indicator paper, to cover the pH range 2 through 12.
- Manufacturer's operation manual.

5.5.1.4 Measurement Techniques for Field Determination of pH

pH Meter

The following procedure is used for measuring pH with a pH meter (meter standardization AND CALIBRATION is according to manufacturer's instructions):

- Inspect the instrument and batteries prior to initiation of the field effort.
- Check the integrity of the buffer solutions used for field calibration. Buffer solutions need to be changed often as a result of degradation upon exposure to the atmosphere.

- Immerse the tip of the electrodes in water overnight. If this is not possible due to field conditions, immerse the electrode tip in water for at least an hour before use. The electrode tip may be immersed in a rubber or plastic sack containing buffer solution for field transport or storage. This is not applicable for all electrodes as some must be stored dry.

- If applicable, make sure all electrolyte solutions within the electrode(s) are at their proper levels and that no air bubbles are present within the electrode(s).

- Calibrate on a daily use basis following manufacturer's instructions. Record STANDARD SOLUTION LOT NUMBERS AND calibration data on an equipment calibration log sheet.

- Immerse the electrode(s) in the unknown solution, slowly stirring the probe until the pH stabilizes (±0.1 pH STANDARD UNITS). Stabilization may take several seconds to minutes. If the pH continues to drift, the sample temperature may not be stable, a physical reaction (e.g., degassing) may be taking place in the sample, or the meter or electrode may be malfunctioning. This must be clearly noted in the logbook.

- Read and record the pH of the solution. pH shall be recorded to the nearest 0.1 STANDARD pH unit. Also record the sample temperature.

- Rinse the electrode(s) with deionized water.

- Store the electrode(s) in an appropriate manner when not in use.

Any visual observation of conditions which may interfere with pH measurement, such as oily materials, or turbidity, shall be noted.

pH Paper

Use of pH paper is very simple and requires no sample preparation, standardization, etc. pH paper is available in several ranges, including wide-range (indicating approximately pH 1 to 12), mid-range (approximately pH 0 to 6, 6 to 9, 8 to 14) and narrow-range (many available, with ranges as narrow as 1.5 pH units). The appropriate range of pH paper shall be selected. If the pH is unknown the investigation shall start with wide-range paper and proceed with successively narrower range paper until the sample pH is adequately determined.

5.5.2 Measurement of Specific Conductance

5.5.2.1 General

Conductance provides a measure of dissolved ionic species in water and can be used to identify the direction and extent of migration of contaminants in groundwater or surface water. It can also be used as a measure of subsurface biodegradation or to indicate alternate sources of groundwater contamination.

Conductivity is a numerical expression of the ability of a water sample to carry an electric current. This value depends on the total concentration of the ionized substances dissolved in the water and the temperature at which the measurement is made. The mobility of each of the various dissolved ions, their valences, and their actual and relative concentrations affect conductivity.

It is important to obtain a specific conductance measurement soon after taking a sample, since temperature changes, precipitation reactions, and absorption of carbon dioxide from the air all affect the specific conductance.

5.5.2.2 Principles of Equipment Operation

An aqueous system containing ions will conduct an electric current. In a direct-current field, the positive ions migrate toward the negative electrode, while the negatively charged ions migrate toward the positive electrode. Most inorganic acids, bases and salts (such as hydrochloric acid, sodium carbonate, or sodium chloride, respectively) are relatively good conductors. Conversely, organic compounds such as sucrose or benzene, which do not disassociate in aqueous solution, conduct a current very poorly, if at all.

A conductance cell and a Wheatstone Bridge (for the measurement of potential difference) may be used for measurement of electrical resistance. The ratio of current applied to voltage across the cell may also be used as a measure of conductance. The core element of the apparatus is the conductivity cell containing the solution of interest. Depending on ionic strength of the aqueous solution to be tested, a potential difference is developed across the cell which can be converted directly or indirectly (depending on instrument type) to a measurement of specific conductance.

5.5.2.3 Equipment

The following equipment is needed for taking specific conductance (SC) measurements:

- Stand alone portable conductivity meter, or combination meter (e.g., Horiba U-10), or combination meter equipped with an in-line sample chamber.
- Calibration solution, as specified by the manufacturer.
- Manufacturer's operation manual.

A variety of conductivity meters are available which may also be used to monitor salinity and temperatures. Probe types and cable lengths vary, so equipment must be obtained to meet the specific requirement of the sampling program.

5.5.2.4 Measurement Techniques for Specific Conductance

The steps involved in taking specific conductance measurements are listed below (standardization is according to manufacturer's instructions):

- Check batteries and calibrate instrument before going into the field.
- Calibrate on a daily use basis, according to the manufacturer's instructions and record all pertinent information INCLUDING CALIBRATION STANDARD LOT NUMBERS on an equipment calibration log sheet. Potassium chloride solutions with a SC closest to the values expected in the field shall be used for calibration. Attachment B provides guidance in this regard.
- Rinse the cell with one or more portions of the sample to be tested or with deionized water.

- Immerse the electrode in the sample and measure the conductivity. Adjust the temperature setting to the sample temperature (if applicable).
- Read and record the results in a field logbook or sample log sheet.
- Rinse the electrode with deionized water.

If the specific conductance measurements become erratic, recalibrate the instrument and see the manufacturer's instructions for details. STABLE SC MEASUREMENTS ARE WITHIN ±3 PERCENT.

5.5.3 Measurement of Temperature

5.5.3.1 General

In combination with other parameters, temperature can be a useful indicator of the likelihood of biological action in a water sample. It can also be used to trace the flow direction of contaminated groundwater. Temperature measurements shall be taken in-situ, or as quickly as possible in the field. Collected water samples may rapidly equilibrate with the temperature of their surroundings.

5.5.3.2 Equipment

Temperature measurements may be taken with alcohol-toluene, mercury filled or dial-type thermometers. In addition, various meters such as specific conductance or dissolved oxygen meters, which have temperature measurement capabilities, may also be used. Using such instrumentation along with suitable probes and cables, in-situ measurements of temperature at great depths can be performed.

5.5.3.3 Measurement Techniques for Water Temperature

If a thermometer is used to determine the temperature for a water sample:

- Immerse the thermometer in the sample until temperature equilibrium is obtained (±3 PERCENT ~~(1A 3 minutes~~). To avoid the possibility of cross-contamination, the thermometer shall not be inserted into samples which will undergo subsequent chemical analysis.
- Record values in a field logbook or sample log sheet.

If a temperature meter or probe is used, the instrument shall be calibrated according to manufacturer's recommendations.

5.5.4 Measurement of Dissolved Oxygen Concentration

5.5.4.1 General

Dissolved oxygen (DO) levels in natural water and wastewater depend on the physical, chemical and biochemical activities in the water body. Conversely, the growth of many aquatic organisms as well as the rate of corrosivity, are dependent on the dissolved oxygen concentration. Thus, analysis for dissolved oxygen is a key test in water pollution and waste treatment process control. If at all possible, DO measurements shall be taken in-situ, since concentration may show a large change in a short time if the sample is not adequately preserved.

The monitoring method discussed herein is limited to the use of dissolved oxygen meters only. Chemical methods of analysis (i.e., Winkler methods) are available, but require more equipment and greater sample manipulation. Furthermore, DO meters, using a membrane electrode, are suitable for highly polluted waters, because the probe is completely submersible, and is not susceptible to interference caused by color, turbidity, colloidal material or suspended matter.

5.5.4.2 Principles of Equipment Operation

Dissolved oxygen probes are normally electrochemical cells that have two solid metal electrodes of different nobility immersed in an electrolyte. The electrolyte is retained by an oxygen-permeable membrane. The metal of highest nobility (the cathode) is positioned at the membrane. When a suitable potential exists between the two metals, reduction of oxygen to hydroxide ion (OH) occurs at the cathode surface. An electrical current is developed that is directly proportional to the rate of arrival of oxygen molecules at the cathode.

Since the current produced in the probe is directly proportional to the rate of arrival of oxygen at the cathode, it is important that a fresh supply of sample always be in contact with the membrane. Otherwise, the oxygen in the aqueous layer along the membrane is quickly depleted and false low readings are obtained. It is therefore necessary to stir the sample (or the probe) constantly to maintain fresh solution near the membrane interface. Stirring, however, shall not be so vigorous that additional oxygen is introduced through the air-water interface at the sample surface. To avoid this possibility, some probes are equipped with stirrers to agitate the solution near the probe, while leaving the surface of the solution undisturbed.

Dissolved oxygen probes are relatively unaffected by interferences. Interferences that can occur are reactions with oxidizing gases (such as chlorine) or with gases such as hydrogen sulfide, which are not easily depolarized from the indicating electrode. If a gaseous interference is suspected, it shall be noted in the field log book and checked if possible. Temperature variations can also cause interference because probes exhibit temperature sensitivity. Automatic temperature compensation is normally provided by the manufacturer.

5.5.4.3 Equipment

The following equipment is needed to measure dissolved oxygen concentration:

- Stand alone portable dissolved oxygen meter, or combination meter (e.g., Horiba U-10), or combination meter equipped with an in-line sample chamber.
- Sufficient cable to allow the probe to contact the sample.
- Manufacturer's operation manual.

5.5.4.4 Measurement Techniques for Dissolved Oxygen Determination

Probes differ as to specifics of use. Follow the manufacturer's instructions to obtain an accurate reading. The following general steps shall be used to measure the dissolved oxygen concentration:

- The equipment shall be calibrated and have its batteries checked in the warehouse before going to the field.

- The probe shall be conditioned in a water sample for as long a period as practical before use in the field. Long periods of dry storage followed by short periods of use in the field may result in inaccurate readings.
- The instrument shall be calibrated in the field according to manufacturer's recommendations or in a freshly air-saturated water sample of known temperature. Dissolved oxygen values for air-saturated water can be determined by consulting a table listing oxygen solubilities as a function of temperature and salinity (see Attachment C).
- Record all pertinent information on an equipment calibration sheet.
- Rinse the probe with deionized water.
- Immerse the probe in the sample. Be sure to provide for sufficient flow past the membrane by stirring the sample. Probes without stirrers placed in wells can be moved up and down.
- Record the dissolved oxygen content and temperature of the sample in a field logbook or sample log sheet.
- Rinse the probe with deionized water.
- Recalibrate the probe when the membrane is replaced, or as needed. Follow the manufacturer's instructions.

Note that in-situ placement of the probe is preferable, since sample handling is not involved. This however, may not always be practical. Be sure to record whether the liquid was analyzed in-situ, or if a sample was taken.

Special care shall be taken during sample collection to avoid turbulence which can lead to increased oxygen solubilization and positive test interferences.

5.5.5 Measurement of Oxidation-Reduction Potential

5.5.5.1 General

The oxidation-reduction potential (ORP) provides a measure of the tendency of organic or inorganic compounds to exist in an oxidized state. The ORP parameter therefore provides evidence of the likelihood of anaerobic degradation of biodegradable organics or the ratio of activities of oxidized to reduced species in the sample.

5.5.5.2 Principles of Equipment Operation

When an inert metal electrode, such as platinum, is immersed in a solution, a potential is developed at that electrode depending on the ions present in the solution. If a reference electrode is placed in the same solution, an ORP electrode pair is established. This electrode pair allows the potential difference between the two electrodes to be measured and is dependent on the concentration of the ions in solution. By this measurement, the ability to oxidize or reduce species in solution may be determined. Supplemental measurements, such as dissolved oxygen, may be correlated with ORP to provide a knowledge of the quality of the solution, water, or wastewater.

5.5.5.3 Equipment

The following equipment is needed for measuring the oxidation-reduction potential of a solution:

- Portable pH meter or equivalent, with a millivolt scale.
- Platinum electrode to fit above pH meter.
- Reference electrode such as a calomel, silver-silver chloride, or equivalent.
- Reference solution as specified by the manufacturer.
- Manufacturer's operation manual.

5.5.5.4 Measurement Techniques for Oxidation-Reduction Potential

The following procedure is used for measuring oxidation-reduction potential:

- The equipment shall be calibrated and have its batteries checked before going to the field.

- Check that the platinum probe is clean and that the platinum bond or tip is unoxidized. If dirty, polish with emery paper or, if necessary, clean the electrode using aqua regia, nitric acid, or chromic acid, in accordance with manufacturer's instructions.

- Thoroughly rinse the electrode with deionized water.

- Verify the sensitivity of the electrodes by noting the change in millivolt reading when the pH of the test solution is altered. The ORP will increase when the pH of the test solution decreases and the ORP will decrease if the test solution pH is increased. Place the sample in a clean container and agitate the sample. Insert the electrodes and note the ORP drops sharply when the caustic is added (i.e., pH is raised) thus indicating the electrodes are sensitive and operating properly. If the ORP increases sharply when the caustic is added, the polarity is reversed and must be corrected in accordance with the manufacturer's instructions. If the ORP does not respond as above when the caustic is added, the electrodes shall be cleaned and the above procedure repeated.

- After the assembly has been checked for sensitivity, wash the electrodes with three changes of water or by means of a flowing stream of deionized water from a wash bottle. Place the sample in a clean container and insert the electrodes. Set temperature compensator throughout the measurement period. Read the millivolt potential of the solution, allowing sufficient time for the system to stabilize and reach temperature equilibrium. Measure successive portions of the sample until readings on two successive portions differ by no more than 10 mV. A system that is very slow to stabilize properly will not yield a meaningful ORP. Record all results in a field logbook or sample logsheet, including ORP (to nearest 10 mV), sample temperature and pH at the time of measurement.

5.5.6 Measurement of Turbidity

5.5.6.1 General

Turbidity in water is caused by suspended matter, such as clay, silt, finely divided organic and inorganic matter, soluble colored organic compounds, and microscopic organisms, including plankton. Turbidity is an expression of the optical property that causes light to be scattered and absorbed rather than transmitted in a straight line through the sample.

It is important to obtain a turbidity reading immediately after taking a sample, since irreversible changes in turbidity may occur if the sample is stored too long.

5.5.6.2 Principles of Equipment Operation

Turbidity is measured by the Nephelometric Method. This method is based on a comparison of the intensity of light scattered by the sample under defined conditions with the intensity of light scattered by a standard reference suspension under the same conditions. The higher the scattered light intensity, the higher the turbidity.

Formazin polymer is used as the reference turbidity standard suspension because of its ease of preparation combined with a higher reproducibility of its light-scattering properties than clay or turbid natural water. The turbidity of a specified concentration of formazin suspension is defined as 40 nephelometric units. This same suspension has an approximate turbidity of 40 Jackson units when measured on the candle turbidmeter. Therefore, nephelometric turbidity units (NTU) based on the formazin preparation will approximate units derived from the candle turbidimeter but will not be identical to them.

5.5.6.3 Equipment

The following equipment is needed for turbidity measurement:

- Stand alone portable turbidity meter, or combination meter (e.g., Horiba U-10), or combination meter equipped with an in-line sample chamber.
- Calibration solution, as specified by the manufacturer.
- Manufacturer's operation manual.

5.5.6.4 Measurement~~s~~ Techniques for TURBIDITY ~~Specific Conductance~~

The steps involved in taking turbidity measurements are listed below (standardization AND CALIBRATION is according to manufacturer's instructions):

- Check batteries and calibrate instrument before going into the field.
- Check the expiration date (etc.) of the solutions used for field calibration.
- Calibrate on a daily use basis, according to the manufacturer's instructions and record all pertinent information INCLUDING CALIBRATION STANDARD LOT NUMBERS on an equipment calibration log sheet.
- Rinse the cell with one or more portions of the sample to be tested or with deionized water.
- Immerse the probe in the sample and measure the turbidity. The reading must be taken immediately as suspended solids will settle over time resulting in a lower, inaccurate turbidity reading.

- Read and record the results in a field logbook or sample log sheet. Include a physical description of the sample, including color, qualitative estimate of turbidity, etc.

- Rinse the electrode with deionized water.

5.6 Sampling

5.6.1 Sampling Plan

The sampling approach consisting of the following, shall be developed as part of the project plan documents which are approved prior to beginning work in the field:

- Background and objectives of sampling.

- Brief description of area and waste characterization.

- Identification of sampling locations, with map or sketch, and applicable well construction data (well size, depth, screened interval, reference elevation).

- Intended number, sequence volumes, and types of samples. If the relative degrees of contamination between wells is unknown or insignificant, a sampling sequence which facilitates sampling logistics may be followed. Where some wells are known or strongly suspected of being highly contaminated, these shall be sampled last to reduce the risk of cross-contamination between wells as a result of the sampling procedures.

- Sample preservation requirements.

- Work schedule.

- List of team members.

- List of observers and contacts.

- Other information, such as the necessity for a warrant or permission of entry, requirement for split samples, access problems, location of keys, etc.

5.6.2 Sampling Methods

The collection of a groundwater sample consists of the following steps:

1. The site Health & Safety Officer (or designee) will first open the well cap and use volatile organic detection equipment (PID or FID) on the escaping gases at the well head to determine the need for respiratory protection.

2. When proper respiratory protection has been donned, sound the well for total depth and water level (using clean equipment) and record these data on a groundwarer sampling log sheet (see SOP SA-6.3); then calculate the fluid volume in the well pipe (as previously described in this SOP).

3. Calculate well volume to be removed as stated in Section 5.3.

STABILIZATION IS ACHIEVED AND SAMPLING CAN BEGIN WHEN THREE CONSECUTIVE READINGS, TAKEN AT 5- TO 10-MINUTE INTERVALS, ARE WITHIN THE FOLLOWING LIMITS:

- PH ±0.1 STANDARD UNIT
- SPECIFIC CONDUCTANCE ±3 PERCENT
- TEMPERATURE ±3 PERCENT
- TURBIDITY LESS THAN 10 NEPHELOMETRIC TURBIDITY UNITS

4. Select the appropriate purging equipment (see Attachment A). If an electric submersible pump with packer is chosen, go to Step 10.

5. Lower the purging equipment or intake into the well to a short distance below the water level and begin water removal. Collect the purged water and dispose of it in an acceptable manner (as applicable). Lower the purging device, as required, to maintain submergence.

6. Measure the rate of discharge frequently. A graduated bucket and stopwatch are most commonly used; other techniques include use of pipe trajectory methods, weir boxes or flow meters.

7. Observe the peristaltic pump intake for degassing "bubbles." If bubbles are abundant and the intake is fully submerged, this pump is not suitable for collecting samples for volatile organics. Never collect volatile organics samples using a vacuum pump.

8. Purge a minimum of three to five casing volumes before sampling. In low-permeability strata (i.e., if the well is pumped to dryness), one volume will suffice. Purged water shall be collected in a designated container and disposed in an acceptable manner.

9. If sampling using a pump, lower the pump intake to midscreen (or the middle of the open section in uncased wells) and collect the sample. If sampling with a bailer, lower the bailer to the sampling level before filling.

10. (For pump and packer assembly only). Lower the assembly into the well so that the packer is positioned just above the screen or open section. Inflate the packer. Purge a volume equal to at least twice the screened interval (or unscreened open section volume below the packer) before sampling. Packers shall always be tested in a casing section above ground to determine proper inflation pressures for good sealing.

11. In the event that recovery time of the well is very slow (e.g., 24 hours or greater), sample collection can be delayed until the following day. If the well has been purged early in the morning, sufficient water may be standing in the well by the day's end to permit sample collection. If the well is incapable of producing a sufficient volume of sample at any time, take the largest quantity available and record this occurrence in the site logbook.

12. Fill sample containers (preserve and label as described in SOPs SA-6.2 and SA-6.1).

13. Replace the well cap and lock as appropriate. Make sure the well is readily identifiable as the source of the samples.

14. Process sample containers as described in SOPs SA-6.2 and SA-6.1.

15. Decontaminate equipment as described in SOP SA-7.1.

5.7 Low Flow Purging and Sampling

5.7.1 Scope & Application

Low flow purging and sampling techniques are sometimes required for groundwater sampling activities. The purpose of low flow purging and sampling is to collect groundwater samples that contain

"representative" amounts of mobile organic and inorganic constituents in the vicinity of the selected open well interval, at near natural flow conditions. The minimum stress procedure emphasizes negligible water level drawdown and low pumping rates in order to collect samples with minimal alterations in water chemistry. This procedure is designed primarily to be used in wells with a casing diameter of 2 inches or more and a saturated screen, or open interval, length of ten feet or less. Samples obtained are suitable for analyses of common types of groundwater contaminants (volatile and semi-volatile organic compounds, pesticides, PCBs, metals and other inorganic ions [cyanide, chloride, sulfate, etc.]). This procedure is not designed to collect non-aqueous phase liquids samples from wells containing light or dense non-aqueous phase liquids (LNAPLs or DNAPLs), using the low flow pumps.

The procedure is flexible for various well construction types and groundwater yields. The goal of the procedure is to obtain a turbidity level of less than 5 NTU and to achieve a water level drawdown of less than 0.3 feet during purging and sampling. If these goals cannot be achieved, sample collection can take place provided the remaining criteria in this procedure are met.

5.7.2 Equipment

The following equipment is required (as applicable) for low flow purging and sampling:

- Adjustable rate, submersible pump (e.g., centrifugal or bladder pump constructed of stainless steel or Teflon). Peristaltic pumps may be used only for inorganic sample collection.
- Disposable clear plastic bottom filling bailers may be used to check for and obtain samples of LNAPLs or DNAPLs.
- Tubing - Teflon, Teflon lined polyethylene, polyethylene, PVC, tygon steel tubing can be used to collect samples for analysis, depending on the analyses to be performed and regulatory requirements.
- Water level measuring device, 0.01 foot accuracy, (electronic devices are preferred for tracking water level drawdown during all pumping operations).
- Flow measurement supplies.
- Interface probe, if needed.
- Power source (generator, nitrogen tank, etc.). If a gasoline generator is used, it must be located downwind and at a safe distance from the well so that the exhaust fumes do not contaminate the samples.
- Indicator parameter monitoring instruments - pH, turbidity, specific conductance, and temperature. Use of a flow-through cell is recommended. Optional Indicators - eH and dissolved oxygen, flow-through cell is required. Standards to perform field calibration of instruments.
- Decontamination supplies.
- Logbook(s), and other forms (e.g., well purging forms).
- Sample Bottles.

- Sample preservation supplies (as required by the analytical methods).
- Sample tags or labels.
- Well construction data, location map, field data from last sampling event.
- Field Sampling Plan.
- PID or FID instrument for measuring VOCs (volatile organic compounds).

5.7.3 Purging and Sampling Procedure

Use a submersible pump to purge and sample monitoring wells which have a 2.0 inch or greater well casing diameter.

Measure and record the water level immediately prior to placing the pump in the well.

Lower pump, safety cable, tubing and electrical lines slowly into the well so that the pump intake is located at the center of the saturated screen length of the well. If possible keep the pump intake at least two feet above the bottom of the well, to minimize mobilization of sediment that may be present in the bottom of the well. Collection of turbid free water samples may be difficult if there is three feet or less of standing water in the well.

When starting the pump, slowly increase the pump speed until a discharge occurs. Check water level. Adjust pump speed to maintain little or no water level drawdown. The target drawdown should be less than 0.3 feet and it should stabilize. If the target of less than 0.3 feet cannot be achieved or maintained, the sampling is acceptable if remaining criteria in the procedure are met. Subsequent sampling rounds will probably have intake settings and extraction rates that are comparable to those used in the initial sampling rounds.

Monitor water level and pumping rate every three to five minutes (or as appropriate) during purging. Record pumping rate adjustments and depths to water. Pumping rates should, as needed, be reduced to the minimum capabilities of the pump (e.g., 0.1-0.2 l/min) to ensure stabilization of indicator parameters. Adjustments are best made in the first fifteen minutes of pumping in order to help minimize purging time. During initial pump start-up, drawdown may exceed the 0.3 feet target and then recover as pump flow adjustments are made (minimum purge volume calculations should utilize stabilized drawdown values, not the initial drawdown). If the recharge rate of the well is less than minimum capability of the pump do not allow the water level to fall to the intake level (if the static water level is above the screen, avoid lowering the water level into the screen). Shut off the pump if either of the above is about to occur and allow the water level to recover. Repeat the process until field indicator parameters stabilize and the minimum purge volume is removed. The minimum purge volume with negligible drawdown (0.3 feet or less) is two saturated screen length volumes. In situations where the drawdown is greater than 0.3 feet and has stabilized, the minimum purge volume is two times the saturated screen volume plus the stabilized drawdown volume. After the minimum purge volume is attained (and field parameters have stabilized) begin sampling. For low yields wells, commence sampling as soon as the well has recovered sufficiently to collect the appropriate volume for all anticipated samples.

During well purging, monitor field indicator parameters (turbidity, temperature, specific conductance, pH, etc.) every three to five minutes (or as appropriate). Purging is complete and sampling may begin when

all field indicator parameters have stabilized (variations in values are within ten percent of each other, pH +/- 0.2 units, for three consecutive readings taken at three to five minute intervals). If the parameters have stabilized, but turbidity remains above 5 NTU goal, decrease pump flow rate, and continue measurement of parameters every three to five minutes. If pumping rate cannot be decreased any further and stabilized turbidity values remain above 5 NTU goal record this information. Measurements of field parameters should be obtained (as per Section 5.5) and recorded.

VOC samples are preferably collected first and directly into pre-preserved sample containers. Fill all sample containers by allowing the pump discharge to flow gently down the inside of the container with minimal turbulence.

If the water column in the pump tubing collapses (water does not completely fill the tubing) before exiting the tubing, use one of the following procedures to collect VOC samples: (1) Collect the non-VOCs samples first, then increase the flow rate incrementally until the water column completely fills the tubing, collect the sample and record the new flow rate; (2) reduce the diameter of the existing tubing until the water column fills the tubing either by adding a connector (Teflon or stainless steel), or clamp which should reduce the flow rate by constricting the end of the tubing; (3) insert a narrow diameter Teflon tube into the pump's tubing so that the end of the tubing is in the water column and the other end of the tubing protrudes beyond the pump's tubing, collect sample from the narrow diameter tubing.

Prepare samples for shipping as per SOP SA-6.1.

6.0 REFERENCES

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ATTACHMENT A

PURGING EQUIPMENT SELECTION

Diameter Casing		Bailer	Peristaltic Pump	Vacuum Pump	Air-lift	Diaphragm "Trash" Pump	Submersible Diaphragm Pump	Submersible Electric Pump	Submersible Electric Pump w/Packer
1.25-Inch	Water level <25 feet		X	X	X	X			
	Water Level >25 feet				X				
2-Inch	Water level <25 feet	X	X	X	X	X	X		
	Water Level >25 feet	X			X		X		
4-Inch	Water level <25 feet	X	X	X	X	X	X	X	X
	Water Level >25 feet	X			X		X	X	X
6-Inch	Water level <25 feet				X	X		X	X
	Water Level >25 feet				X			X	X
8-Inch	Water level <25 feet				X	X		X	X
	Water Level >25 feet				X			X	X

ATTACHMENT A
PURGING EQUIPMENT SELECTION
PAGE 2

Manufacturer	Model Name/Number	Principle of Operation	Maximum Outside Diameter/ Length (Inches)	Construction Materials (w/Lines and Tubing)	Lift Range (ft)	Delivery Rates or Volumes	1982 Price (Dollars)	Comments
BarCad Systems, Inc.	BarCad Sampler	Dedicated; gas drive (positive displacement)	1.5/16	PE, brass, nylon, aluminum oxide	0-150 with std. tubing	1 liter for each 10-15 feet of submergence	$220-350	Requires compressed gas; custom sizes and materials available; acts as piezometer.
Cole-Parmer Inst. Co.	Master Flex 7570 Portable Sampling Pump	Portable; peristaltic (suction)	<1.0/NA	(not submersible) Tygon®, silicone Viton®	0-30	670 mL/min with 7015-20 pump head	$500-600	AC/DC; variable speed control available; other models may have different flow rates.
ECO Pump Corp.	SAMPLifier	Portable; venturi	<1.5 or <2.0/NA	PP, PE, PVC, SS, Teflon®, Tefzel®	0-100	0-500 mL/min depending on lift	$400-700	AC, DC, or gasoline-driven motors available; must be primed.
Geltek Corp.	Bailer 219-4	Portable; grab (positive displacement)	1.66/38	Teflon®	No limit	1,075 mL	$120-135	Other sizes available.
GeoEngineering, Inc.	GEO-MONITOR	Dedicated; gas drive (positive displacement)	1.5/16	PE, PP, PVC, Viton®	Probably 0-150	Approximately 1 liter for each 10 feet of submergence	$185	Acts as piezometer; requires compressed gas.
Industrial and Environmental Analysts, Inc. (IEA)	Aquarius	Portable; bladder (positive displacement)	1.75/43	SS, Teflon®, Viton®	0-250	0-2,800 mL/min	$1,500-3,000	Requires compressed gas; other models available; AC, DC, manual operation possible.
IEA	Syringe Sampler	Portable; grab (positive displacement)	1.75/43	SS, Teflon®	No limit	850 mL sample volum e	$1,100	Requires vacuum and/or pressure from hand pump.
Instrument Specialties Co. (ISCO)	Model 2600 Well Sampler	Portable; bladder (positive displacement)	1.75/50	PC, silicone, Teflon®, PP, PE, Detrin®, acetal	0-150	0-7,500 mL/min	$990	Requires compressed gas (40 psi minimum).
Keck Geophysical Instruments, Inc.	SP-81 Submersible Sampling Pump	Portable; helical rotor (positive displacement)	1.75/25	SS, Teflon®, PP, EPDM, Viton®	0-160	0-4,500 mL/min	$3,500	DC operated.
Leonard Mold and Die Works, Inc.	GeoFilter Small Diameter Well Pump (#0500)	Portable; bladder (positive displacement)	1.75/38	SS, Teflon®, PC, Neoprene®	0-400	0-3,500 mL/min	$1,400-1,500	Requires compressed gas (55 psi minimum); pneumatic or AC/DC control module.
Oil Recovery Systems, Inc.	Surface Sampler	Portable; grab (positive displacement)	1.75/12	acrylic, Detrin®	No limit	Approximately 250 mL	$125-160	Other materials and models available; for measuring thickness of "floating" contaminants.
Q.E.D. Environmental Systems, Inc.	Well Wizard® Monitoring System (P-100)	Dedicated; bladder (positive displacement)	1.66/36	PVC	0-230	0-2,000 mL/min	$300-400	Requires compressed gas; piezometric level indicator; other materials available.

ATTACHMENT A
PURGING EQUIPMENT SELECTION
PAGE 3

Manufacturer	Model Name/Number	Principle of Operation	Maximum Outside Diameter/ Length (Inches)	Construction Materials (w/Lines and Tubing)	Lift Range (ft)	Delivery Rates or Volumes	1982 Price (Dollars)	Comments
Randolph Austin Co.	Model 500 Vari-Flow Pump	Portable; peristaltic (suction)	<0.5/NA	(Not submersible) Rubber, Tygon®, or Neoprene®	0-30	See comments	$1,200-1,300	Flow rate dependent on motor and tubing selected; AC operated; other models available.
Robert Bennett Co.	Model 180	Portable; piston (positive displacement)	1.8/22	SS, Teflon®, Delrin® PP, Viton®, acrylic, PE	0-500	0-1,800 mL/min	$2,600-2,700	Requires compressed gas; water level indicator and flow meter; custom models available.
Slope Indicator Co. (SINCO)	Model 514124 Pneumatic Water Sampler	Portable; gas drive (positive displacement)	1.9/18	PVC, nylon	0-1,100	250 mL/ flushing cycle	$250-350	Requires compressed gas; SS available; piezometer model available; dedicated model available.
Solinst Canada Ltd.	5W Water Sampler	Portable; grab (positive displacement)	1.9/27	PVC, brass, nylon, Neoprene®	0-330	500 mL	$1,300-1,800	Requires compressed gas; custom models available.
TIMCO Mfg. Co., Inc.	Std. Bailer	Portable; grab (positive displacement)	1.66/Custom	PVC, PP	No limit	250 mL/ft of bailer	$20-60	Other sizes, materials, models available; optional bottom-emptying device available; no solvents used.
TIMCO	Air or Gas Lift Sampler	Portable; gas drive (positive displacement)	1.66/30	PVC, Tygon®, Teflon®	0-150	350 mL/ flushing cycle	$100-200	Requires compressed gas; other sizes, materials, models available; no solvents used.
Tole Devices Co.	Sampling Pump	Portable; bladder (positive displacement)	1.38/48	SS, silicone, Delrin®, Tygon®	0-125	0-4,000 mL/min	$800-1,000	Compressed gas required; DC control module; custom built.

Construction Material Abbreviations:

- PE Polyethylene
- PP Polypropylene
- PVC Polyvinyl chloride
- SS Stainless steel
- PC Polycarbonate
- EPDM Ethylene-propylene diene (synthetic rubber)

Other Abbreviations:

- NA Not applicable
- AC Alternating current
- DC Direct current

NOTE: Other manufacturers market pumping devices which could be used for groundwater sampling, though not expressly designed for this purpose. The list is not meant to be all-inclusive and listing does not constitute endorsement for use. Information in the table is from sales literature and/or personal communication. No skimmer, scavenger-type, or high-capacity pumps are included.

Source: Barcelona et al., 1983.

ATTACHMENT B

SPECIFIC CONDUCTANCE OF 1 MOLAR KCl AT VARIOUS TEMPERATURES[1]

Temperature (°C)	Specific Conductance (umhos/cm)
15	1,147
16	1,173
17	1,199
18	1,225
19	1,251
20	1,278
21	1,305
22	1,332
23	1,359
24	1,368
25	1,413
26	1,441
27	1,468
28	1,496
29	1,524
30	1,552

[1] Data derived from the International Critical Tables 1-3-8.

ATTACHMENT C

VARIATION OF DISSOLVED OXYGEN CONCENTRATION IN WATER AS A FUNCTION OF TEMPERATURE AND SALINITY

Temperature (°C)	Dissolved Oxygen (mg/L)					
	Chloride Concentration in Water					Difference/ 100 mg Chloride
	0	5,000	10,000	15,000	20,000	
0	14.6	13.8	13.0	12.1	11.3	0.017
1	14.2	13.4	12.6	11.8	11.0	0.016
2	13.8	13.1	12.3	11.5	10.8	0.015
3	13.5	12.7	12.0	11.2	10.5	0.015
4	13.1	12.4	11.7	11.0	10.3	0.014
5	12.8	12.1	11.4	10.7	10.0	0.014
6	12.5	11.8	11.1	10.5	9.8	0.014
7	12.2	11.5	10.9	10.2	9.6	0.013
8	11.9	11.2	10.6	10.0	9.4	0.013
9	11.6	11.0	10.4	9.8	9.2	0.012
10	11.3	10.7	10.1	9.6	9.0	0.012
11	11.1	10.5	9.9	9.4	8.8	0.011
12	10.8	10.3	9.7	9.2	8.6	0.011
13	10.6	10.1	9.5	9.0	8.5	0.011
14	10.4	9.9	9.3	8.8	8.3	0.010
15	10.2	9.7	9.1	8.6	8.1	0.010
16	10.0	9.5	9.0	8.5	8.0	0.010
17	9.7	9.3	8.8	8.3	7.8	0.010
18	9.5	9.1	8.6	8.2	7.7	0.009
19	9.4	8.9	8.5	8.0	7.6	0.009
20	9.2	8.7	8.3	7.9	7.4	0.009
21	9.0	8.6	8.1	7.7	7.3	0.009
22	8.8	8.4	8.0	7.6	7.1	0.008
23	8.7	8.3	7.9	7.4	7.0	0.008
24	8.5	8.1	7.7	7.3	6.9	0.008
25	8.4	8.0	7.6	7.2	6.7	0.008

ATTACHMENT C
VARIATION OF DISSOLVED OXYGEN CONCENTRATION IN WATER AS A FUNCTION OF TEMPERATURE AND SALINITY
PAGE TWO

Temperature (°C)	Dissolved Oxygen (mg/L)					
	Chloride Concentration in Water					Difference/ 100 mg Chloride
	0	5,000	10,000	15,000	20,000	
26	8.2	7.8	7.4	7.0	6.6	0.008
27	8.1	7.7	7.3	6.9	6.5	0.008
28	7.9	7.5	7.1	6.8	6.4	0.008
29	7.8	7.4	7.0	6.6	6.3	0.008
30	7.6	7.3	6.9	6.5	6.1	0.008
31	7.5					
32	7.4					
33	7.3					
34	7.2					
35	7.1					
36	7.0					
37	6.9					
38	6.8					
39	6.7					
40	6.6					
41	6.5					
42	6.4					
43	6.3					
44	6.2					
45	6.1					
46	6.0					
47	5.9					
48	5.8					
49	5.7					
50	5.6					

Note: In a chloride solution, conductivity can be roughly related to chloride concentration (and therefore, used to correct measured D.O. concentration) using Attachment B.

TABLE OF CONTENTS

SECTION **PAGE**

1.0 PURPOSE 2

2.0 SCOPE 2

3.0 GLOSSARY 2

4.0 RESPONSIBILITIES 3

5.0 PROCEDURES 3

5.1 Sample Containers 3
5.2 Sample Preservation 4
5.2.1 Overview 4
5.2.2 Preparation and Addition of Reagents 4
5.3 Field Filtration 6
5.4 Sample Packaging and Shipping 6
5.4.1 Environmental Samples 6
5.4.2 Determination of Shipping Classification for Hazardous Material Samples 7
5.4.3 Packaging and Shipping of Samples Classified as Flammable (or Solid) 8
5.5 Shipment of Lithium Batteries 10

6.0 REFERENCES 11

ATTACHMENTS

A GENERAL SAMPLE CONTAINER AND PRESERVATION REQUIREMENTS 12
B ADDITIONAL REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES 13
C DOT HAZARDOUS MATERIAL CLASSIFICATION (49 CFR 173.2(a)) 16
D GUIDE FOR HAZARDOUS MATERIALS SHIPPERS 18
E HAZARDOUS MATERIALS SHIPPING CHECK LIST 20
F DOT SEGREGATION AND SEPARATION CHART 21
G LITHIUM BATTERY SHIPPING PAPERS 22

1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to provide information on sample preservation, packaging, and shipping procedures to be used in handling environmental samples submitted for chemical constituent, biological, or geotechnical analysis. Sample chain-of-custody procedures and other aspects of field documentation are addressed in SOP SA-6.3. Sample identification is addressed in SOP CT-04.

2.0 SCOPE

This procedure:

- Describes the appropriate containers to be used for samples depending on the analyses to be performed, and the steps necessary to preserve the samples when shipped off site for chemical analysis.
- Provides instruction for sample packaging and shipping in accordance with current U.S. Department of Transportation (DOT) regulations.

3.0 GLOSSARY

Hazardous Material - A substance or material which has been determined by the Secretary of Transportation to be capable of posing an unreasonable risk to health, safety, and property when transported in commerce, and which has been so designated. Under 49 CFR, the term includes hazardous substances, hazardous wastes, marine pollutants, and elevated temperature materials, as well as materials designated as hazardous under the provisions of §172.101 and §172.102 and materials that meet the defining criteria for hazard classes and divisions in Part 173.

Hazardous Waste - Any substance listed in 40 CFR, Subpart D (ÿ261.30 et seq.), or otherwise characterized as ignitable, corrosive, reactive, or toxic (as defined by Toxicity Characteristic Leaching Procedure, TCLP, analysis) as specified under 40 CFR, Subpart C (ÿ261.20 et seq.), that would be subject to manifest requirements specified in 40 CFR 262. Such substances are defined and regulated by EPA.

Marking - A descriptive name, identification number, instructions, cautions, weight, specification or UN marks, or combination thereof required on outer packaging of hazardous materials.

n.o.i - Not otherwise indicated (may be used interchangeably with n.o.s.).

n.o.s. - Not otherwise specified.

ORM - Other regulated material (see DOT 49 CFR 173.144).

Packaging - A receptacle and any other components or materials necessary for compliance with the minimum packaging requirements of 49 CFR 174, including containers (other than freight containers or overpacks), portable tanks, cargo tanks, tank cars, and multi-unit tank-car tanks to perform a containment function in conformance with the minimum packaging requirements of 49 CFR 173.24(a) & (b).

Placard - Color-coded, pictorial sign which depicts the hazard class symbol and name and which is placed on the side of a vehicle transporting certain hazardous materials.

Common Preservatives:

- Hydrochloric Acid - HCl
- Sulfuric Acid - H_2SO_4
- Nitric Acid - HNO_3
- Sodium Hydroxide - NaOH

Other Preservatives

- Zinc Acetate
- Sodium Thiosulfate - $Na_2S_2O_3$

Normality (N) - Concentration of a solution expressed as equivalent per liter, an equivalent being the amount of a substance containing 1 gram-atom of replaceable hydrogen or its equivalent. Thus, a one-molar solution of HCl, containing 1 gram-atom of H, is "one normal," whereas a one-molar solution of H_2SO_4, containing 2 gram-atoms of H, is "two normal."

Reportable Quantity (RQ) - For the purposes of this SOP, means the quantity specified in column 3 of the Appendix to DOT 49 CFR §172.101 for any material identified in column 1 of the appendix. A spill greater than the amount specified must be reported to the National Response Center.

Sample - A sample is physical evidence collected from a facility or the environment, which is representative of conditions at the location and time of collection.

4.0 RESPONSIBILITIES

Field Operations Leader - Directly responsible for the bottling, preservation, labeling, packaging, shipping, and custody of samples up to and including release to the shipper.

Field Samplers - Responsible for initiating the Chain-of-Custody Record (per SOP SA-6.3), implementing the packaging and shipping requirements, and maintaining custody of samples until they are relinquished to another custodian or to the common carrier.

5.0 PROCEDURES

Sample identification, labeling, documentation, and chain-of-custody are addressed by SOP SA-6.3.

5.1 Sample Containers

Different types of chemicals react differently with sample containers made of various materials. For example, trace metals adsorb more strongly to glass than to plastic, whereas many organic chemicals may dissolve various types of plastic containers. Attachments A and B show proper containers (as well as other information) per 40 CFR 136. In general, the sample container shall allow approximately 5-10 percent air space ("ullage") to allow for expansion/vaporization if the sample warms during transport. However, for collection of volatile organic compounds, head space shall be omitted. The analytical laboratory will generally provide certified-clean containers for samples to be analyzed for chemical constituents. Shelby tubes or other sample containers are generally provided by the driller for samples requiring geotechnical analysis. Sufficient lead time shall be allowed for a delivery of bottle orders. Therefore, it is critical to use the correct container to maintain the integrity of the sample prior to analysis.

Once opened, the container must be used at once for storage of a particular sample. Unused but opened containers are to be considered contaminated and must be discarded; because of the potential for introduction of contamination, they cannot be reclosed and saved for later use. Likewise, any unused containers which appear contaminated upon receipt, or which are found to have loose caps or a missing Teflon liner (if required for the container), shall be discarded.

5.2 Sample Preservation

Many water and soil samples are unstable and therefore require preservation to prevent changes in either the concentration or the physical condition of the constituent(s) requiring analysis. Although complete and irreversible preservation of samples is not possible, preservation does retard the chemical and biological changes that inevitably take place after the sample is collected. Preservation techniques are usually limited to pH control, chemical addition(s), and refrigeration/ freezing (certain biological samples only).

5.2.1 Overview

The preservation techniques to be used for various analytes are listed in Attachments A and B. Reagents required for sample preservation will either be added to the sample containers by the laboratory prior to their shipment to the field or be added in the field (in a clean environment). Only high purity reagents shall be used for preservation. In general, aqueous samples of low-concentration organics (or soil samples of low- or medium-concentration organics) are cooled to 4°C. Medium-concentration aqueous samples and high-hazard organics samples are typically not preserved. Low-concentration aqueous samples for metals are acidified with HNO_3, whereas medium-concentration and high-hazard aqueous metal samples are not preserved. Low- or medium-concentration soil samples for metals are cooled to 4°C, whereas high-hazard samples are not preserved.

The following subsections describe the procedures for preparing and adding chemical preservatives. Attachments A and B indicate the specific analytes which require these preservatives.

5.2.2 Preparation and Addition of Reagents

Addition of the following acids or bases may be specified for sample preservation; these reagents shall be analytical reagent (AR) grade or purer and shall be diluted to the required concentration with deionized water before field sampling commences. To avoid uncontrolled reactions, be sure to Add Acid to water (not vice versa). A dilutions guide is provided below.

Acid/Base	Dilution	Concentration	Estimated Amount Required for Preservation
Hydrochloric Acid (HCl)	1 part concentrated HCl: 1 part double-distilled, deionized water	6N	5-10 mL
Sulfuric Acid (H_2SO_4)	1 part concentrated H_2SO_4: 1 part double-distilled, deionized water	18N	2 - 5 mL
Nitric Acid (HNO_3)	Undiluted concentrated HNO_3	16N	2 - 5 mL
Sodium Hydroxide (NaOH)	400 grams solid NaOH dissolved in 870 mL double-distilled, deionized water; yields 1 liter of solution	10N	2 mL

The amounts required for preservation shown in the above table assumes proper preparation of the preservative and addition of the preservative to one liter of aqueous sample (assuming that the sample is initially at pH 7, is poorly buffered, and does not contain particulate matter; as these conditions vary, more preservative may be required). Consequently, the final sample pH must be checked using narrow-range pH paper, as described in the generalized procedure detailed below:

- Pour off 5-10 mL of sample into a dedicated, clean container. Use some of this sample to check the initial sample pH using wide range (0-14) pH paper. Never dip the pH paper into the sample; always apply a drop of sample to the pH paper using a clean stirring rod or pipette.

- Add about one-half of the estimated preservative required to the original sample bottle. Cap and invert gently several times to mix. Check pH (as described above) using medium range pH paper (pH 0-6 or pH 7.5-14, as applicable).

- Cap sample bottle and seal securely.

Additional considerations are discussed below:

- To text if ascorbic acid must be used to remove oxidizing agents present in the sample before it can be properly preserved, place a drop of sample on KI-starch paper. A blue color indicates the need for ascorbic acid addition.

  If required, add a few crystals of ascorbic acid to the sample and retest with the KI-starch paper. Repeat until a drop of sample produces no color on the KI-starch paper. Then add an additional 0.6 grams of ascorbic acid per each liter of sample volume.

  Continue with proper base preservation of the sample as described, generally, above.

- Samples for sulfide analysis must be treated by the addition of 4 drops (0.2 mL) of 2N zinc acetate solution per 100 ml of sample.

  The 2N zinc acetate solution is made by dissolving 220 grams of zinc acetate in 870 mL of double-distilled, deionized water to make 1 liter of solution.

  The sample pH is then raised to 9 using the NaOH preservative.

- To test of sodium thiosulfate must be added to remove residual chlorine from a sample, test the sample for residual chlorine using a field test kit especially made for this purpose.

  If residual chlorine is present, add 0.08 grams of sodium thiosulfate per liter of sample to remove the residual chlorine.

  Continue with proper acidification of the sample as described, generally, above.

For biological samples, 10% buffered formalin or isopropanol may also be required for preservation. Questions regarding preservation requirements should be resolved through communication with the laboratory <u>before</u> sampling begins.

5.3 Field Filtration

At times, field-filtration may be required to provide for the analysis of dissolved chemical constituents. Field-filtration must be performed prior to the preservation of samples as described above. General procedures for field filtration are described below:

- The sample shall be filtered through a non-metallic, 0.45-micron membrane filter, immediately after collection. The filtration system shall consist of dedicated filter canister, dedicated silicon tubing, and a peristaltic pump with pressure or vacuum pumping squeeze action (since the sample is filtered by mechanical peristalsis, the sample travels only through the tubing).

- To perform filtration, thread the silicon tubing through the peristaltic pump head. Attach the filter canister to the discharge end of the silicon tubing (note flow direction arrow); attach the aqueous sample container to the intake end of the silicon tubing. Turn the peristaltic pump on and perform filtration.

- Continue by preserving the filtrate (contained in the filter canister), as applicable and generally described above.

5.4 Sample Packaging and Shipping

Samples collected for shipment from a site shall be classified as either environmental or hazardous material samples. Samples from drums containing materials other than Investigative Derived Waste (IDW) and samples obtained from waste piles or bulk storage tanks are generally shipped as hazardous materials. A distinction must be made between the two types of samples in order to:

- Determine appropriate procedures for transportation of samples (if there is any doubt, a sample shall be considered hazardous and shipped accordingly.)

- Protect the health and safety of transport and laboratory personnel receiving the samples (special precautions are used by the shipper and at laboratories when hazardous materials are received.)

Detailed procedures for packaging environmental and hazardous material samples are outlined in the remainder of this section.

5.4.1 Environmental Samples

Environmental samples are packaged as follows:

- Place sample container, properly identified and with lid securely fastened in a plastic bag (e.g. Ziploc baggie), and seal the bag.

- Place sample in a cooler constructed of sturdy material which has been lined with a large, plastic (e.g. "garbage" bag).

- Pack with enough noncombustible, absorbent, cushioning materials such as vermiculite (shoulders of bottles must be iced if required) to minimize the possibility of the container breaking.

- If cooling is required (see Attachments A and B), double-bag ice in Ziploc baggies and place around container shoulders, and on top of absorbent packing material (minimum of 8 pounds of ice for a medium-size cooler).

- Seal (i.e., tape or tie top in knot) large liner bag.

- Attach appropriate completed chain-of-custody form to inside lid of container (no DOT shipping papers are required).

- Close and seal outside of cooler as described in SOP SA-6.3. Signed custody seals must be used.

Coolers must be marked as containing "Environmental Samples." The appropriate side of the container must be marked "This End Up" and arrows placed appropriately. No DOT marking or labeling is required; there are no DOT restrictions on mode of transportation.

5.4.2 Determination of Shipping Classification for Hazardous Material Samples

Samples not determined to be environmental samples, or samples known or expected to contain hazardous materials, must be considered hazardous material samples and transported according to the requirements listed below.

5.4.2.1 Known Substances

If the substance in the sample is known or can be identified, package, mark, label, and ship according to the specific instructions for that material (if it is listed) in the DOT Hazardous Materials Table, 49 CFR 172.101. (DOT Guide for shippers can be found in Attachment D of this document.)

To determine the proper shipping name, use the following steps to help locate the shipping name on the Hazardous Materials Table, DOT 49 CFR 172.101.

1. Look first for the chemical or technical name of the material, for example, ethyl alcohol. Note that many chemicals have more than one technical name, for example, perchloroethylene (not listed in 172.101) is listed as tetrachloroethylene (listed 172.101). It may be useful to consult a chemist for all possible technical names a material can have. If your material is not listed by its technical name, then . . .

2. Look for the chemical family name. For example, pentyl alcohol is not listed but the chemical family name is: alcohol, n.o.s. (not otherwise specified). If the chemical family name is not listed, then . . .

3. Look for a generic name based on end use. For example, Paint, n.o.s or Fireworks, n.o.s. If a generic name based on end use is not listed, then . . .

4. Look for a generic family name based on end use, for example, drugs, n.o.s. or cosmetics, n.o.s. Finally, if your material is not listed by a generic family name but you suspect or know the material is hazardous because it meets the definition of one or more hazardous classes, then . . .

5. You will have to use the general hazard class for a proper shipping name. For example, Flammable Liquid, n.o.s, or Oxidizer, n.o.s.

5.4.2.2 Unknown Substances

For samples of hazardous substances of unknown content, select the appropriate transportation category according to the DOT hazardous materials classification of a material having more than one hazard. This procedure is outlined in DOT Regulation 49 CFR 173.2a. (This can be found in Attachment C of this SOP.)

The correct shipping classification for an unknown sample is selected through a process of elimination, as outlined in DOT Regulation 49 CFR 172.101(c)(11). By using the provisions in this paragraph, the proper shipping name and description will be determined. A step-by-step guide is provided by the Department of Transportation (DOT) and can be found in Attachment D of this SOP.

5.4.3 Packaging and Shipping of Samples Classified as Flammable Liquid (or Solid)

5.4.3.1 Packaging

Applying the word "flammable" to a sample does not imply that it is in fact flammable. The word prescribes the class of packaging according to DOT regulations.

1. Containerize sample as required (see Attachments A and B). To prevent leakage, fill container no more than 90 percent full. Seal lid with teflon tape or wire.

2. Complete sample label and attach securely to sample container.

3. Seal container and place in 2-mil-thick (or thicker) polyethylene bag (e.g., Ziploc baggie), one sample per bag. Position sample identification label so that it can be read through bag. Seal bag.

4. For soil jars, place sealed bag inside metal can (available from laboratory or laboratory supplier) and cushion it with enough noncombustible, absorbent material (for example, vermiculite or diatomaceous earth) between the bottom and sides of the can and bag to prevent breakage and absorb leakage. Pack one bag per can. Use clips, tape, or other positive means to hold can lid securely, tightly and permanently. Mark can as indicated in Paragraph 1 of Section 5.3.4.2, below. Single 1-gallon bottles do not need to be placed in metal cans.

5. Place one or more metal cans (or a single 1-gallon bottle) into a strong outside container, such as a metal picnic cooler or a DOT-approved fiberboard box. Surround cans (or bottle) with noncombustible, absorbent cushioning materials for stability during transport. The absorbent material should be able to absorb the entire contents of the container. Mark container as indicated in Paragraph 2 below.

5.4.3.2 Marking/Labeling

1. Use abbreviations only where specified. Place the following information, either hand-printed or in label form, on the metal can (or 1-gallon bottle):

   - Laboratory name and address.

- Proper shipping name from the hazardous materials table (DOT Regulation CFR 49 172.101). Example: "Flammable Liquid, n.o.s. UN1993" or "Flammable Solid, n.o.s. UN1325." This will include packing group (see Section 5.3.4.2, No. 2.)

Not otherwise specified (n.o.s) is not used if the flammable liquid (or solid) is identified. If identified, the name of the specific material is listed before the category (for example, Acetone, Flammable Liquid), followed by its appropriate UN number found in the DOT Hazardous Materials table (49 CFR 172.101).

2. Determine packing group. The packing group is part of the proper shipping name and must be included on the shipping papers in the description section.

   I. Most Hazardous
   II. Medium Hazard
   III. Least Hazardous

   The packing group will be listed in the hazardous materials table, column 5.

3. Place all information on outside shipping container as on can (or bottle), specifically:

   - Proper shipping name
   - UN or NA number
   - Proper label(s)
   - Addressee and sender

   Place the following labels on the outside shipping container: "Cargo Aircraft Only" and DOT label such as: "Flammable Liquid" (or "Flammable Solid"). "Dangerous When Wet" label shall be used if the Flammable Solid has not been exposed to a wet environment. "Laboratory Samples" and "THIS SIDE UP" or "THIS END UP" shall also be marked on the top of the outside container, and upward-pointing arrows shall be placed on all four sides of the container.

5.4.3.3 <u>Shipping Papers</u>

1. Use abbreviations only where specified. Complete the carrier-provided bill of lading and sign certification statement. Provide the following information in the order listed (one form may be used for more than one exterior container):

   - Proper shipping name. (Example: "Flammable Liquid, n.o.s. UN1993" or "Flammable Solid, n.o.s. UN1325 Packing Group I, II, III").
   - "Limited Quantity" (or "Ltd. Qty."). (See No. 3, below.)
   - "Cargo Aircraft Only."
   - Net weight (wt) or net volume (vol), just before or just after "Flammable Liquid, n.o.s." or "Flammable Solid, n.o.s.," by item, if more than one metal can is inside an exterior container.
   - "Laboratory Samples" (if applicable).

2. Include Chain-of-Custody Record, properly executed in outside container; use custody seals.

3. "Limited Quantity" means the maximum amount of a hazardous material for which there is a specific labeling or packaging exception (DOT CFR 49 171.8). This may mean that packages are exempted from labeling requirements. To determine if your sample meets the Limited Quantity Exception, refer to DOT Regulation CFR 49 Subpart C 173.50 through 173.156. First, determine the proper classification and shipping name for the material; then refer to the exception requirements for that particular class of material beginning with 173.50.

   Example: "Flammable Liquid n.o.s. UN1993 Packing Group 1." The outer package can weigh no more than 66 pounds gross weight. The inner package or container can weigh no more than 0.1 gallon net capacity for each container.

   To determine whether the material can be shipped as a "Limited Quantity," you must check the specific requirement for that class of material.

5.4.3.4 Transportation

1. The majority of unknown hazardous substance samples will be classified as flammable liquids. The samples will be transported by rented or common carrier truck, railroad, or express overnight package services. Do not transport samples on any passenger-carrying air transport system, even if the system has cargo-only aircraft. DOT regulations permit regular airline cargo-only aircraft, but difficulties with most suggest avoiding them. Instead, ship by airline carriers that carry only cargo. If unsure of what mode of transportation to use, consult the FOL or Project Manager.[1]

2. For transport by government-owned vehicle, including aircraft, DOT regulations do not apply. However, procedures described above, with the exception of execution of the bill of lading with certification, shall still be followed.

3. Use the hazardous materials shipping check list (Attachment E) as a guidance to ensure that all sample-handling requirements are satisfied.

4. In some cases, various materials may react if they break during shipment. To determine if you are shipping such materials, refer to the DOT compatibility chart in Attachment F.

5.5 Shipment of Lithium Batteries

Monitoring well data are analyzed using either the Hermit SE 1000 or the Hermit SE 2000 environmental data logger. These instruments are powered by lithium batteries. The Department of Transportation has determined that lithium batteries are a hazardous material and are to be shipped using the following information:

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[1] Note: If you are unsure as how to ship the sample (hazardous or environmental sample), contact the FOL or Project Manager so that a decision can be made as to the proper shipping practices. The DOT penalties for improper shipment of a hazardous material are stringent and may include a prison term for intentional violations.

- Product Designation
  - Hermit SE 1000
  - Hermit SE 2000

- DOT Proper Shipping Name
  - Lithium batteries, contained in equipment, UN3091

- Classification or Division
  - Class 9

Shipment of equipment containing lithium batteries must be accompanied by shipping papers completed as indicated in Attachment G. The instrument will be shipped by Federal Express as a Hazardous Material. Place the instrument in the same container in which it was received. This container or case is a DOT-approved shipping container. For Federal Express procedures to ship hazardous materials, call 1-800-238-5355, extension 922-1666. In most cases, the return shipping papers and DOT labels will be shipped to you from the company warehouse or the vendor. An example of the types of labels used for shipment and the wording are shown in Attachment G. These labels will be attached to the outside container with the following wording:

- Lithium Batteries Contained in Equipment
  - UN-3091
  - Shipped Under CA-9206009

6.0 REFERENCES

American Public Health Association, 1981. Standard Methods for the Examination of Water and Wastewater, 15th Edition. APHA, Washington, D.C.

U.S. Department of Transportation, 1993. Hazardous Materials Regulations, 49 CFR 171-177.

U.S. EPA, 1984. "Guidelines Establishing Test Procedures for the Analysis of Pollutants under Clean Water Act." Federal Register, Volume 49 (209), October 26, 1984, p. 43234.

U.S. EPA, 1979. Methods for Chemical Analysis of Water and Wastes. EPA-600/4-79-020, U.S. EPA-EMSL, Cincinnati, Ohio.

ATTACHMENT A

GENERAL SAMPLE CONTAINER AND PRESERVATION REQUIREMENTS

Sample Type and Concentration			Container[1]	Sample Size	Preservation[2]	Holding Time[2]
WATER						
Organics (GC&GC/MS)	VOC	Low	Borosilicate glass	2 x 40 mL	Cool to 4°C HCl to ≤ 2	14 days[3]
	Extractables (Low SVOCs and pesticide/PCBs)		Amber glass	2x2 L or 4x1 L	Cool to 4°C	7 days to extraction; 40 days after extraction
	Extractables (Medium SVOCs and pesticide/PCBs)		Amber glass	2x2 L or 4x1 L	None	7 days to extraction; 40 days after extraction
Inorganics	Metals	Low	High-density polyethylene	1 L	HNO_3 to pH ≤2	6 months (Hg-28 days)
		Medium	Wide-mouth glass	16 oz.	None	6 months
	Cyanide	Low	High-density polyethylene	1 L	NaOH to pH>12	14 days
	Cyanide	Medium	Wide-mouth glass	16 oz.	None	14 days
Organic/ Inorganic	High Hazard		Wide-mouth glass	8 oz.	None	14 days
SOIL						
Organics (GC&GC/MS)	VOC		Wide-mouth glass with teflon liner	2 x 4 oz.	Cool to 4°C	14 days
	Extractables (Low SVOCs and pesticides/PCBs)		Wide-mouth glass	8 oz.	Cool to 4°C	14 days to extraction; 40 days after extraction
	Extractables (Medium SVOCs and pesticides/PCBs)		Wide-mouth glass	8 oz.	Cool to 4°C	14 days to extraction; 40 days after extraction
Inorganics	Low/Medium		Wide-mouth glass	8 oz.	Cool to 4°C	6 months (Hg - 28 days) Cyanide (14 days)
Organic/ Inorganic	High Hazard		Wide-mouth glass	8 oz.	None	NA
Dioxin/Furan	All		Wide-mouth glass	4 oz.	None	7 days until extraction; 40 days after extraction
TCLP	All		Wide-mouth glass	8 oz.	None	7 days until preparation; analysis as per fraction
AIR						
Volatile Organics	Low/Medium		Charcoal tube – 7 cm long, 6 mm OD, 4 mm ID	100 L air	Cool to 4°C	5 days recommended

(1) All glass containers should have Teflon cap liners or septa.
(2) See Attachment E. Preservation and maximum holding time allowances per 40 CFR 136.

ATTACHMENT B

ADDITIONAL REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES

Parameter Number/Name	Container(1)	Preservation(2)(3)	Maximum Holding Time(4)
INORGANIC TESTS:			
Acidity	P, G	Cool, 4°C	14 days
Alkalinity	P, G	Cool, 4°C	14 days
Ammonia - Nitrogen	P, G	Cool, 4°C; H_2SO_4 to pH 2	28 days
Biochemical Oxygen Demand (BOD)	P, G	Cool, 4°C	48 hours
Bromide	P, G	None required	28 days
Chemical Oxygen Demand (COD)	P, G	Cool, 4°C; H_2SO_4 to pH 2	28 days
Chloride	P, G	None required	28 days
Chlorine, Total Residual	P, G	None required	Analyze immediately
Color	P, G	Cool, 4°C	48 hours
Cyanide, Total and Amenable to Chlorination	P, G	Cool, 4°C; NaOH to pH 12; 0.6 g ascorbic acid(5)	14 days(6)
Fluoride	P	None required	28 days
Hardness	P, G	HNO_3 to pH 2; H_2SO_4 to pH 2	6 months
Total Kjeldahl and Organic Nitrogen	P, G	Cool, 4°C; H_2SO_4 to pH 2	28 days
Nitrate - Nitrogen	P, G	None required	48 hours
Nitrate-Nitrite - Nitrogen	P, G	Cool, 4°C; H_2SO_4 to pH 2	28 days
Nitrite - Nitrogen	P, G	Cool, 4°C	48 hours
Oil & Grease	G	Cool, 4°C; H_2SO_4 to pH 2	28 days
Total Organic Carbon (TOC)	P, G	Cool, 4°C; HCl or H_2SO_4 to pH 2	28 days
Orthophosphate	P, G	Filter immediately; Cool, 4°C	48 hours
Oxygen, Dissolved-Probe	G Bottle & top	None required	Analyze immediately
Oxygen, Dissolved-Winkler	G Bottle & top	Fix on site and store in dark	8 hours
Phenols	G	Cool, 4°C; H_2SO_4 to pH 2	28 days
Phosphorus, Total	P, G	Cool, 4°C; H_2SO_4 to pH 2	28 days
Residue, Total	P, G	Cool, 4°C	7 days
Residue, Filterable (TDS)	P, G	Cool, 4°C	7 days
Residue, Nonfilterable (TSS)	P, G	Cool, 4°C	7 days
Residue, Settleable	P, G	Cool, 4°C	48 hours
Residue, Volatile (Ash Content)	P, G	Cool, 4°C	7 days
Silica	P	Cool, 4°C	28 days
Specific Conductance	P, G	Cool, 4°C	28 days
Sulfate	P, G	Cool, 4°C	28 days

ATTACHMENT B
ADDITIONAL REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES
PAGE TWO

Parameter Number/Name	Container(1)	Preservation(2)(3)	Maximum Holding Time(4)
INORGANIC TESTS (Cont'd):			
Sulfide	P, G	Cool, 4°C; add zinc acetate plus sodium hydroxide to pH 9	7 days
Sulfite	P, G	None required	Analyze immediately
Turbidity	P, G	Cool, 4°C	48 hours
METALS:(7)			
Chromium VI (Hexachrome)	P, G	Cool, 4°C	24 hours
Mercury (Hg)	P, G	HNO_3 to pH 2	28 days
Metals, except Chromium VI and Mercury	P, G	HNO_3 to pH 2	6 months
ORGANIC TESTS:(8)			
Purgeable Halocarbons	G, Teflon-lined septum	Cool, 4°C; 0.008% $Na_2S_2O_3$(5)	14 days
Purgeable Aromatic Hydrocarbons	G, Teflon-lined septum	Cool, 4°C; 0.008% $Na_2S_2O_3$(5) HCl to pH 2 (9)	14 days
Acrolein and Acrylonitrile	G, Teflon-lined septum	Cool, 4°C; 0.008% $Na_2S_2O_3$(5) adjust pH to 4-5 (10)	14 days
Phenols(11)	G, Teflon-lined cap	Cool, 4°C; 0.008% $Na_2S_2O_3$(5)	7 days until extraction; 40 days after extraction
Benzidines(11), (12)	G, Teflon-lined cap	Cool, 4°C; 0.008% $Na_2S_2O_3$(5)	7 days until extraction(13)
Phthalate esters(11)	G, Teflon-lined cap	Cool, 4°C	7 days until extraction; 40 days after extraction
Nitrosamines(11), (14)	G, Teflon-lined cap	Cool, 4°C; store in dark; 0.008% $Na_2S_2O_3$(5)	7 days until extraction; 40 days after extraction
PCBs(11)	G, Teflon-lined cap	Cool, 4°C	7 days until extraction; 40 days after extraction
Nitroaromatics & Isophorone(11)	G, Teflon-lined cap	Cool, 4°C; 0.008% $Na_2S_2O_3$(5); store in dark	7 days until extraction; 40 days after extraction
Polynuclear Aromatic Hydrocarbons (PAHs)(11),(14)	G, Teflon-lined cap	Cool, 4°C; 0.008% $Na_2S_2O_3$(5); store in dark	7 days until extraction; 40 days after extraction
Haloethers(11)	G, Teflon-lined cap	Cool, 4°C; 0.008% $Na_2S_2O_3$(5)	7 days until extraction; 40 days after extraction
Dioxin/Furan (TCDD/TCDF)(11)	G, Teflon-lined cap	Cool, 4°C; 0.008% $Na_2S_2O_3$(5)	7 days until extraction; 40 days after extraction

ATTACHMENT B
ADDITIONAL REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES
PAGE THREE

Parameter Number/Name	Container(1)	Preservation(2)(3)	Maximum Holding Time(4)
RADIOLOGICAL TESTS:			
1-5 Alpha, beta and radium	P, G	HNO_3 to pH 2	6 months

(1) Polyethylene (P): generally 500 ml or Glass (G): generally 1L.
(2) Sample preservation should be performed immediately upon sample collection. For composite chemical samples each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then chemical samples may be preserved by maintaining at 4°C until compositing and sample splitting is completed.
(3) When any sample is to be shipped by common carrier or sent through the United States Mail, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172).
(4) Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid. Samples may be held for longer periods only if the permittee, or monitoring laboratory, has data on file to show that the specific types of samples under study are stable for the longer periods, and has received a variance from the Regional Administrator.
(5) Should only be used in the presence of residual chlorine.
(6) Maximum holding time is 24 hours when sulfide is present. Optionally, all samples may be tested with lead acetate paper before pH adjustments are made to determine if sulfide is present. If sulfide is present, it can be removed by the addition of cadmium nitrate powder until a negative spot test is obtained. The sample is filtered and then NaOH is added to pH 12.
(7) Samples should be filtered immediately on site before adding preservative for dissolved metals.
(8) Guidance applies to samples to be analyzed by GC, LC, or GC/MS for specific compounds.
(9) Sample receiving no pH adjustment must be analyzed within 7 days of sampling.
(10) The pH adjustment is not required if acrolein will not be measured. Samples for acrolein receiving no pH adjustment must be analyzed within 3 days of sampling.
(11) When the extractable analytes of concern fall within a single chemical category, the specified preservative and maximum holding times should be observed for optimum safeguard of sample integrity. When the analytes of concern fall within two or more chemical categories, the sample may be preserved by cooling to 4°C, reducing residual chlorine with 0.008% sodium thiosulfate, storing in the dark, and adjusting the pH to 6-9; samples preserved in this manner may be held for 7 days before extraction and for 40 days after extraction. Exceptions to this optional preservation and holding time procedure are noted in footnote 5 (re: the requirement for thiosulfate reduction of residual chlorine) and footnotes 12, 13 (re: the analysis of benzidine).
(12) If 1,2-diphenylthydrazine is likely to be present, adjust the pH of the sample to 4.0±0.2 to prevent rearrangement to benzidine.
(13) Extracts may be stored up to 7 days before analysis if storage is conducted under an inert (oxidant-free) atmosphere.
(14) For the analysis of diphenylnitrosamine, add 0.008% $Na_2S_2O_3$ and adjust pH to 7-10 with NaOH within 24 hours of sampling.
(15) The pH adjustment may be performed upon receipt at the laboratory and may be omitted if the samples are extracted within 72 hours of collection. For the analysis of aldrin, add 0.008% $Na_2S_2O_3$.

ATTACHMENT C

DOT HAZARDOUS MATERIAL CLASSIFICATION (49 CFR 173.2a)

1. Radioactive material (except a limited quantity)
2. Division 2.3, Poisonous Gases
3. Division 2.1, Flammable Gas
4. Division 2.2, Nonflammable gas
5. Division 6.1, Poisonous Liquids, Packing Group 1 (poison by inhalation only)
6. Division 4.2, Pyrophoric Material
7. Division 4.1, Self-Reactive Material
8. Class 3, Flammable Liquids*
9. Class 8, Corrosive Material
10. Division 4.1, Flammable Solid*
11. Division 4.2, Spontaneously Combustible Materials*
12. Division 4.3, Dangerous When Wet Materials*
13. Division 5.1, Oxidizers*
14. Division 6.1, Poisonous Liquids or Solids (other than Packing Group 1)*
15. Combustible liquid
16. Class 9, Miscellaneous Hazardous Materials

* If a material has or meets the criteria for more than one hazard class, use the precedence of hazardous table on the following page for Classes 3 and 8 and Divisions 4.1, 4.2, 4.3, 5.1, and 6.1. The following table ranks those materials that meet the definition of Classes 3 and 8 and Divisions 4.1, 4.2, 4.3, 5.1, and 6.1.

ATTACHMENT C (Continued)

PRECEDENCE OF HAZARD TABLE

(Hazard Class and Packing Group)

Class	Packing Group	4.2	4.3	5.1 I[a]	5.1 II[a]	5.1 III[a]	6.1 I (Dermal)	6.1 I (Oral)	6.1 II	6.1 III	8 I (Liquid)	8 I (Solid)	8 II (Liquid)	8 II (Solid)	8 III (Liquid)	8 III (Solid)
3	I						3	3	3	3	3	[c]	3	[c]	3	[c]
3	II						3	3	3	3	8	[c]	3	[c]	3	[c]
3	III						6.1	6.1	6.1	3[d]	8	[c]	8	[c]	3	[c]
4.1	II[b]	4.2	4.3	5.1	4.1	4.1	6.1	6.1	4.1	4.1	[c]	8	[c]	4.1	[c]	4.1
4.1	III[b]	4.2	4.3	5.1	4.1	4.1	6.1	6.1	6.1	4.1	[c]	8	[c]	8	[c]	4.1
4.2	II		4.3	5.1	4.2	4.2	6.1	6.1	4.2	4.2	[c]	8	[c]	4.2	[c]	4.2
4.2	III		4.3	5.1	4.2	4.2	6.1	6.1	6.1	4.2	[c]	8	[c]	8	[c]	4.2
4.3	I			5.1	4.3	4.3	6.1	4.3	4.3	4.3	4.3	4.3	4.3	4.3	4.3	4.3
4.3	II			5.1	4.3	4.3	6.1	4.3	4.3	4.3	8	8	8	4.3	4.3	4.3
4.3	III			5.1	4.3	4.3	6.1	6.1	6.1	4.3	8	8	8	8	4.3	4.3
5.1	I[a]						5.1	5.1	5.1	5.1	5.1	5.1	5.1	5.1	5.1	5.1
5.1	II[a]						6.1	5.1	5.1	5.1	8	8	8	5.1	5.1	5.1
5.1	III[a]						6.1	6.1	6.1	5.1	8	8	8	8	5.1	5.1
6.1	I, Dermal										8	6.1	6.1	6.1	6.1	6.1
6.1	I, Oral										8	6.1	6.1	6.1	6.1	6.1
6.1	II, Inhalation										8	6.1	6.1	6.1	6.1	6.1
6.1	II, Dermal										8	6.1	8	6.1	6.1	6.1
6.1	II, Oral										8	8	8	6.1	6.1	6.1
6.1	III										8	8	8	8	8	8

(a) There are at present no established criteria for determining Packing Groups for liquids in Division 5.1. At present, the degree of hazard is to be assessed by analogy with listed substances, allocating the substances to Packing Group I, Great; Group II, Medium; or Group III, Minor Danger.
(b) Substances of Division 4.1 other than self-reactive substances.
(c) Denotes an impossible combination.
(d) For pesticides only, where a material has the hazards of Class 3, Packing Group III, and Division 6.1, Packing Group III, the primary hazard is Division 6.1, Packing Group III.

ATTACHMENT D

GUIDE FOR HAZARDOUS MATERIALS SHIPPERS

USE OF GUIDE - This guide is presented as an aid to shippers of hazardous materials. It does not contain or refer to all of the DOT requirements for shipping hazardous materials. For specific details, refer to all of the DOT requirements for shipping hazardous materials, as provided in the Code of Federal Regulations (CFR), Title 49, Transportation, Parts 100-199.

The following is offered as a step-by-step procedure to aid in compliance with the applicable DOT regulations.

STEP 1 - DETERMINE THE PROPER SHIPPING NAME. The shipper must determine the proper shipping name of the materials as listed in the Hazardous Materials Table, 49 CFR 172.101, Column (2).

STEP 2 - DETERMINE THE HAZARD CLASS OR CLASSES.

a. Refer to the Table, 49 CFR 172.101, Column (3), and locate the hazard class of the material.
b. If more than one class is shown for the proper shipping name, determine the proper class by definition.
c. If the materials have more than one hazard, classify the material based on the order of hazards in 49 CFR 173.2.

STEP 3 - SELECT THE PROPER IDENTIFICATION NUMBERS.

a. Refer to the Table, 49 CFR 172.101, Column (3a), and select the Identification Number (ID) that corresponds to the proper shipping name and hazard class.
b. Enter the ID number(s) on the shipping papers and display them, as required, on packagings, placards and/or orange panels.

STEP 4 - DETERMINE THE MODE(S) OF TRANSPORT TO ULTIMATE DESTINATION.

a. As a shipper, you must assure yourself that the shipment complies with various modal requirements.
b. The modal requirements may affect the following: (1) Packaging; (2) Quantity per Package; (3) Marking; (4) Labeling; (5) Shipping Papers; and (6) Certification.

STEP 5 - SELECT THE PROPER LABEL(S) AND APPLY AS REQUIRED.

a. Refer to the Table, 49 CFR 172.101, Column (4) for required labels.
b. For details on labeling refer to (1) Additional Labels, 49 CFR 172.402; (2) Placement of Labels, 49 CFR 172.406; (3) Packagings (Mixed or Consolidated), 49 CFR 172.404(a) and (h); (4) Packages Containing Samples, 49 CFR 172.402(h); (5) Radioactive Materials, 49 CFR 172.403; and (6) Authorized Label Modifications, 49 CFR 172.405.

STEP 6 - DETERMINE AND SELECT THE PROPER PACKAGES.

a. Refer to the Table, 49 CFR 172.101, Column (5a) for exceptions and Column (5b) for specification packagings. Consider the following when selecting an authorized package: Quantity per Package; Cushioning Material, if required; Proper Closure and Reinforcement; Proper Pressure; Outage; etc., as required.
b. If packaged by a prior shipper, make sure the packaging is correct and in proper condition for transportation.

ATTACHMENT D (Continued)
GUIDE FOR HAZARDOUS MATERIALS SHIPPERS

STEP 7 - MARK THE PACKAGING (INCLUDING OVERPACKS).

a. Apply the required markings (49 CFR 172.300); Proper shipping name and ID number, when required (49 CFR 172.301); Name and address of Consignee or Consignor (49 CFR 172.306).
b. For details and other required markings, see 49 CFR 172.300 through 172.338.

STEP 8 - PREPARE THE SHIPPING PAPERS.

a. The basic requirements for preparing shipping papers include Proper Shipping Name; Hazard Class; ID Number; Total Quantity; Shipper's Certification; and Emergency Response Telephone Number.
b. Make all entries on the shipping papers using the information required and in proper sequence (49 CFR 172.202).

STEP 9 - CERTIFICATION.

a. Each shipper must certify by printing (manually or mechanically) on the shipping papers that the materials being offered for shipment are properly classified, described, packaged, marked and labeled, and in proper condition for transportation according to the applicable DOT Regulations (49 CFR 172.202).

STEP 10 - LOADING, BLOCKING, AND BRACING. When hazardous materials are loaded into the transport vehicle or freight container, each package must be loaded, blocked, and braced in accordance with the requirements for mode of transport.

a. If the shipper loads the freight container or transport vehicle, the shipper is responsible for the proper loading, blocking, and bracing of the materials.
b. If the carrier does the loading, the carrier is responsible.

STEP 11 - DETERMINE THE PROPER PLACARD(S). Each person who offers hazardous materials for transportation must determine that the placarding requirements have been met.

a. For Highway, unless the vehicle is already correctly placarded, the shipper must provide the required placard(s) and required ID number(s) (49 CFR 172.506).
b. For Rail, if loaded by the shipper, the shipper must placard the rail car if placards are required (49 CFR 172.508).
c. For Air and Water shipments, the shipper has the responsibility to apply the proper placards.

STEP 12 - HAZARDOUS WASTE/HAZARDOUS SUBSTANCE.

a. If the material is classed as a hazardous waste or hazardous substance, most of the above steps will be applicable.
b. Pertinent Environmental Protection Agency regulations are found in the Code of Federal Regulations, Title 40, Part 262.

As a final check and before offering the shipment for transportation, visually inspect your shipment. The shipper should ensure that emergency response information is on the vehicle for transportation of hazardous materials.

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Revised March 1995.

ATTACHMENT E

HAZARDOUS MATERIALS SHIPPING CHECK LIST

PACKAGING

1. Check DOT 173.24 for appropriate type of package for hazardous substance.
2. Check for container integrity, especially the closure.
3. Check for sufficient absorbent material in package.
4. Check for sample tags and log sheets for each sample and for chain-of-custody record.

SHIPPING PAPERS

1. Check that entries contain only approved DOT abbreviations.
2. Check that entries are in English.
3. Check that hazardous material entries are specially marked to differentiate them from any nonhazardous materials being sent using same shipping paper.
4. Be careful that all hazardous classes are shown for multiclass materials.
5. Check total amounts by weight, quantity, or other measures used.
6. Check that any limited-quantity exemptions are so designated on the shipping paper.
7. Check that certification is signed by shipper.
8. Make certain driver signs for shipment.

RCRA MANIFEST

1. Check that approved state/federal manifests are prepared.
2. Check that transporter has the following: valid EPA identification number, valid driver's license, valid vehicle registration, insurance protection, and proper DOT labels for materials being shipped.
3. Check that destination address is correct.
4. Check that driver knows where shipment is going.
5. Check that the driver is aware of emergency procedures for spills and accidents.
6. Make certain driver signs for shipment.
7. Make certain one copy of executed manifest and shipping document is retained by shipper.

ATTACHMENT F

DOT SEGREGATION AND SEPARATION CHART

Class or Division	Notes	1.1-1.2	1.3	1.4	1.5	1.6	2.1	2.2	2.3 gas Zone A*	2.3 gas Zone B*	3	4.1	4.2	4.3	5.1	5.2	6.1 liquids PG-I Zone A*	7	8 liquids only
Explosives . . . . . 1.1 and 1.2	A	*	*	*	*	*	X	X	X	X	X	X	X	X	X	X	X	X	X
Explosives 1.3		*	*	*	*	*	X		X	X	X		X	X	X	X	X		X
Explosives 1.4		*	*	*	*	*	O		O	O	O		O				O		O
Very insensitive explosives 1.5	A	*	*	*	*	*	X	X	X	X	X	X	X	X	X	X	X	X	X
Extremely insensitive explosives 1.6		*	*	*	*	*													
Flammable gases 2.1		X	X	O	X				X	O							O	O	
Non-toxic, non-flammable gases 2.2		X			X														
Poisonous gas - Zone A** 2.3		X	X	O	X		X				X	X	X	X	X	X			X
Poisonous gas - Zone B** 2.3		X	X	O	X		O				O	O	O	O	O	O			O
Flammable liquids 3		X	X	O	X				X	O					O		X		
Flammable solids 4.1		X			X				X	O							X		O
Spontaneously combustible materials 4.2		X	X	O	X				X	O							X		X
Dangerous-when-wet materials 4.3		X	X		X				X	O							X		O
Oxidizers 5.1	A	X	X		X				X	O	O						X		O
Organic peroxides 5.2		X	X		X				X	O							X		O
Poisonous liquids PG I - Zone A** . . 6.1		X	X	O	X		O				X	X	X	X	X	X			X
Radioactive materials 7		X			X		O												
Corrosive liquids 8		X	X	O	X				X	O		O	X	O	O	O	X		

No entry means that the materials are compatible (have no restrictions).

X These materials may not be loaded, transported, or stored together in the same vehicle or facility.
O The materials may not be loaded, transported, or stored together in the same vehicle or facility unless they are separated for 4 feet on all sides.
* Check the explosives compatibility chart in 49 CFR 179.848(f).
A Ammonium nitrate fertilizers may be stored with Division 1.1 materials.
** Denotes inhalation hazardous for poisons; consult field team leader or project manager if you encounter a material in this class before shipment.

ATTACHMENT G
LITHIUM BATTERY SHIPPING PAPERS

3224637861

TRANSPORT DETAILS

This shipment is within the limitations prescribed for: *(delete non applicable)*

[redacted] | CARGO AIRCRAFT ONLY

Airport of Departure

Airport of Destination: 19CYS

Two completed and signed copies of this Declaration must be handed to the operator.

WARNING

Failure to comply in all respects with the applicable Dangerous Goods Regulations may be in breach of the applicable law, subject to legal penalties. This Declaration must not, in any circumstances, be completed and/or signed by a consolidator, a forwarder or an IATA cargo agent.

Shipment type: *(delete non-applicable)*

NON-RADIOACTIVE | [redacted]

NATURE AND QUANTITY OF DANGEROUS GOODS

Dangerous Goods Identification						
Proper Shipping Name	Class or Division	UN or ID No.	Subsidiary Risk	Quantity and type of packing	Packing Inst.	Authorization
LITHIUM BATTERIES CONTAINED IN EQUIPMENT	9	UN3091		1 PLASTIC BOX X 55 GRAMS	912 II	PER CA-9206009

Additional Handling Information

1 HERMIT SERIES DATALOGGER X 55 GRAMS (11 GRAMS/CELL)

I hereby declare that the contents of this consignment are fully and accurately described above by proper shipping name and are classified, packed, marked, and labeled, and are in all respects in the proper condition for transport by air according to the applicable International and National Government Regulations.

Name/Title of Signatory

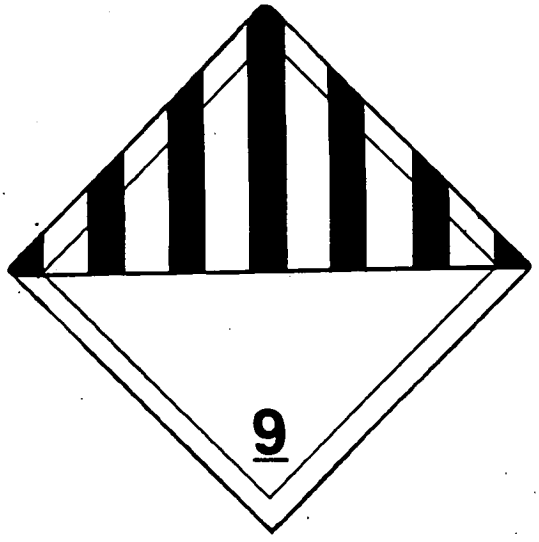
Place and Date

Signature *(see warning above)*

Emergency Telephone Number (Required for US Origin or Destination Shipments)
800-535-5053

IF ACCEPTABLE FOR PASSENGER AIRCRAFT, THIS SHIPMENT CONTAINS RADIOACTIVE MATERIAL INTENDED FOR USE IN, OR INCIDENT TO, RESEARCH, MEDICAL DIAGNOSIS, OR TREATMENT.

ATTACHMENT G (CONTINUED)
LITHIUM BATTERY SHIPPING PAPERS



LITHIUM BATTERIES CONTAINED IN EQUIPMENT.
UN-3091.
SHIPPED UNDER CA-9206009

TABLE OF CONTENTS

SECTION		PAGE
1.0	PURPOSE	3
2.0	SCOPE	3
3.0	GLOSSARY	3
4.0	RESPONSIBILITIES	3
5.0	PROCEDURES	3
5.1	Site Logbook	3
5.1.1	General	3
5.1.2	Photographs	4
5.2	Site Notebooks	4
5.3	Sample Forms	5
5.3.1	Sample Collection, Labeling, Shipment and Request for Analysis	5
5.3.2	Geohydrological and Geotechnical Forms	6
5.3.3	Equipment Calibration and Maintenance Form	6
5.4	Field Reports	7
5.4.1	Weekly Status Reports	7
5.4.2	Daily Activities Report	7
6.0	ATTACHMENTS	8

TABLE OF CONTENTS (Continued)

ATTACHMENTS (EXAMPLES) **PAGE**

A	TYPICAL SITE LOGBOOK ENTRY	9
B-1	EXAMPLE GROUNDWATER SAMPLE LOG SHEET	10
B-2	EXAMPLE SURFACE WATER SAMPLE LOG SHEET	11
B-3	EXAMPLE SOIL/SEDIMENT SAMPLE LOG SHEET	12
B-4	CONTAINER SAMPLE LOG SHEET FORM	13
B-5	SAMPLE LABEL	14
B-6	CHAIN-OF-CUSTODY RECORD FORM	15
B-7	CHAIN-OF-CUSTODY SEAL	16
C-1	EXAMPLE GROUNDWATER LEVEL MEASUREMENT SHEET	17
C-2	EXAMPLE PUMPING TEST DATA SHEET	18
C-3	PACKER TEST REPORT FORM	19
C-4	EXAMPLE BORING LOG	20
C-5	EXAMPLE OVERBURDEN MONITORING WELL SHEET	22
C-5A	EXAMPLE OVERBURDEN MONITORING WELL SHEET (FLUSHMOUNT)	23
C-6	EXAMPLE CONFINING LAYER MONITORING WELL SHEET	24
C-7	EXAMPLE BEDROCK MONITORING WELL SHEET - OPEN HOLE WELL	25
C-8	EXAMPLE BEDROCK MONITORING WELL SHEET, WELL INSTALLED IN BEDROCK	26
C-8A	EXAMPLE BEDROCK MONITORING WELL SHEET, WELL INSTALLED IN BEDROCK (FLUSHMOUNT)	27
C-9	EXAMPLE TEST PIT LOG	28
D	EXAMPLE EQUIPMENT CALIBRATION LOG	29
E	EXAMPLE DAILY ACTIVITIES RECORD	30
F	FIELD TRIP SUMMARY REPORT	31

1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to identify and designate the field data record forms, logs and reports generally initiated and maintained for documenting Brown & Root Environmental field activities.

2.0 SCOPE

Documents presented within this procedure (or equivalents) shall be used for all Brown & Root Environmental field activities, as applicable. Other or additional documents may be required by specific client contracts.

3.0 GLOSSARY

None

4.0 RESPONSIBILITIES

Project Manager - The Project Manager is responsible for obtaining hardbound, controlled-distribution logbooks (from the appropriate source), as needed. In addition, the Project Manager is responsible for placing all forms used in site activities (i.e., records, field reports, and upon the completion of field work, the site logbook) in the project's central file.

Field Operations Leader (FOL) - The Field Operations Leader is responsible for ensuring that the site logbook, notebooks, and all appropriate forms and field reports illustrated in this guideline (and any additional forms required by the contract) are correctly used, accurately filled out, and completed in the required time-frame.

5.0 PROCEDURES

5.1 Site Logbook

5.1.1 General

The site logbook is a hard-bound, paginated controlled-distribution record book in which all major onsite activities are documented. At a minimum, the following activities/events shall be recorded (daily) in the site logbook:

- All field personnel present
- Arrival/departure of site visitors
- Arrival/departure of equipment
- Start or completion of borehole/trench/monitoring well installation or sampling activities
- Daily onsite activities performed each day
- Sample pickup information
- Health and Safety issues (level of protection observed, etc.)
- Weather conditions

A site logbook shall be maintained for each project. The site logbook shall be initiated at the start of the first onsite activity (e.g., site visit or initial reconnaissance survey). Entries are to be made for every day that onsite activities take place which involve Brown & Root Environmental or subcontractor personnel. Upon completion of the fieldwork, the site logbook must become part of the project's central file.

The following information must be recorded on the cover of each site logbook:

- Project name
- Brown & Root Environmental project number
- Sequential book number
- Start date
- End date

Information recorded daily in the site logbook need not be duplicated in other field notebooks (see Section 5.2), but must summarize the contents of these other notebooks and refer to specific page locations in these notebooks for detailed information (where applicable).. An example of a typical site logbook entry is shown in Attachment A.

If measurements are made at any location, the measurements and equipment used must either be recorded in the site logbook or reference must be made to the site notebook in which the measurements are recorded (see Attachment A).

All logbook, notebook, and log sheet entries shall be made in indelible ink (black pen is preferred). No erasures are permitted. If an incorrect entry is made, the data shall be crossed out with a single strike mark, and initialed and dated. At the completion of entries by any individual, the logbook pages used must be signed and dated. The site logbook must also be signed by the Field Operations Leader at the end of each day.

5.1.2 Photographs

When movies, slides, or photographs are taken of a site or any monitoring location, they must be numbered sequentially to correspond to logbook entries. The name of the photographer, date, time, site location, site description, and weather conditions must be entered in the logbook as the photographs are taken. A series entry may be used for rapid-sequence photographs. The photographer is not required to record the aperture settings and shutter speeds for photographs taken within the normal automatic exposure range. However, special lenses, films, filters, and other image-enhancement techniques must be noted in the logbook. If possible, such techniques shall be avoided, since they can adversely affect the admissibility of photographs as evidence. Chain-of-custody procedures depend upon the subject matter, type of film, and the processing it requires. Film used for aerial photography, confidential information, or criminal investigation require chain-of-custody procedures. Adequate logbook notation and receipts must be compiled to account for routine film processing. Once processed, the slides of photographic prints shall be consecutively numbered and labeled according to the logbook descriptions. The site photographs and associated negatives must be docketed into the project's central file.

5.2 Site Notebooks

Key field team personnel may maintain a separate dedicated notebook to document the pertinent field activities conducted directly under their supervision. For example, on large projects with multiple investigative sites and varying operating conditions, the Health and Safety Officer may elect to maintain a separate site notebook. Where several drill rigs are in operation simultaneously, each site geologist assigned to oversee a rig must maintain a site notebook.

5.3 Sample Forms

A summary of the forms illustrated in this procedure is shown as the listing of Attachments in the Table of Contents for this SOP. Forms may be altered or revised for project-specific needs contingent upon client approval. Care must be taken to ensure that all essential information can be documented. Guidelines for completing these forms can be found in the related sampling SOP.

5.3.1 Sample Collection, Labeling, Shipment and Request for Analysis

5.3.1.1 Sample Log Sheet

Sample Log Sheets are used to record specified types of data while sampling. Attachments B-1 to B-4 are examples of Sample Log Sheets. The data recorded on these sheets are useful in describing the waste source and sample as well as pointing out any problems encountered during sampling. A log sheet must be completed for each sample obtained, including field quality control (QC) samples.

5.3.1.2 Sample Label

A typical sample label is illustrated in Attachment B-5. Adhesive labels must be completed and applied to every sample container. Sample labels can usually be obtained from the appropriate Program source or are supplied from the laboratory subcontractor.

5.3.1.3 Chain-of-Custody Record Form

The Chain-of-Custody (COC) Record is a multi-part form that is initiated as samples are acquired and accompanies a sample (or group of samples) as they are transferred from person to person. This form must be used for any samples collected for chemical or geotechnical analysis whether the analyses are performed on site or off site. One part of the completed form is retained by the field crew while the other two portions are sent to the laboratory. An example of a Chain-of-Custody Record form is provided as Attachment B-6. A supply of these forms are purchased and stocked by the field department of the various Brown & Root Environmental offices. Alternately, COC forms supplied by the laboratory may be used. Once the samples are received at the laboratory, the sample cooler and contents are checked and any problems are noted on the enclosed COC form (any discrepancies between the sample labels and COC form and any other problems that are noted are resolved through communication between the laboratory point-of-contact and the Brown & Root Environmental Project Manager). The COC form is signed and one of the remaining two parts are retained by the laboratory while the last part becomes part of the samples' corresponding analytical data package. Internal laboratory chain-of-custody procedures are documented in the Laboratory Quality Assurance Plan (LQAP).

5.3.1.4 Chain-of-Custody Seal

Attachment B-7 is an example of a custody seal. The Custody seal is also an adhesive-backed label. It is part of a chain-of-custody process and is used to prevent tampering with samples after they have been collected in the field and sealed in coolers for transit to the laboratory. The COC seals are signed and dated by the samplers and affixed across the opening edges of each cooler containing environmental samples. COC seals may be available from the laboratory; these seals may also be purchased from a supplier.

5.3.2 Geohydrological and Geotechnical Forms

5.3.2.1 Groundwater Level Measurement Sheet

A groundwater level measurement sheet, shown in Attachment C-1 must be filled out for each round of water level measurements made at a site.

5.3.2.2 Data Sheet for Pumping Test

During the performance of a pumping test (or an in-situ hydraulic conductivity test), a large amount of data must be recorded, often within a short time period. The pumping test data sheet (Attachment C-2) facilitates this task by standardizing the data collection format, and allowing the time interval for collection to be laid out in advance.

5.3.2.3 Packer Test Report Form

A packer test report form shown in Attachment C-3 must be completed for each well upon which a packer test is conducted following well installation.

5.3.2.4 Summary Log of Boring

During the progress of each boring, a log of the materials encountered, operation and driving of casing, and location of samples must be kept. The Summary Log of Boring (Attachment C-4) is used for this purpose and must be completed for each soil boring performed. In addition, if volatile organics are monitored on cores, samples or cuttings from the borehole (using HNU or OVA detectors), these results must be entered on the boring log (under the "Remarks" column) at the appropriate depth. The "Remarks" column can also be used to subsequently enter the laboratory sample number and the concentration of a few key analytical results. This feature allows direct comparison of contaminant concentrations with soil characteristics.

5.3.2.5 Monitoring Well Construction Details Form

A Monitoring Well Construction Details Form must be completed for every monitoring well piezometer or temporary well point installed. This form contains specific information on length and type of well riser pipe and screen, backfill, filter pack, annular seal and grout characteristics, and surface seal characteristics. This information is important in evaluating the performance of the monitoring well, particularly in areas where water levels show temporal variation, or where there are multiple (immiscible) phases of contaminants. Depending on the type of monitoring well (in overburden or bedrock), different forms are used (see Attachments C-5 through C-9). Similar forms are used for flush-mount well completions. The Monitoring Well Construction Details Form is not a controlled document.

5.3.2.6 Test Pit Log

When a test pit or trench is constructed for investigative or sampling purposes, a Test Pit Log (Attachment C-10) must be filled out by the responsible field geologist or sampling technician.

5.3.3 Equipment Calibration and Maintenance Form

The calibration or standardization of monitoring, measuring or test equipment is necessary to assure the proper operation and response of the equipment, to document the accuracy, precision or sensitivity of the measurement, and determine if correction should be applied to the readings. Some items of

equipment require frequent calibration, others infrequent. Some are calibrated by the manufacturer, others by the user.

Each instrument requiring calibration has its own Equipment Calibration Log (Attachment D) which documents that the manufacturer's instructions were followed for calibration of the equipment, including frequency and type of standard or calibration device. An Equipment Calibration Log must be maintained for each electronic measuring device used in the field; entries must be made for each day the equipment is used.

5.4 Field Reports

The primary means of recording onsite activities is the site logbook. Other field notebooks may also be maintained. These logbooks and notebooks (and supporting forms) contain detailed information required for data interpretation or documentation, but are not easily useful for tracking and reporting of progress. Furthermore, the field logbook/notebooks remain onsite for extended periods of time and are thus not accessible for timely review by project management.

5.4.1 Weekly Status Reports

To facilitate timely review by project management, Xeroxed copies of logbook/notebook entries may be made for internal use. To provide timely oversight of onsite contractors, Daily Activities Reports are completed and submitted as described below.

It should be noted that in addition to the summaries described herein, other summary reports may also be contractually required.

5.4.2 Daily Activities Report

5.4.2.1 Description

The Daily Activities Report (DAR) documents the activities and progress for each day's field work. This report must be filled out on a daily basis whenever there are drilling, test pitting, well construction, or other related activities occurring which involve subcontractor personnel. These sheets summarize the work performed and form the basis of payment to subcontractors (Attachment E is an example of a Daily Activities Report).

5.4.2.2 Responsibilities

It is the responsibility of the rig geologist to complete the DAR and obtain the driller's signature acknowledging that the times and quantities of material entered are correct.

5.4.2.3 Submittal and Approval

At the end of the shift, the rig geologist must submit the Daily Activities Report to the Field Operations Leader (FOL) for review and filing. The Daily Activities Report is not a formal report and thus requires no further approval. The DAR reports are retained by the FOL for use in preparing the site logbook and in preparing weekly status reports for submission to the Project Manager.

6.0 ATTACHMENTS

Attachment A	TYPICAL SITE LOGBOOK ENTRY
Attachment B-1	EXAMPLE GROUNDWATER SAMPLE LOG SHEET
Attachment B-2	EXAMPLE SURFACE WATER SAMPLE LOG SHEET
Attachment B-3	EXAMPLE SOIL/SEDIMENT SAMPLE LOG SHEET
Attachment B-4	CONTAINER SAMPLE LOG SHEET FORM
Attachment B-5	SAMPLE LABEL
Attachment B-6	CHAIN-OF-CUSTODY RECORD FORM
Attachment B-7	CHAIN-OF-CUSTODY SEAL
Attachment C-1	EXAMPLE GROUNDWATER LEVEL MEASUREMENT SHEET
Attachment C-2	EXAMPLE PUMPING TEST DATA SHEET
Attachment C-3	PACKER TEST REPORT FORM
Attachment C-4	EXAMPLE BORING LOG
Attachment C-5	EXAMPLE OVERBURDEN MONITORING WELL SHEET
Attachment C-5A	EXAMPLE OVERBURDEN MONITORING WELL SHEET (FLUSHMOUNT)
Attachment C-6	EXAMPLE CONFINING LAYER MONITORING WELL SHEET
Attachment C-7	EXAMPLE BEDROCK MONITORING WELL SHEET - OPEN HOLE WELL
Attachment C-8	EXAMPLE BEDROCK MONITORING WELL SHEET - WELL INSTALLED IN BEDROCK
Attachment C-8A	EXAMPLE BEDROCK MONITORING WELL SHEET - WELL INSTALLED IN BEDROCK (FLUSHMOUNT)
Attachment C-9	EXAMPLE TEST PIT LOG
Attachment D	EXAMPLE EQUIPMENT CALIBRATION LOG
Attachment E	EXAMPLE DAILY ACTIVITIES RECORD
Attachment F	FIELD TRIP SUMMARY REPORT

ATTACHMENT A
TYPICAL SITE LOGBOOK ENTRY

START TIME: _____ DATE: _____

SITE LEADER: _____

PERSONNEL:

BROWN & ROOT ENV.	DRILLER	EPA
_____	_____	_____
_____	_____	_____
_____	_____	_____

WEATHER: Clear, 68°F, 2-5 mph wind from SE

ACTIVITIES:

1. Steam jenney and fire hoses were set up.
2. Drilling activities at well ____ resumes. Rig geologist was _____. See Geologist's Notebook, No. 1, page 29-30, for details of drilling activity. Sample No. 123-21-S4 collected; see sample logbook, page 42. Drilling activities completed at 11:50 and a 4-inch stainless steel well installed. See Geologist's Notebook, No. 1, page 31, and well construction details for well _____.
3. Drilling rig No. 2 steam-cleaned at decontamination pit. Then set up at location of well _____.
4. Well _____ drilled. Rig geologist was _____. See Geologist's Notebook, No. 2, page ____ for details of drilling activities. Sample numbers 123-22-S1, 123-22-S2, and 123-22-S3 collected; see sample logbook, pages 43, 44, and 45.
5. Well _____ was developed. Seven 55-gallon drums were filled in the flushing stage. The well was then pumped using the pitcher pump for 1 hour. At the end of the hour, water pumped from well was "sand free."
6. EPA remedial project manger arrives on site at 14:25 hours.
7. Large dump truck arrives at 14:45 and is steam-cleaned. Backhoe and dump truck set up over test pit _____.
8. Test pit _____ dug with cuttings placed in dump truck. Rig geologist was _____. See Geologist's Notebook, No. 1, page 32, for details of test pit activities. Test pit subsequently filled. No samples taken for chemical analysis. Due to shallow groundwater table, filling in of test pit ___ resulted in a very soft and wet area. A mound was developed and the area roped off.
9. Express carrier picked up samples (see Sample Logbook, pages 42 through 45) at 17:50 hours. Site activities terminated at 18:22 hours. All personnel off site, gate locked.

_____
Field Operations Leader

ATTACHMENT B-1
EXAMPLE GROUNDWATER SAMPLE LOG SHEET

GROUNDWATER
SAMPLE LOG SHEET

Page ____ of ____

Project Site Name: _____

Project No.: _____

☐ Domestic Well Data
☐ Monitoring Well Data
☐ Other Well Type: _____
☐ QA Sample Type: _____

Sample ID No.: _____

Sample Location: _____

Sampled By: _____

C.O.C. No.: _____

Sampling Data

Date: / Time: / Method:	pH	S.C.	Temp. (°C)	Turbidity	Color	TBD	TBD	TBD

Purge Data

Date: / Method:	Volume	pH	S.C.	Temp.(°C)	Turbidity	Color	TBD	TBD
	Initial							
Monitor Reading (ppm):	1							
Well Casing Dia. & Material Type:	2							
	3							
Total Well Depth (TD):	4							
Static Water Level (WL):	5							
TD-WL (ft.) =								
One Casing Volume: (gal/L)								
Start Purge (hrs.):								
End Purge (hrs.):								
Total Purge Time (min):								
Total Amount Purged (gal/L):								

Analysis	Preservative	Container Requirements	Collected (✓)

Observations/Notes:

Circle if Applicable:		Signature(s):
MS/MSD	Duplicate ID No:	

TBD: To Be Determined

ATTACHMENT B-2
EXAMPLE SURFACE WATER SAMPLING LOG SHEET

SURFACE WATER SAMPLING LOG SHEET

Page ____ of ____

Project Site Name: _____ Sample ID No.: _____

Project No.: _____ Sample Location: _____

☐ Spring ☐ Pond Sampled By: _____

☐ Stream ☐ Lake

☐ Other _____

☐ QA Sample Type: _____ C.O.C. No.: _____

Sample Date		
Date and Time	Method	Depth

pH	S.C.	Temp. (°C)	Turbidity	Color	TBD	TBD	TBD

Analysis	Preservative	Container Requirements	Collected (✓)

Observations/Notes:

Circle if Applicable:		Signature(s):
MS/MSD	Duplicate ID No.:	

TBD: To Be Determined

ATTACHMENT B-3
EXAMPLE SOIL/SEDIMENT SINGLE SAMPLE LOG SHEET

SOIL/SEDIMENT
SINGLE SAMPLE LOG SHEET

Page ___ of ___

Project Site Name: _____

Project No.: _____

- ☐ Surface Soil
- ☐ Subsurface Soil
- ☐ Sediment
- ☐ Other _____
- ☐ QA Sample Type: _____

Sample ID No.: _____

Sample Location: _____

Sampled By: _____

C.O.C. No.: _____

Sample Method:	Composite Sample Data		
	Sample	Time	Color/Description
Depth Sampled:			
Sample Date and Time:			
Type of Sample			
☐ Grab			
☐ Composite			
☐ Grab-Composite	Grab Sample Data		
☐ High Concentration	Color	Description: (Sand, Clay, Dry, Moist, Wet, etc.)	
☐ Low Concentration			

Analysis	Container Requirements	Collected (✓)	Map:

Observations/Notes:

Circle if Applicable:		Signature(s):
MS/MSD	Duplicate ID No:	

ATTACHMENT B-4
CONTAINER SAMPLE LOG SHEET FORM

Brown & Root Environmental

Page ___ of _

☐ Container Data

Case #: _____

By: _____

Project Site Name: _____ Project Site No. _____

Brown & Root Env. Source No. _____ Source Location: _____

Container Source	Container Description
☐ Drum ☐ Bung Top ☐ Lever Lock ☐ Bolted Ring ☐ Other _____ ☐ Bag/Sack ☐ Tank ☐ Other _____	Color: _____ Condition: _____ Markings: _____ Vol. of Contents: _____ Other: _____

Disposition of Sample	Sample Description			
☐ Container Sampled ☐ Container opened but not sampled. Reason: _____ ☐ Container not opened. Reason: _____		Layer 1	Layer 2	Layer 3
	Phase	☐Sol. ☐Liq.	☐Sol. ☐Liq.	☐Sol. ☐Liq.
	Color	_____	_____	_____
	Viscosity	☐L ☐M ☐H	☐L ☐M ☐H	☐L ☐M ☐H
	% of Total Volume	_____	_____	_____
	Other	_____	_____	_____

Monitor Reading:	Type of Sample ☐ Grab ☐ Low Concentration ☐ Composite ☐ High Concentration ☐ Grab-composite
Sample Method:	

Sample Date & Time:	Sample Identification	Organic	Inorganic
Sampled by:			
Signature(s):			
	Date Shipped		
Analysis:	Time Shipped		
	Lab		
	Volume		

ATTACHMENT B-5

SAMPLE LABEL

Brown & Root Environmental PROJECT: _____

STATION LOCATION: _____

DATE: ____/____/____ TIME: _____ hrs.

MEDIA: WATER ☐ SOIL ☐ SEDIMENT ☐ _____ ☐

CONCENTRATION: LOW ☐ MEDIUM ☐ HIGH ☐

TYPE: GRAB ☐ COMPOSITE ☐

ANALYSIS		PRESERVATION	
VOA ☐	BNAs ☐	Cool to 4°C	☐
PCBs ☐	PESTICIDES ☐	HNO_3 to pH < 2	☐
METALS: TOTAL ☐	DISSOLVED ☐	NaOH to pH > 12	☐
CYANIDE ☐			
_____ ☐		_____	☐

Sampled by: _____

Remarks:

ATTACHMENT B-6

CHAIN-OF-CUSTODY RECORD FORM
(Original is 8.5 x 11")

PROJECT NO.	PROJECT NAME	NO. OF CONTAINERS	REMARKS
SAMPLERS: *(Signature)*			

STA NO.	DATE	TIME			STATION LOCATION								

Relinquished by: (Signature)	Date/Time	Received by: (Signature)	Relinquished by: (Signature)	Date/Time	Received by: (Signature)
Relinquished by: (Signature)	Date/Time	Received by: (Signature)	Relinquished by: (Signature)	Date/Time	Received by: (Signature)
Relinquished by: (Signature)	Date/Time	Received for Laboratory by: (Signature)	Date/Time	Remarks:	

ATTACHMENT B-7

CHAIN-OF-CUSTODY SEAL

CUSTODY SEAL	**CUSTODY SEAL**
Date	**Date**
Signature	**Signature**

ATTACHMENT C-1
EXAMPLE GROUNDWATER LEVEL MEASUREMENT SHEET

GROUNDWATER LEVEL MEASUREMENT SHEET

Page ___ of ___

PROJECT NAME: _____ LOCATION: _____
PROJECT NUMBER: _____ MEASURING DEVICE: _____
PERSONNEL: _____ ADJUSTMENT FACTOR: _____
DATE: _____ REMARKS: _____
WEATHER CONDITIONS: _____

Well or Piezometer Number	Time	Elevation of Reference Point (Feet)*	Water Level Indicator Reading (Feet)*	Groundwater Elevation (Feet)*	Comments

*Measurements to nearest 0.01 foot.

Signature(s): _____

ATTACHMENT C-2
EXAMPLE PUMPING TEST DATA SHEET

PUMPING TEST DATA SHEET

Page ___ of ___

PROJECT NAME: _____
PROJECT NUMBER: _____
PUMPING TEST: [ ]
TEST NUMBER: _____
METHOD OF MEASUREMENT: _____
DATE(s): _____
STATIC H_2O LEVEL (ft) (SO) _____

PUMPING WELL NUMBER: _____
MEASURED WELL NUMBER: _____
STEP DRAW DOWN TEST [ ]
MONITORING POINT: _____
DEPTH CORRECTION (ft) _____
PUMP SETTING (Ft. below monitoring point): _____
DISTANCE FROM PUMPING WELL (ft) (r): _____

PUMPING TEST PERFORMED BY: _____
REMARKS: _____

MILITARY TIME	ELAPSED TIME SINCE PUMP START OR STOP (Min.)	WATER LEVEL (Ft.)	CORRECTION (Ft.)	DRAW DOWN OR RECOVERY (Ft.)	FLOW METER READING (Gals.)	PUMPING RATE (GPM)	REMARKS

SIGNATURE(s): _____

ATTACHMENT C-3

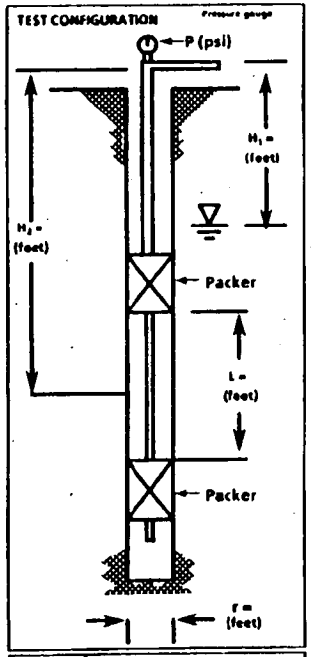
PACKER TEST REPORT FORM

PROJECT: _____ PROJECT NO.: _____ TEST NO: _____ PAGE ____ OF ____

BORING NO.: _____ CASING DEPTH: _____ CONTRACTOR: _____ STATIC WATER LEVEL _____

TEST INTERVAL: _____ BY: _____ CHECKED: _____ PACKER PRESSURE _____

Flow Test					Calculated Results							
Pressure, P (psi)	Elapsed Time (min)	Flow Reading	Δ Flow, ΔQ (ft^3)	Δ Flow, ΔQ (gal)	Flow Rate Q (gal/min)	H_1* (ft)	Hp(ft) = 2.31·(psi)	H_2* (ft)	H = Hp + (H1 or H2)	Cp	K (ft/yr) = Cp Q/H	K (cm/sec) = 9.67 x 10^{-1} K (ft/yr)



Length of Test Section in feet, L	Cp Drilling Bit Size EX (1.5")	AX (1.875")	BX (2.31")	NX (3")
1	31,000	28,500	25,800	23,300
2	19,400	18,100	16,800	15,500
5	9,800	9,300	8,800	8,300
8	6,800	6,500	6,100	5,800
10	5,700	5,400	5,400	4,900
15	4,100	3,900	3,700	3,600
20	3,200	3,100	3,000	2,800

CP = (1/(2 x L)) ln(L/r) (70,315 5)
7.48 Gallons = 1 Ft^3
1 psi = 2.31 ft head

* H_1 is used when the test length is below the water table.
H_2 is used when the test length is above the water table.

Remarks: _____

ATTACHMENT C-4
EXAMPLE BORING LOG

BORING LOG

Page___ of___

PROJECT NAME: _____ BORING NUMBER: _____
PROJECT NUMBER: _____ DATE : _____
DRILLING COMPANY: _____ GEOLOGIST: _____
WATER LEVEL DATA: _____

Sample No. and Type or RQD	Depth (Ft.) or Run No.	Blows/ 6" or RQD (%)	Sample Recovery/ Sample Length	Lithology Change (Depth/Ft.) or Screened Interval	MATERIAL DESCRIPTION: Soil Density/ Consistency or Rock Hardness	Color	Material Classification	* USCS	Remarks	PID or FID Reading (ppm)

*When rock coring enter rock brokeness.

CONVERTED TO WELL : ___ Yes ___ No; WELL I.D.#: _____
REMARKS: _____

Signature(s): _____

LEGEND

SOIL TERMS

UNIFIED SOIL CLASSIFICATION (USCS)

COARSE-GRAINED SOILS — More Than Half of Material is LARGER Than No. 200 Sieve Size

FIELD IDENTIFICATION PROCEDURES (Excluding Particles Larger Than 3 Inches and Basing Fractions on Estimated Weights)			GROUP SYMBOL	TYPICAL NAMES
GRAVELS (50%(+)>1/4"Ø)	CLEAN GRAVELS (Low % Fines)	Wide range in grain size and substantial amounts of all intermediate particle sizes.	GW	Well graded gravels, gravel-sand mixtures, little or no fines.
		Predominantly one size or a range of sizes with some intermediate sizes missing.	GP	Poorly graded gravels, gravel-sand mixtures, little or no fines.
	GRAVELS W/FINES (High % Fines)	Non-plastic fines (for identification procedures, see ML)	GM	Silty gravels, poorly graded gravel-sand-silt mixtures.
		Plastic fines (for identification procedures, see CL)	GC	Clayey gravels, poorly graded gravel-sand-clay mixtures.
SANDS 50%(+)<1/4"Ø	CLEAN SANDS (Low % Fines)	Wide range in grain size and substantial amounts of all intermediate particle sizes.	SW	Well graded sand, gravelly sands, little or no fines.
		Predominantly one size or a range of sizes with some intermediate sizes missing.	SP	Poorly graded sands, gravelly sands, little or no fines.
	SANDS W/FINES (High % Fines)	Non-plastic fines (for identification procedures, see MCL)	SM	Silty sands, poorly graded sand-silt mixtures.
		Plastic fines (for identification procedures, see CL)	SC	Clayey sands, poorly graded sand-clay mixtures.

FINE-GRAINED SOILS — More Than Half of Material is SMALLER Than No. 200 Sieve Size

FIELD IDENTIFICATION PROCEDURES (Excluding Particles Larger Than 3 Inches and Basing Fractions on Estimated Weights)	Identification Procedures on Fraction Smaller than No. 40 Sieve Size: DRY STRENGTH (Crushing Characteristics)	DILATANCY (Reaction to Shaking)	TOUGHNESS (Consistency Near Plastic Limit)	GROUP SYMBOL	TYPICAL NAMES
SILTS AND CLAYS Liquid Limit <50	None to Slight	Quick to Slow	None	ML	Inorganic silts and very fine sands, rock flour, silty or clayey fine sands with slight plasticity.
	Medium to High	None to Very Slow	Medium	CL	Inorganic clays of low to medium plasticity, gravelly clays, sandy clays, silty clays, lean clays.
	Slight to Medium	Slow	Slight	OL	Organic silts and organic silt-clays of low plasticity.
SILTS AND CLAYS Liquid Limit >50	Slight to Medium	Slow to None	Slight to Medium	MH	Inorganic silts, micaceous or diatomaceous fine sandy or silty soils, elastic silts.
	High to Very High	None	High	CH	Inorganic clays of high plasticity, fat clays.
	Medium to High	None to Very Slow	Slight to Medium	OH	Organic clays of medium to high plasticity.
HIGHLY ORGANIC SOILS	Readily identified by color, odor, spongy feel and frequently by fibrous texture.			Pt	Peat and other organic soils

Boundary classifications: Soils possessing characteristics of two groups are designated by combining group symbols. For example, GW-GC, well graded gravel-sand mixture with clay binder.
All sieve sizes on this chart are U.S. Standard.

DENSITY OF GRANULAR SOILS

DESIGNATION	STANDARD PENETRATION RESISTANCE-BLOWS/FOOT
Very Loose	0-4
Loose	5-10
Medium Loose	11-30
Dense	31-50
Very Dense	Over 50

CONSISTENCY OF COHESIVE SOILS

CONSISTENCY	UNC COMPRESSIVE STRENGTH (TONS/SQ. FT.)	STANDARD PENETRATION RESISTANCE-BLOWS/FOOT	FIELD IDENTIFICATION METHODS
Very Soft	Less than 0.25	0 to 2	Easily pentrated several inches by fist
Soft	0.25 to 0.50	2 to 4	Easily penetrated several inches by thumb.
Medium Stiff	0.50 to 1.0	4 to 8	Can be penetrated several inches by thumb.
Stiff	1.0 to 2.0	8 to 15	Readily indented by thumb.
Very Stiff	2.0 to 4.0	15 to 30	Readily indented by thumbnail.
Hard	More than 4.0	Over 30	Indented with difficulty by thumbnail.

ROCK TERMS

ROCK HARDNESS (FROM CORE SAMPLES)

Descriptive Terms	Screwdriver or Knife Effects	Hammer Effects
Soft	Easily Gouged	Crushes when pressed with hammer
Medium Soft	Can be Gouged	Breaks (one blow); crumbly edges
Medium Hard	Can be scratched	Breaks (one blow); sharp edges
Hard	Cannot be scratched	Breaks conchoidally (several blows); sharp edges

ROCK BROKENESS

Descriptive Terms	Abbreviation	Spacing
Very Broken	(V. Br.)	0-2"
Broken	(Br.)	2"-1'
Blocky	(Bl.)	1'-3"
Massive	(M.)	3'-10'

LEGEND:

SOIL SAMPLES - TYPES

S-2" Split-Barrel Sample
ST-3" O.D. Undisturbed Sample
O - Other Samples, Specify in Remarks

ROCK SAMPLES - TYPES

X-NX (Conventional) Core (-2-1/8" O.D.)
Q-NQ (Wireline) Core (-1-7/8" O.D.)
Z - Other Core Sizes, Specify in Remarks

WATER LEVELS

12/18 ▼ 12.6' Initial Level w/Date & Depth
12/18 ▼ 12.6' Stabilized Level w/Date & Depth

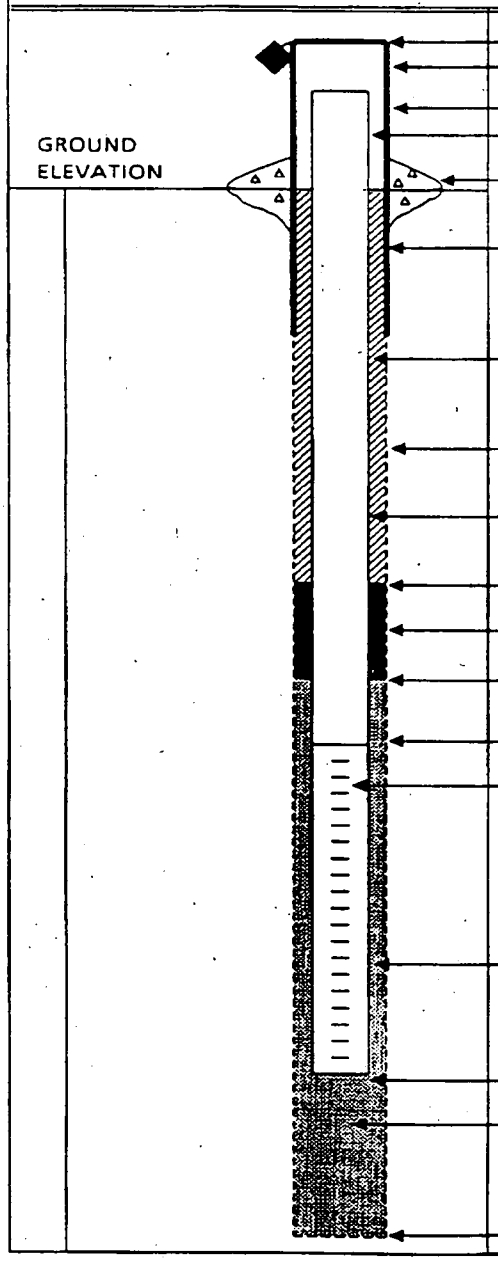
ATTACHMENT C-5
EXAMPLE OVERBURDEN MONITORING WELL SHEET

BORING NO.: _____

OVERBURDEN
MONITORING WELL SHEET

PROJECT _____ LOCATION _____
PROJECT NO. _____ BORING _____
ELEVATION _____ DATE _____
FIELD GEOLOGIST _____

DRILLER _____
DRILLING METHOD _____
DEVELOPMENT METHOD _____



ELEVATION OF TOP OF SURFACE CASING : _____
ELEVATION OF TOP OF RISER PIPE: _____

STICK - UP TOP OF SURFACE CASING: _____
STICK - UP RISER PIPE : _____

TYPE OF SURFACE SEAL: _____

I.D. OF SURFACE CASING: _____
TYPE OF SURFACE CASING: _____

RISER PIPE I.D. _____
TYPE OF RISER PIPE: _____

BOREHOLE DIAMETER: _____

TYPE OF BACKFILL: _____

ELEVATION / DEPTH TOP OF SEAL: _____ / _____

TYPE OF SEAL: _____

DEPTH TOP OF SAND PACK: _____

ELEVATION / DEPTH TOP OF SCREEN: _____ / _____

TYPE OF SCREEN: _____

SLOT SIZE x LENGTH: _____

I.D. OF SCREEN: _____

TYPE OF SAND PACK: _____

ELEVATION / DEPTH BOTTOM OF SCREEN: _____ / _____

ELEVATION / DEPTH BOTTOM OF SAND PACK: _____ / _____

TYPE OF BACKFILL BELOW OBSERVATION WELL: _____

ELEVATION / DEPTH OF HOLE: _____ / _____

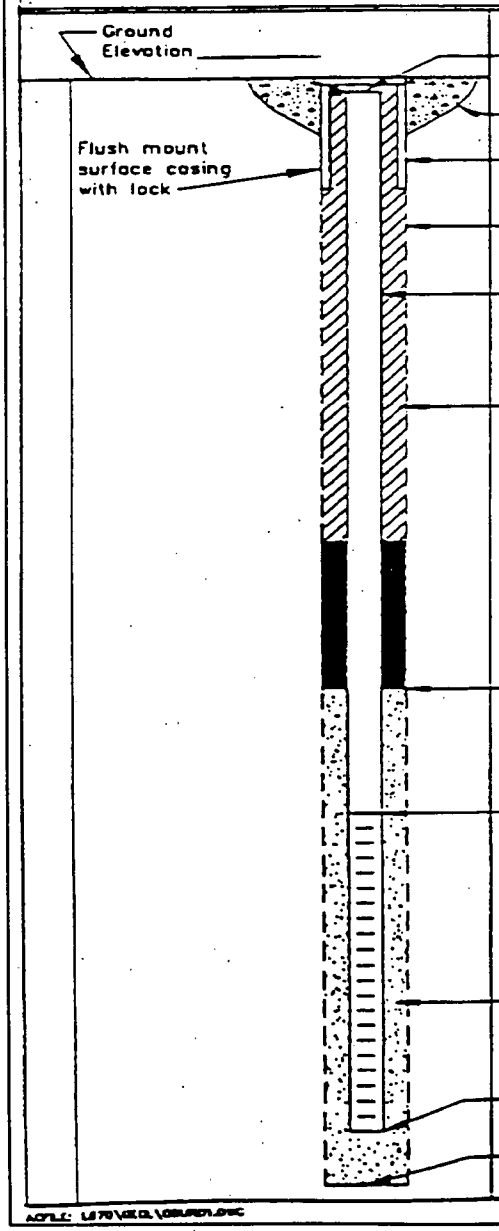
ATTACHMENT C-5A
EXAMPLE OVERBURDEN MONITORING WELL SHEET (FLUSHMOUNT)

BORING NO.: _____

MONITORING WELL SHEET

PROJECT _____ LOCATION _____
PROJECT NO. _____ BORING _____
ELEVATION _____ DATE _____
FIELD GEOLOGIST _____

DRILLER _____
DRILLING METHOD _____
DEVELOPMENT METHOD _____



ELEVATION TOP OF RISER: _____

TYPE OF SURFACE SEAL: _____

TYPE OF PROTECTIVE CASING: _____

I.D. OF PROTECTIVE CASING: _____

DIAMETER OF HOLE: _____

TYPE OF RISER PIPE: _____

RISER PIPE I.D.: _____

TYPE OF BACKFILL/SEAL: _____

_____

DEPTH/ELEVATION TOP OF SAND: _____ / _____

DEPTH/ELEVATION TOP OF SCREEN: _____ / _____

TYPE OF SCREEN: _____

SLOT SIZE x LENGTH: _____

TYPE OF SAND PACK: _____

DIAMETER OF HOLE IN BEDROCK: _____

DEPTH/ELEVATION BOTTOM OF SCREEN: _____ / _____

DEPTH/ELEVATION BOTTOM OF SAND: _____ / _____

DEPTH/ELEVATION BOTTOM OF HOLE: _____ / _____

BACKFILL MATERIAL BELOW SAND: _____

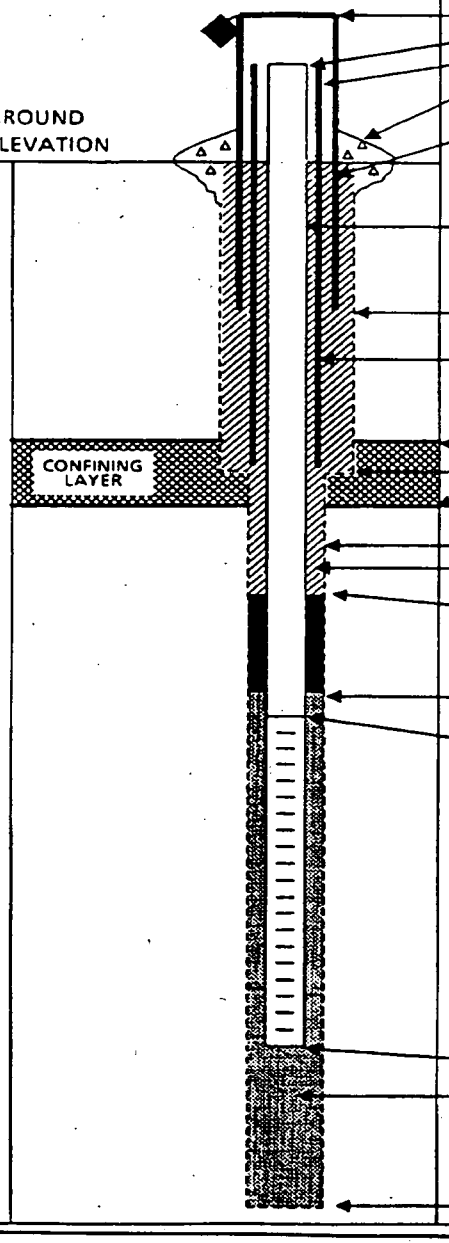
ATTACHMENT C-6
EXAMPLE CONFINING LAYER MONITORING WELL SHEET

BORING NO.: _____

CONFINING LAYER
MONITORING WELL SHEET

PROJECT _____ LOCATION _____
PROJECT NO. _____ BORING _____
ELEVATION _____ DATE _____
FIELD GEOLOGIST _____

DRILLER _____
DRILLING METHOD _____
DEVELOPMENT METHOD _____



ELEVATION OF TOP OF SURFACE CASING : _____
ELEVATION OF TOP OF RISER PIPE: _____
ELEVATION TOP OF PERM. CASING: _____
TYPE OF SURFACE SEAL: _____

I.D. OF SURFACE CASING: _____
TYPE OF SURFACE CASING: _____

RISER PIPE I.D. _____
TYPE OF RISER PIPE: _____

BOREHOLE DIAMETER: _____

PERM. CASING I.D. _____
TYPE OF CASING & BACKFILL: _____

ELEVATION / DEPTH TOP CONFINING LAYER: _____
ELEVATION / DEPTH BOTTOM OF CASING: _____
ELEVATION / DEPTH BOT. CONFINING LAYER: _____

BOREHOLE DIA. BELOW CASING: _____
TYPE OF BACKFILL: _____

ELEVATION / DEPTH TOP OF SEAL: _____
TYPE OF SEAL: _____

DEPTH TOP OF SAND PACK: _____

ELEVATION/DEPTH TOP OF SCREEN: _____
TYPE OF SCREEN: _____

TYPE OF SAND PACK: _____

ELEVATION / DEPTH BOTTOM OF SCREEN: _____

ELEVATION / DEPTH BOTTOM OF SAND PACK: _____
TYPE OF BACKFILL BELOW OBSERVATION WELL: _____

ELEVATION / DEPTH OF HOLE: _____

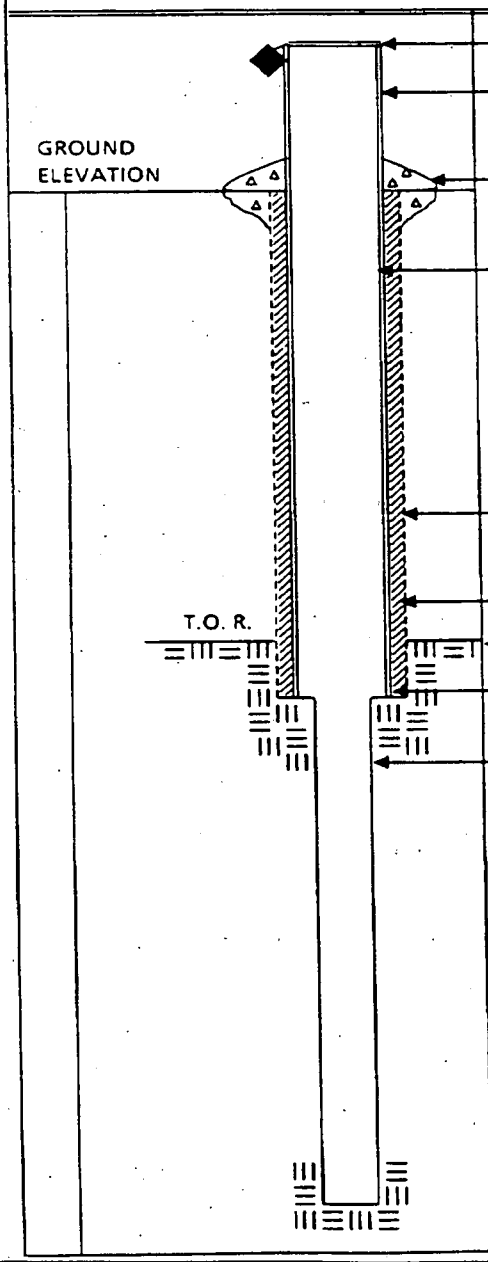
ATTACHMENT C-7
EXAMPLE BEDROCK MONITORING WELL SHEET - OPEN HOLE WELL

BORING NO.: _____

BEDROCK
MONITORING WELL SHEET
OPEN HOLE WELL

PROJECT _____ LOCATION _____
PROJECT NO. _____ BORING _____
ELEVATION _____ DATE _____
FIELD GEOLOGIST _____

DRILLER _____
DRILLING METHOD _____
DEVELOPMENT METHOD _____



ELEVATION OF TOP OF CASING: _____

STICK UP OF CASING ABOVE GROUND SURFACE: _____

TYPE OF SURFACE SEAL: _____

I.D. OF CASING: _____
TYPE OF CASING: _____

TEMP. / PERM.: _____

DIAMETER OF HOLE: _____

TYPE OF CASING SEAL: _____

DEPTH TO TOP OF ROCK: _____

DEPTH TO BOTTOM CASING: _____

DIAMETER OF HOLE IN BEDROCK: _____

DESCRIBE IF CORE / REAMED WITH BIT:

_____
_____
_____

DESCRIBE JOINTS IN BEDROCK AND DEPTH:

_____
_____
_____

ELEVATION / DEPTH OF HOLE: _____ / _____

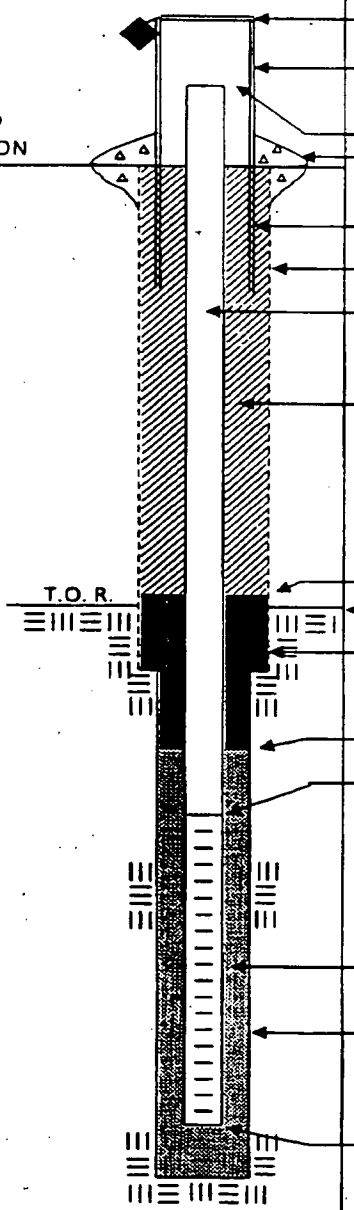
ATTACHMENT C-8
EXAMPLE BEDROCK MONITORING WELL SHEET - WELL INSTALLED IN BEDROCK

BORING NO.: _____

BEDROCK
MONITORING WELL SHEET
WELL INSTALLED IN BEDROCK

PROJECT _____ LOCATION _____
PROJECT NO. _____ BORING _____
ELEVATION _____ DATE _____
FIELD GEOLOGIST _____

DRILLER _____
DRILLING METHOD _____
DEVELOPMENT METHOD _____



ELEVATION OF TOP OF SURFACE CASING: _____

STICK UP OF CASING ABOVE GROUND SURFACE: _____

ELEVATION TOP OF RISER: _____

TYPE OF SURFACE SEAL: _____

I.D. OF SURFACE CASING: _____

DIAMETER OF HOLE: _____

RISER PIPE I.D.: _____

TYPE OF RISER PIPE: _____

TYPE OF BACKFILL: _____

ELEVATION / DEPTH TOP OF SEAL: ____/____

ELEVATION / DEPTH TOP OF BEDROCK: ____/____

TYPE OF SEAL: _____

ELEVATION / DEPTH TOP OF SAND: ____/____

ELEVATION / DEPTH TOP OF SCREEN: ____/____

TYPE OF SCREEN: _____

SLOT SIZE x LENGTH: _____

I.D. SCREEN: _____

TYPE OF SAND PACK: _____

DIAMETER OF HOLE IN BEDROCK:

CORE / REAM: _____

ELEVATION / DEPTH BOTTOM SCREEN: ____/____

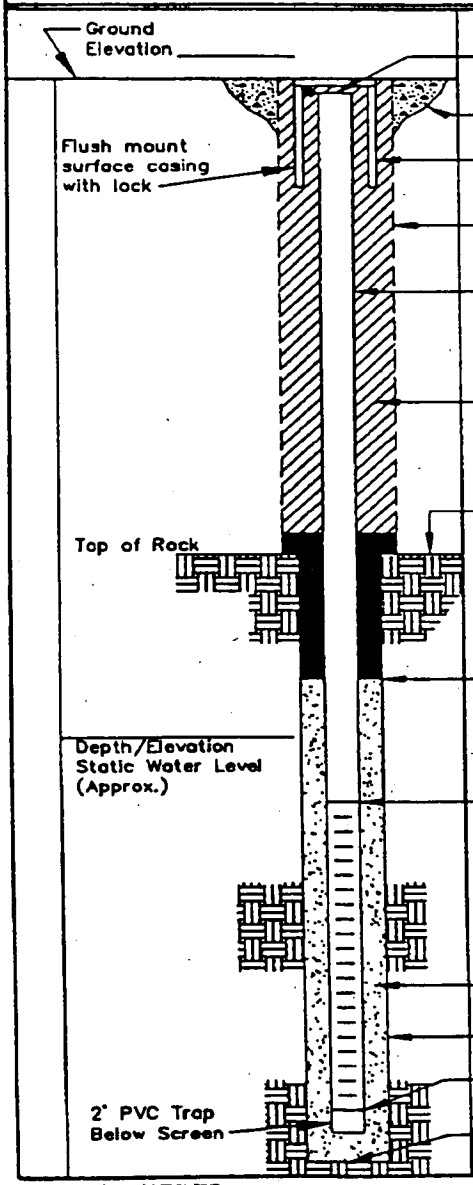
ELEVATION / DEPTH BOTTOM OF HOLE: ____/____

ATTACHMENT C-8A
EXAMPLE BEDROCK MONITORING WELL SHEET
WELL INSTALLED IN BEDROCK (FLUSHMOUNT)

BORING NO.: _____

BEDROCK
MONITORING WELL SHEET
WELL INSTALLED IN BEDROCK

PROJECT: _____ LOCATION: _____ DRILLER: _____
PROJECT NO.: _____ BORING: _____ DRILLING METHOD: _____
ELEVATION: _____ DATE: _____ DEVELOPMENT METHOD: _____
FIELD GEOLOGIST: _____



ELEVATION TOP OF RISER: _____
TYPE OF SURFACE SEAL: _____
TYPE OF PROTECTIVE CASING: _____
I.D. OF PROTECTIVE CASING: _____
DIAMETER OF HOLE: _____
TYPE OF RISER PIPE: _____
RISER PIPE I.D.: _____
TYPE OF BACKFILL/SEAL: _____
_____
DEPTH/ELEVATION TOP OF BEDROCK: _____ / _____
DEPTH/ELEVATION TOP OF SAND: _____ / _____
DEPTH/ELEVATION TOP OF SCREEN: _____ / _____
TYPE OF SCREEN: _____
SLOT SIZE x LENGTH: _____
TYPE OF SAND PACK: _____
DIAMETER OF HOLE IN BEDROCK: _____
DEPTH/ELEVATION BOTTOM OF SCREEN: _____ / _____
DEPTH/ELEVATION BOTTOM OF SAND: _____ / _____
DEPTH/ELEVATION BOTTOM OF HOLE: _____ / _____
BACKFILL MATERIAL BELOW SAND: _____

ACFILE: L670\GEOL\BKDRIL.DWG

ATTACHMENT C-9
EXAMPLE TEST PIT LOG

TEST PIT LOG

Brown & Root Environmental

PROJECT: _____ TEST PIT NO.:

PROJECT NO _____ DATE: _____

LOCATION: _____

FIELD GEOLOGIST: _____

DEPTH (FT.)	LITHOLOGY CHANGE (Depth/Ft.)	MATERIAL DESCRIPTION (Soil Density / Consistency, Color)	USCS	REMARKS

Test Pit Cross Section and / or Plan View

REMARKS _____

PHOTO LOG _____ TEST PIT _____

PAGE ____ OF ____

ATTACHMENT D
EXAMPLE EQUIPMENT CALIBRATION LOG

Brown & Root Environmental

EQUIPMENT CALIBRATION LOG

INSTRUMENT NAME / MODEL : _____ JOB NAME : _____

MANUFACTURER : _____ JOB NUMBER : _____

CALIBRATION DATE	INITIAL SETTINGS	STANDARDS USED	PROCEDURE	ADJUSTMENTS MADE	FINAL SETTINGS	SIGNATURE	COMMENTS

ATTACHMENT E
EXAMPLE DAILY ACTIVITIES RECORD

Brown & Root Environmental

DAILY ACTIVITIES RECORD

PROJECT _____ LOCATION _____
CLIENT _____ ARRIVAL TIME _____ JOB NO. _____
DATE _____ DEPARTURE TIME _____
CONTRACTOR _____ DRILLER _____
BORING NO. _____ HNUS REPRESENTATIVE _____

ITEM	QUANTITY ESTIMATE	QUANTITY TODAY	PREVIOUS TOTAL QUANTITY	CUMULATIVE QUANTITY TO DATE

COMMENTS: _____

APPROVED BY:

_____ _____
HNUS FIELD REPRESENTATIVE DRILLER OR REPRESENTATIVE

ATTACHMENT F
FIELD TRIP SUMMARY REPORT
PAGE 1 OF 2

SUNDAY

Date: _____ Personnel: ____
Weather: _____ Onsite: ____
____

Site Activities: _____

MONDAY

Date: _____ Personnel: ____
Weather: _____ Onsite: ____
____

Site Activities: _____

TUESDAY

Date: _____ Personnel: ____
Weather: _____ Onsite: ____
____

Site Activities: _____

WEDNESDAY

Date: _____ Personnel: ____
Weather: _____ Onsite: ____
____

Site Activities: _____

ATTACHMENT F
PAGE 2 OF 2
FIELD TRIP SUMMARY REPORT

THURSDAY

Date: _____ Personnel: ____
Weather: _____ Onsite: ____
____

Site Activities: _____
_____
_____
_____

FRIDAY

Date: _____ Personnel: ____
Weather: _____ Onsite: ____
____

Site Activities: _____
_____
_____
_____

SATURDAY

Date: _____ Personnel: ____
Weather: _____ Onsite: ____
____

Site Activities: _____
_____
_____
_____

TABLE OF CONTENTS

SECTION **PAGE**

1.0 PURPOSE 2

2.0 SCOPE 2

3.0 GLOSSARY 2

4.0 RESPONSIBILITIES 2

5.0 PROCEDURES 2

5.1 Drilling Equipment 2
5.2 Sampling Equipment 3
5.2.1 Bailers and Bailing Line 3
5.2.2 Sampling Pumps 4
5.2.3 Filtering Equipment 4
5.2.4 Other Sampling Equipment 5
5.3 Field Analytical Equipment 5
5.3.1 Water Level Indicators 5
5.3.2 Probes 5
5.4 Waste Handling 5
5.5 Sources of Contaminated Materials and Containment Methods 6
5.5.1 Decontamination Solutions 6
5.5.2 Disposal Equipment 6
5.5.3 Drilling Muds and Well-Development Fluids 6
5.5.4 Spill-Contaminated Materials 7
5.6 Disposal of Contaminated Materials 7

6.0 REFERENCES 8

ATTACHMENTS

A TWO TYPES OF MUD PITS USED IN WELL DRILLING 9

1.0 PURPOSE

The purpose of this procedure is to provide guidelines regarding the appropriate procedures to be followed when decontaminating drilling equipment, monitoring well materials, chemical sampling equipment and field analytical equipment.

2.0 SCOPE

This procedure addresses drilling equipment and monitoring well materials decontamination, as well as chemical sampling and field analytical equipment decontamination. This procedure also provides general reference information on the control of contaminated materials.

3.0 GLOSSARY

None.

4.0 RESPONSIBILITIES

Project Manager - Responsible for ensuring that all field activities are conducted in accordance with approved project plan(s) requirements.

Field Operations Leader (FOL) - Responsible for the onsite verification that all field activities are performed in compliance with approved Standards Operating Procedures or as otherwise dictated by the approved project plan(s).

5.0 PROCEDURES

To ensure that analytical chemical results reflect actual contaminant concentrations present at sampling locations, the various drilling equipment and chemical sampling and analytical equipment used to acquire the environment sample must be properly decontaminated. Decontamination minimizes the potential for cross-contamination between sampling locations, and the transfer of contamination offsite.

5.1 Drilling Equipment

Prior to the initiation of a drilling program, all drilling equipment involved in field sampling activities shall be decontaminated by steam cleaning at a predetermined area. The steam cleaning procedure shall be performed using a high-pressure spray of heated potable water producing a pressurized stream of steam. This steam shall be sprayed directly onto all surfaces of the various equipment which might contact environmental samples. The decontamination procedure shall be performed until all equipment is free of all visible potential contamination (dirt, grease, oil, noticeable odors, etc.) In addition, this decontamination procedure shall be performed at the completion of each sampling and/or drilling location, including soil borings, installation of monitoring wells, test pits, etc. Such equipment shall include drilling rigs, backhoes, downhole tools, augers, well casings, and screens. Where the drilling rig is set to perform multiple borings at a single area of concern, the steam-cleaning of the drilling rig itself may be waived with proper approval. Downhole equipment, however, must always be steam-cleaned between borings. Where PVC well casings are to be installed, decontamination is not required if the manufacturer provides these casings in factory-sealed, protective, plastic sleeves (so long as the protective packaging is not compromised until immediately before use.

The steam cleaning area shall be designed to contain decontamination wastes and waste waters and can be a lined excavated pit or a bermed concrete or asphalt pad. For the latter, a floor drain must be

provided which is connected to a holding facility. A shallow above-ground tank may be used or a pumping system with discharge to a waste tank may be installed.

In certain cases such an elaborate decontamination pad is not possible. In such cases, a plastic lined gravel bed pad with a collection system may serve as an adequate decontamination area. Alternately, a lined sloped pad with a collection pump installed at the lower end may be permissible. The location of the steam cleaning area shall be onsite in order to minimize potential impacts at certain sites.

Guidance to be used when decontaminating drilling equipment shall include:

- As a general rule, any part of the drilling rig which extends over the borehole, shall be steam cleaned.
- All drilling rods, augers, and any other equipment which will be introduced to the hole shall be steam cleaned.
- The drilling rig, all rods and augers, and any other potentially contaminated equipment shall be decontaminated between each well location to prevent cross contamination of potential hazardous substances.

Prior to leaving at the end of each work day and/or at the completion of the drilling program, drilling rigs and transport vehicles used onsite for personnel or equipment transfer shall be steam cleaned, as practicable. A drilling rig left at the drilling location does not need to be steam cleaned until it is finished drilling at that location.

5.2 Sampling Equipment

5.2.1 Bailers and Bailing Line

The potential for cross-contamination between sampling points through the use of a common bailer or its attached line is high unless strict procedures for decontamination are followed. For this reason, it is preferable to dedicate an individual bailer and its line to each sample point, although this does not eliminate the need for decontamination of dedicated bailers. For non-dedicated sampling equipment, the following conditions and/or decontamination procedures must be followed.

Before the initial sampling and after each successive sampling point, the bailer must be decontaminated. The following steps are to be performed when sampling for organic contaminants. Note: contract-specific requirements may permit alternative procedures.

- Potable water rinse
- Alconox or Liquinox detergent wash
- Scrubbing of the line and bailer with a scrub brush (may be required if the sample point is heavily contaminated with heavy or extremely viscous compounds)
- Potable water rinse
- Rinse with 10 percent nitric acid solution*
- Deionized water rinse

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* Due to the leaching ability of nitric acid on stainless steel, this step is to be omitted if a stainless steel sampling device is being used and metals analysis is required with detection limits less than approximately 50 ppb.

- Acetone or methanol rinse (in some EPA Regions, isopropanol is used instead)
- Hexane rinse**
- Copious distilled/Deionized water rinse
- Air dry

If sampling for volatile organic compounds (VOCs) only, the nitric acid, acetone, methanol, and hexane rinses may be omitted. Only reagent grade or purer solvents are to be used for decontamination. When isopropanol is used, the bailer must be thoroughly dry before using to acquire the next sample.

In general, specially purchased pre-cleaned disposable sampling equipment is not decontaminated (nor is an equipment rinsate blank collected) so long as the supplier has provided certification of cleanliness. If decontamination is performed on several bailers at once (i.e., in batches), bailers not immediately used may be completely wrapped in aluminum foil (shiny-side toward equipment) and stored for future use. When batch decontamination is performed, one equipment rinsate is generally collected from one of the bailers belonging to the batch before it is used for sampling.

It is recommended that clean, dedicated braided nylon or polypropylene line be employed with each bailer use.

5.2.2 Sampling Pumps

Most sampling pumps are low volume (less than 2 gpm) pumps. These include peristaltic, diaphragm, air-lift, pitcher and bladder pumps, to name a few. If these pumps are used for sampling from more than one sampling point, they must be decontaminated prior to initial use and after each use.

The procedures to be used for decontamination of sampling pumps compare to those used for a bailer except that the 10 percent nitric acid solution is omitted. Each of the liquid factions is to be pumped through the system. The amount of pumping is dependent upon the size of the pump and the length of the intake and discharge hoses. Certain types of pumps are unacceptable for sampling purposes. For peristaltic pumps, the tubing is replaced rather than cleaned.

An additional problem is introduced when the pump relies on absorption of water via an inlet or outlet hose. For organic sampling, this hose should be Teflon. Other types of hoses leach organics (especially phthalate esters) into the water being sampled or adsorb organics from the sampled water. For all other sampling, the hose should be Viton, polyethylene, or polyvinyl chloride (listed in order of preference). Whenever possible, dedicated hoses should be used. It is preferable that these types of pumps not be used for sampling, only for purging.

5.2.3 Filtering Equipment

On occasion, the sampling plan may require acquisition of filtered groundwater samples. Field-filtering is addressed in SOP SA-6.1 and should be conducted as soon after sample acquisition as possible. To this end, three basic filtration systems are most commonly used: the in-line disposable Teflon filter, the inert gas over-pressure filtration system, and the vacuum filtration system.

For the in-line filter, decontamination is not required since the filter cartridge is disposable, however, the cartridge must be disposed of in an approved receptacle and the intake and discharge lines must still be decontaminated or replaced before each use.

** If sampling for pesticides, PCBs, or fuels.

For the over-pressure and the vacuum filtration systems, the portions of the apparatus which come in contact with the sample must be decontaminated as outlined in the paragraphs describing the decontamination of bailers. (Note: Varieties of both of these systems come equipped from the manufacturer with Teflon-lined surfaces for those that would come into contact with the sample. These filtration systems are preferred when decontamination procedures must be employed.)

5.2.4 Other Sampling Equipment

Field tools such as trowels and mixing bowls are to be decontaminated in the same manner as described above.

5.3 Field Analytical Equipment

5.3.1 Water Level Indicators

Water level indicators that come into contact with groundwater must be decontaminated using the following steps:

- Rinse with potable water
- Rinse with deionized water
- Acetone or methanol rinse (unless otherwise directed by manufacturer)
- Rinse with deionized water

Water level indicators that do not come in contact with the groundwater but may encounter incidental contact during installation or retrieval need only undergo the first and last steps stated above.

5.3.2 Probes

Probes (e.g., pH or specific-ion electrodes, geophysical probes, or thermometers) which would come in direct contact with the sample, will be decontaminated using the procedures specified above unless manufacturer's instructions indicate otherwise (e.g., dissolved oxygen probes). Probes that contact a volume of groundwater not used for laboratory analyses can be rinsed with deionized water. For probes which make no direct contact, (e.g., OVA equipment) the probe is self-cleaning when exposure to uncontaminated air is allowed and the housing can be wiped clean with paper-towels or cloth wetted with alcohol.

5.4 Waste Handling

For the purposes of these procedures, contaminated materials are defined as any byproducts of field activities that are suspected or known to be contaminated with hazardous substances. These byproducts include such materials as decontamination solutions, disposable equipment, drilling muds, well-development fluids, and spill-contaminated materials and Personal Protection Equipment (PPE).

The procedures for obtaining permits for investigations of sites containing hazardous substances are not clearly defined at present. In the absence of a clear directive to the contrary by the EPA and the states, it must be assumed that hazardous wastes generated during field activities will require compliance with Federal agency requirements for generation, storage, transportation, or disposal. In addition, there may be state regulations that govern the disposal action. This procedure exclusively describes the technical methods used to control contaminated materials.

The plan documents for site activities must include a description of control procedures for contaminated materials. This planning strategy must assess the type of contamination, estimate the amounts that would be produced, describe containment equipment and procedures, and delineate storage or disposal methods. As a general policy, it is wise to select investigation methods that minimize the generation of contaminated spoils. Handling and disposing of potentially hazardous materials can be dangerous and expensive. Until sample analysis is complete, it is assumed that all produced materials are suspected of contamination from hazardous chemicals and require containment.

5.5 Sources of Contaminated Materials and Containment Methods

5.5.1 Decontamination Solutions

All waste decontamination solutions and rinses must be assumed to contain the hazardous chemicals associated with the site unless there are analytical or other data to the contrary. The waste solution volumes could vary from a few gallons to several hundred gallons in cases where large equipment required cleaning.

Containerized waste rinse solutions are best stored in 55-gallon drums (or equivalent containers) that can be sealed until ultimate disposal at an approved facility. Larger equipment such as backhoes and tractors must be decontaminated in an area provided with an impermeable liner and a liquid collection system. A decontamination area for large equipment could consist of a bermed concrete pad with a floor drain leading to a buried holding tank.

5.5.2 Disposable Equipment

Disposable equipment that could become contaminated during use typically includes PPE, rubber gloves, boots, broken sample containers, and cleaning-wipes. These items are small and can easily be contained in 55-gallon drums with lids. These containers should be closed at the end of each work day and upon project completion to provide secure containment until disposed.

5.5.3 Drilling Muds and Well-Development Fluids

Drilling muds and well-development fluids are materials that may be used in groundwater monitoring well installations. Their proper use could result in the surface accumulation of contaminated liquids and muds that require containment. The volumes of drilling muds and well-development fluids used depend on well diameter and depth, groundwater characteristics, and geologic formations. There are no simple mathematical formulas available for accurately predicting these volumes. It is best to rely on the experience of reputable well drillers familiar with local conditions and the well installation techniques selected. These individuals should be able to estimate the sizes (or number) of containment structures required. Since guesswork is involved, it is recommended that an slight excess of the estimated amount of containers required will be available.

Drilling muds are mixed and stored in what is commonly referred to as a mud pit. This mud pit consists of a suction section from which drilling mud is withdrawn and pumped through hoses, down the drill pipe to the bit, and back up the hole to the settling section of the mud pit. In the settling section, the mud's velocity is reduced by a screen and several flow-restriction devices, thereby allowing the well cuttings to settle out of the mud/fluid.

The mud pit may be either portable above-ground tanks commonly made of steel (which is preferred) or stationary in-ground pits as depicted in Attachment A. The above-ground tanks have a major advantage over the in-ground pits because the above-ground tanks isolate the natural soils from the

contaminated fluids within the drilling system. These tanks are also portable and can usually be cleaned easily.

As the well is drilled, the cuttings that accumulate in the settling section must be removed. This is best done by shoveling them into drums or other similar containers. When the drilling is complete, the contents of the above-ground tank are likewise shoveled or pumped into drums, and the tank is cleaned and made available for its next use.

If in-ground pits are used, they should not extend into the natural water table. They should also be lined with a bentonite-cement mixture followed by a layer of flexible impermeable material such as plastic sheeting. Of course, to maintain its impermeable seal, the lining material used would have to be nonreactive with the wastes. An advantage of the in-ground pits is that well cuttings do not necessarily have to be removed periodically during drilling because the pit can be made deep enough to contain them. Depending on site conditions, the in-ground pit may have to be totally excavated and refilled with uncontaminated natural soils when the drilling operation is complete.

When the above-ground tank or the in-ground pit is used, a reserve tank or pit should be located at the site as a backup system for leaks, spills, and overflows. In either case, surface drainage should be such that any excess fluid could be controlled within the immediate area of the drill site.

The containment procedure for well-development fluids is similar to that for drilling muds. The volume and weight of contaminated fluid will be determined by the method used for development. When a new well is pumped or bailed to produce clear water, substantially less volume and weight of fluid result than when backwashing or high-velocity jetting is used.

5.5.4 Spill-Contaminated Materials

A spill is always possible when containers of liquids are opened or moved. Contaminated sorbents and soils resulting from spills must be contained. Small quantities of spill-contaminated materials are usually best contained in drums, while larger quantities can be placed in lined pits or in other impermeable structures. In some cases, onsite containment may not be feasible and immediate transport to an approved disposal site will be required.

5.6 Disposal of Contaminated Materials

Actual disposal techniques for contaminated materials are the same as those for any hazardous substance, that is, incineration, landfilling, treatment, and so on. The problem centers around the assignment of responsibility for disposal. The responsibility must be determined and agreed upon by all involved parties before the field work starts. If the site owner or manager was involved in activities that precipitated the investigation, it seems reasonable to encourage his acceptance of the disposal obligation. In instances where a responsible party cannot be identified, this responsibility may fall on the public agency or private organization investigating the site.

Another consideration in selecting disposal methods for contaminated materials is whether the disposal can be incorporated into subsequent site cleanup activities. For example, if construction of a suitable onsite disposal structure is expected, contaminated materials generated during the investigation should be stored at the site for disposal with other site materials. In this case, the initial containment structures should be evaluated for use as long-term storage structures. Also, other site conditions such as drainage control, security, and soil type must be considered so that proper storage is provided. If onsite storage is expected, then the containment structures should be specifically designed for that purpose.

6.0 REFERENCES

Brown & Root Environmental: Standard Operating Procedure No. 4.33, Control of Contaminated Material.

ATTACHMENT A

TWO TYPES OF MUD PITS USED IN WELL DRILLING

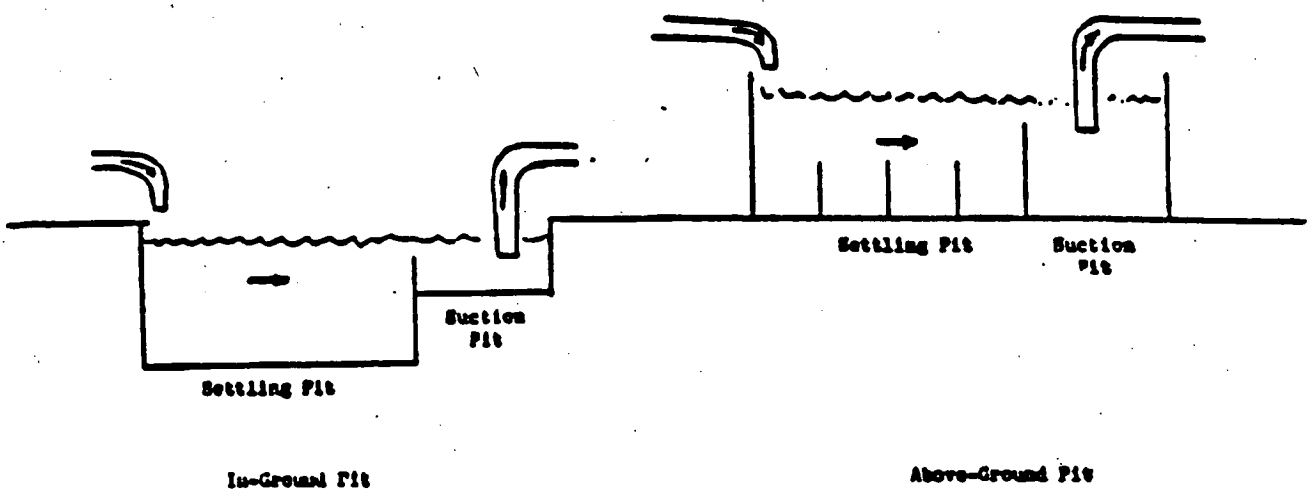

TABLE OF CONTENTS

SECTION		PAGE
1.0	PURPOSE	2
2.0	SCOPE	2
3.0	GLOSSARY	2
4.0	RESPONSIBILITIES	2
5.0	PROCEDURES	2
5.1	Principles of Operation	2
5.2	Calibration	4
5.3	Routine Maintenance	6
5.3.1	Battery Charging	6
5.3.2	Emptying the Hydrogen Cylinder	7
5.3.3	Replacing the Sample Inlet Filter	7
5.4	Troubleshooting	8
5.4.1	MicroFID Fault Messages	8
5.5	Transporting MicroFID	12
6.0	SHIPPING	13
7.0	REFERENCES	13

FIGURES

NUMBER		PAGE
1-1	DOCUMENTATION OF FIELD CALIBRATION	5
6-1	EXAMPLE OF A HAZARDOUS AIRBILL FOR HYDROGEN	14
6-2	EXAMPLE OF A HAZARDOUS AIRBILL FOR METHANE IN AIR	15

1.0 PURPOSE

To establish procedures for the use, calibration, maintenance, troubleshooting, and shipment of the Photovac MicroFID handheld flame ionization detector.

2.0 SCOPE

Applies to all Brown & Root Environmental personnel who operate the MicroFID instrument during the performance of their work.

3.0 GLOSSARY

None.

4.0 RESPONSIBILITIES

Health and Safety Manager (HSM) - The HSM shall ensure that the user has been appropriately trained and certified in the usage of the Photovac 2020 instrument.

Equipment Manager - The Equipment Manager shall ensure that all air monitoring instrumentation slated for field activities has been operationally checked out, fully charged, and calibrated prior to issuance for field service. Maintenance deficiencies identified by the Equipment Manager will require those instruments to be pulled from service until repairs can be facilitated.

Field Operations Leader (FOL)/Field Team Leader (FTL) - The FOL/FTL shall ensure all field team members using monitoring instruments as part of their assigned duties are adequately trained in their proper operation and limitations. The FOL/FTL shall ensure that the air monitoring instruments are employed as directed by site guidance documents (i.e., Work Plan, Health and Safety Plan, etc.). Additionally, the FOL/FTL shall ensure that the appropriate documentation and recordkeeping requirements are fulfilled including Documentation of Calibration and Direct Reading Instrument Response Data Sheets for air monitoring activities.

Health and Safety Officer (HSO) - The HSO is responsible for determining air monitoring requirements for the site activities, and providing direction for air monitoring during specific site activities. This identification of types of air monitoring and direction for use are indicated within the Site-Specific Health and Safety Plan (HASP).

Site Safety Officer (SSO) - The SSO shall ensure the instruments identified are employed in the manner directed by the HSO and action levels employed as contingencies marks for the application of engineering controls, personal protective equipment (PPE) use, and administrative controls are employed as directed. Additionally, he/she shall ensure the instruments are properly maintained and calibrated prior to use in the field. The SSO during specific air monitoring applications including STEL and TWA mode measurements will be responsible for operation and application of this specialty air monitoring employment duty.

5.0 PROCEDURES

5.1 Principles of Operation

The MicroFID is a flame ionization detector used for the measurement of combustible organic compounds in air at parts per million levels. Permanent air gases (argon, carbon dioxide, nitrogen, oxygen, water vapor, etc.) are not ionized by the flame.

When the MicroFID is turned on, the display prompts you to turn on the hydrogen. The internal pump draws air in through MicroFID's inlet. This sample air provides the oxygen necessary for combustion in the hydrogen-fueled flame. When the proper ratio of hydrogen to air is present in the combustion chamber, the flame is automatically started with a glow plug. A thermocouple is used to monitor the status of the flame. When the sample passes through the flame the combustible organic compounds in the sample will be ionized. After the compounds have been ionized, they are subjected to a continuous electric field between the repeller electrode at the jet and the collector electrode. The ions in the electric field generate a current which is proportional to the concentration of the ionized molecules in the ionization chamber. An electrometer circuit converts the current to a voltage that is then fed to the microprocessor which interprets the current in units of ppm. After the sample passes through the flame and has become ionized, it is vented from the detector through a flame arrestor. The flame arrestor prevents the flame from igniting any flammable gases present in the working atmosphere.

MicroFID is strictly an organic compound detector. It does not respond to inorganic compounds. MicroFID's sensitivity is highly dependent on chemical structure and bonding characteristics. The combustion efficiency of a compound determines its sensitivity. Simple saturated hydrocarbons (methane, ethane, etc.) posses high combustion efficiencies and are among the compounds that produce the highest MicroFID response. Organic fuels (acetylene, refined petroleum products), burn easily and are also extremely well detected.

The presence of substituted functional groups (amino, hydroxyl, halogens) on a simple hydrocarbon reduces its combustion efficiency and the MicroFID's sensitivity to the compound. For example, methanol and chloromethane are detectable with MicroFID, but not at the same sensitivity as methane. The number of carbon atoms can also affect the instrument's sensitivity due to substitution. For example, MicroFID is more sensitive to n-butanol than it is to methanol. For additional information regarding response factors of the MicroFID, consult Appendix 8.5 of the User's Manual.

Beginning Operation

You can operate MicroFID without the flame to print or review logged data. In this way you can conserve the hydrogen fuel.

If you choose to start the flame, MicroFID will attempt to ignite the flame once you have turned on the flow of hydrogen gas. If the MicroFID has not been used for a while, it is possible that the gas supply lines are filled with air. If the flame cannot be started, MicroFID will begin a 30 second purge cycle. During the purge cycle it will flush the gas supply lines with hydrogen. After the purge cycle, it will attempt to light the flame again. If it fails again, another purge cycle will be performed and MicroFID will try a third time to ignite the flame. The following steps summarize proper start-up procedures.

1. Turn the instrument on by pressing the front of the On/Off switch. When the instrument is powered up, the version number and creation date of the instrument software are displayed. Press ENTER.

2. You will be prompted to start the flame. If you do not want to start the flame, use the ARROW keys to select "No Flame Needed" and press ENTER. To start the flame, use the ARROW keys to select Start Flame and press ENTER.

3. If you selected "Start Flame," MicroFID will prompt you to turn on the hydrogen. Turn the shut-off valve counterclockwise to start the flow of hydrogen and press ENTER.

4. The pump will start and MicroFID will then ignite the flame. You will hear a small pop when the flame has been ignited. Once the flame has been started the message "Detector flame has been started OK" will be displayed followed by the default display.

The default display provides the following information: instrument status, current detected concentration, event name (if the datalogger is on), time, and date. If an event name is longer than three characters, the bottom line of the display will scroll through the information.

The instrument status appears at the left of the upper line of the display and on the Print and Graph outputs. Each status has a priority assigned to it. If more than one status is in effect, then the status with the highest priority is displayed until the condition is corrected or until the option is turned off.

5.2 Calibration

The calibration (CAL) key is used to calibrate MicroFID. Before beginning calibration, ensure that you have a reliable source of both zero air and calibration gas. To document calibration efforts, field personnel will record information on Figure 1-1. A brief description of the functions under the CAL key are as follows:

1. When you press the CAL key you will first be prompted to select a Cal Memory. Each Cal Memory stores a unique zero point, sensitivity setting, response factor and alarm level.

2. You will then be prompted to enter a response factor. Refer to Appendix 8.5 of the User's Manual for a list of response factors. If the compound is not in that reference, or if you are measuring gas mixtures, enter a value of 1.00. The concentration detected by MicroFID will be multiplied by the response factor before it is displayed and logged.

3. Next select Low Range or High Range operation. Use Low Range if you are sampling concentrations between 0.5 and 2000 ppm (methane equivalents). Use High Range if you are sampling concentrations between 10 and 50,000 ppm (methane equivalents).

4. You will now be prompted to connect a supply of zero air. You can use ambient air or, for best results, use a clean sampling bag filled with zero grade air. In most cases, ambient air will be used provided calibration is performed in an area in which airborne concentrations of contaminants are not present. If using ambient air, press <ENTER> to being zeroing.

5. If you are using a charcoal filter to clean ambient air, connect the filter by loading the Teflon ferrules into the nut (the ferrules and the nut are supplied with the filter). Connect the nut to MicroFID's inlet. Do not tighten the nut. Remove the charcoal filter from its plastic bag and insert it into the nut. Finger tighten the nut onto the inlet. If the filter is not secure, ensure you have inserted the tube far enough into the nut. Do not over-tighten the fitting. Press <ENTER> and the MicroFID will set its zero point. NOTE: The charcoal filter does not filter methane or ethane. If these compounds are present, use a gas bag with a supply of commercial zero air.

6. If you are using a gas bag with zero air, connect the gas bag to the inlet. Open the bag and press <ENTER>. MicroFID will set its zero point.

7. After MicroFID has set its zero point, you can then enter the concentration of the calibration gas (span gas), and then connect the gas bag adapter to the inlet. Open the bag and press <ENTER>. MicroFID sets its sensitivity. Note: You must have a supply of calibration gas ready before calibrating MicroFID. When calibrating MicroFID, ensure the instrument is level. If MicroFID is tilted from side to side, gravity will affect the flame height and cause erroneous readings.

FIGURE 1-1

DOCUMENTATION OF FIELD CALIBRATION

SITE NAME: _____ PROJECT NO.: _____

Date of Calibration	Instrument Name and Model	B & R Environmental Instrument I.D. Number	Person Performing Calibration	Instrument Settings/Readings		Calibration Standard (Lot Number)	Remarks/ Comments
				Pre-Calibration	Post-Calibration		

8. When MicroFID's display reverts to normal, it is calibrated and ready to use. Remove the span gas bag from the inlet.

9. Press the ALARM key and enter the alarm level for the selected CAL memory.

5.3 Routine Maintenance

5.3.1 Battery Charging

A fully charged battery will power the MicroFID for approximately 15 hours. If the instrument is to be used for more than 15 hours, carry a spare battery pack. Battery life is reduced if the instrument is turned off and then on again repeatedly.

When the instrument status displays "LoBat," the battery pack requires changing. When the "LoBat" status is displayed, you have a few minutes of operation left. MicroFID will turn itself off before the battery pack becomes critically low.

To remove the battery pack:

1. Stop the flow of hydrogen gas by turning the hydrogen shut-off valve fully clockwise. Turn the instrument off by pressing the On/Off switch twice.

2. Use the MicroFID multi-tool to loosen the two captive screws in the bottom of the battery pack.

3. A retainer at the rear of the instrument helps secure the battery pack to the instrument. Free the battery pack from the instrument.

4. Connect the charged battery pack to the retainer at the rear of the instrument.

5. Retighten the two captive screws and the bottom of the battery pack.

To charge the battery pack:

1. Ensure the correct plug is installed on the line cord of the battery charger.

2. Plug the charger into the jack located on the front of the battery pack.

3. Plug the charger into an AC outlet. The LED, on the battery pack indicates the charge state. Red indicates the battery is being charged. Green indicates the battery is fully charged and ready for use. It is normal for a fully charged battery to indicate it is charging (red light) when first plugged in. The LED will turn green as the battery charges.

4. When the battery pack is charged remove the charger, first from the wall outlet then from the battery pack.

Charging a fully discharged battery pack will take approximately 8 hours. Leaving the charger connected to a charged battery pack will not harm the battery or the charger in any way. If a battery pack is to be left indefinitely, leave it connected to the charger so that it will be fully charged and ready for operation.

5.3.2 Emptying the Hydrogen Cylinder

When you transport the MicroFID, you should empty the internal hydrogen cylinder and then refill it when you arrive at your destination.

To empty the cylinder:

1. Turn the MicroFID off and open the hydrogen shut-off valve.
2. Remove the battery pack as described above.
3. Locate the purge outlet. It is located on the underside of the instrument.
4. Use the MicroFID multi-tool to turn the screws counterclockwise. Loosen the screw but do not remove it.
5. Leave the instrument so that the purge outlet is facing up. If the purge outlet is facing down, hydrogen will vent into MicroFID's case.
6. If the cylinder is full, it will take approximately 15 minutes to empty.
7. Watch the Contents gauge. When the cylinder is empty, close the purge outlet. Use the MicroFID multi-tool to turn the screw clockwise.
8. Replace the battery pack as discussed above.

5.3.3 Replacing the Sample Inlet Filter

MicroFID is equipped with a combined dust and water filter to reduce detector contamination. As the filter collects dust, MicroFID's inlet flow rate and sensitivity decrease. The filter will not allow water to pass through, but the filter will not stop all solvents.

Replace the filter on a weekly basis, or more frequently if MicroFID is used in a dusty or wet environment. You must replace the filter if MicroFID has been exposed to liquid water. The pump will sound labored when the filter requires replacement.

1. Turn off the instrument and unscrew the filter housing from the detector housing. Be careful not to lose the o-ring seal.
2. Remove the Teflon/Polypropylene filter and install the new filter. Place the filter in the filter housing with the Teflon side facing down into the filter housing and the mesh side facing the MicroFID. Handle the filter disk only by the edges. The mesh may be damaged or contaminated by excessive handling. Use forceps if possible.
3. Replace the filter housing.
4. Calibrate the CAL Memories that you are using before continuing operation.

5.4 Troubleshooting

This section provides guidance for troubleshooting the MicroFID. If problems are not corrected through these troubleshooting methods, contact the Photovac Service Department.

5.4.1 MicroFID Fault Messages

When the "Check" status is displayed, MicroFID's operation is compromised. Press the <TUTOR> key for a two-line description of the fault. One exception is the flame out fault. When a flame out fault occurs, the instrument status changes to "NoFlm."

Fault: **Detector flame has gone out.**

Cause: The hydrogen gas has run out.

Action: Ensure the shut-off valve is open. Check the hydrogen contents gauge on the side of the instrument and refill the hydrogen cylinder if necessary. Ensure the cylinder purge outlet has been closed.

Cause: Oxygen supply is deficient.

Action: Ensure there is an adequate supply of oxygen. If you are sampling very high concentrations it is possible you are sampling above the flame out concentration. The flame out concentration for methane is approximately 52,000 ppm (5.2 percent methane in air).

A minimum of 17 percent oxygen is required to start the hydrogen flame. The oxygen is supplied from the sample as it is drawn in by the pump. A minimum of 10 percent oxygen is required to maintain the hydrogen flame.

Flame out also may occur when sampling enclosed or confined spaces where vapors and gases cannot escape. Watch for indications of increased flame height such as erratic readings or sudden high concentrations followed by a flame out fault.

If you will be using the MicroFID in a highly contaminated area where it is possible that the oxygen content is below 10 percent, watch for indication of reduced flame height such as lowered detection limits or a flame out fault.

Cause: High concentrations of flammable gases (gases within their flammable range) are present. High concentrations of flammable gases can act as an additional fuel source. When this happens, the flame height may increase beyond the confines of the combustion chamber. The hydrogen supply will then be cut-off and the flame will go out.

Action: Move to a location where there is an adequate supply of air and restart the flame. See the information above. Watch for indications of increased flame height such as erratic readings or sudden height concentrations followed by a flame out fault.

Cause: Exhaust port is blocked.

Action: At low temperatures, water vapor, a by-product of the hydrogen flame, may condense at the exhaust port. At sub-zero temperatures the water will freeze and obstruct the exhaust port. If the exhaust port becomes obstructed, pump operation will be inhibited. Flame out may also result. Operate the MicroFID within the operating temperature range 41 to 105 degrees Fahrenheit. In the event that the flame arrestor becomes clogged, contact the Photovac Service Department.

Cause: Sample line is blocked.

Action: Ensure the sample line is not obstructed in any way. If you are using the long sample probe, ensure flow is maintained through the entire length of tubing.

Cause: Inlet filter is plugged.

Action: Replace the inlet filter.

Fault: **Signal form zero gas is too high.**

Cause: Contamination of sample line or fittings before the detector.

Action: Clean or replace the sample line of the inlet filter.

Cause: Span gas is used instead of zero gas.

Action: Ensure clean gas is used to zero the MicroFID. Mark the calibration and zero gas bags clearly.

Cause: Ambient air is contaminated.

Action: If you are unsure about the quality of the ambient air, use a charcoal filter or a supply of commercial zero grade air.

Cause: Hydrogen supply is contaminated.

Action: Hydrogen may react with the carbon element of the steel tank to produce methane. This will only occur if the cylinder is in poor condition and if the hydrogen has a high moisture content. Replace the hydrogen tank. Empty and refill the MicroFID internal cylinder with fresh hydrogen.

Fault: **Signal form the span gas is too small**

Cause: Span gas and zero air are switched.

Action: Ensure calibration is used to calibrate MicroFID. Mark the calibration and zero gas bags clearly. Ensure the span gas is of a reliable concentration.

Fault: **Detector field voltage is low.**

Cause: Internal fault in electronics.

Action: Contact the Photovac Service Department.

Problem: **No instrument response detected, yet compounds are known to be present.**

Cause: MicroFID has not been calibrated properly.

Action: Ensure calibration gas is of a reliable concentration and then calibrate the instrument. After the instrument has been calibrated, sample the bag of calibration gas. A reading equivalent to the calibration gas should be displayed. If not, contact the Photovac Service Department.

Action: When calibrating the MicroFID, ensure the instrument is level. If the MicroFID is tilted side to side, gravity will affect the flame height and cause erroneous readings. If the sampling location is difficult to reach without tilting the instrument, use the long sample probe.

Cause: Background contamination from the hydrogen.

Action: It is possible that the hydrogen has become contaminated and is contributing a high background signal. If the hydrogen supply tank is more than 6 months old it should be replaced with a new cylinder. When ordering hydrogen, specify ultra-high purity (99.999 percent pure). Empty the MicroFID hydrogen cylinder and then refill with hydrogen from the new cylinder.

Problem: **Date and time settings are not retained.**

Cause: MicroFID has not been used for 3 months or more and the internal battery (not the external battery pack) has been discharged.

Action: Turn MicroFID on and allow it to run until a "LoBat" status appears. This will take approximately 15 hours. Remove the battery pack and recharge it overnight. Repeat this procedure for 3 or 4 days. While MicroFID is running the internal battery is charging.

Problem: **Cannot bill the internal hydrogen cylinder to 1800 psig.**

Cause: Supply tank has less than 1800 psig of pressure. You can only fill the internal cylinder to a pressure of less than or equal to the tank pressure.

Action: Fill the internal cylinder to the pressure of the tank or replace the tank with a full one.

Cause: The hydrogen purge outlet is open.

Action: Close the outlet and fill the cylinder.

Cause: There is a problem with the refill adapter.

Action: Contact the Photovac Service Department

Problem: Instrument status shows "Over."

Cause: Rapid change in signal level. The detector electronics have been momentarily saturated.

Action: Wait a few seconds for the status to return to "Ready."

Cause: The detector has become saturated.

Action: Move the MicroFID to a location where it can sample clean air. Sample zero air until the reading stabilizes around "0." If you were using Low Range, switch to High Range. Calibrate the CAL Memory you were using when the "Over" status appeared.

Problem: Display contrast bars are on or display is blank.

Cause: Battery pack is critically low.

Action: Recharge the battery pack or connect the MicroFID to the battery charger.

Cause: The battery pack is not connected to the instrument properly.

Action: Ensure the battery pack has been aligned correctly. Ensure the battery pack is secured by the retainer at the rear of the instrument.

Problem: Sample flow rate varies from 600 ml/min. +/-10 percent.

Cause: Inlet filter has not been installed.

Action: Install an inlet filter.

Cause: Inlet filter has not been tightened onto the detector cap.

Action: Finger-tighten the filter cap.

Cause: Inlet filter is plugged.

Action: Replace the inlet filter.

Cause: Pump has been damaged.

Action: Contact the Photovac Service Department

Cause: Exhaust port is blocked.

Action: At low temperatures, water vapor (a by-product of the hydrogen flame) may condense at the exhaust port. At sub-zero temperatures the water will freeze and obstruct the exhaust port. If the exhaust port becomes obstructed, pump operation will be inhibited. Flame out may also result. Operate the MicroFID within the operating temperature range 41 to 105 degrees Fahrenheit. In the event that the flame arrestor becomes clogged, contact the Photovac Service Department.

Problem: Flame will not ignite.

Cause: The hydrogen gas has run out.

Action: Ensure the shut-off valve is open. Check the hydrogen contents gauge on the side of the instrument and refill the hydrogen cylinder if necessary. Ensure the hydrogen purge outlet is closed.

Cause: Oxygen supply is deficient.

Action: Ensure there is an adequate supply of oxygen. Do not attempt to ignite the flame in a location where there is greater than 10,000 ppm methane or the equivalent concentration of a flammable gas. Move to a location where there are lower concentrations, start the flame and then begin sampling higher concentrations. If the flame goes out while you are sampling very high concentrations, it is possible you are sampling above the flame out concentration. The flame out concentration of methane is approximately 52,000 ppm (5.2 percent methane in air). A minimum of 17 percent oxygen is required to start the hydrogen flame. Oxygen is supplied from the sample as it is drawn in by the pump. A minimum or 10 percent oxygen is required to maintain the hydrogen flame.

Cause: Exhaust port is blocked.

Action: At low temperatures, water vapor (a by-product of the hydrogen flame) may condense at the exhaust port. At sub-zero temperatures the water will freeze and obstruct the exhaust port. If the exhaust port becomes obstructed, pump operation will be inhibited. Flame out may also result. Operate the MicroFID within the operating temperature range 41 to 105 degrees Fahrenheit. In the event that the flame arrestor becomes clogged, contact the Photovac Service Department.

Cause: Hydrogen supply lines are full of air.

Action: If MicroFID has not been operated for some time, it is possible that the hydrogen supply lines contain air. Fill the hydrogen cylinder and then open the hydrogen shut-off valve. Allow the hydrogen to purge the system for about 5 minutes and then turn MicroFID on and start the flame.

Cause: Hydrogen lines are blocked.

Action: Contact the Photovac Service Department.

Problem: Liquid has been aspirated.

Cause: MicroFID has been exposed to a solvent that can pass through the Teflon/Polypropylene filter.

Action: Contact the Photovac Service Department.

5.5 Transporting MicroFID

When you transport MicroFID, you should empty the internal hydrogen cylinder and then refill it when you arrive at your destination. If you are traveling by passenger aircraft, you must empty the hydrogen cylinder. You cannot transport MicroFID by passenger aircraft with hydrogen in the cylinder.

The MicroFID can be shipped to sites. However, if shipment is to be performed while the cylinder still contains hydrogen, a hazardous materials airbill must be filled out. Examples of various completed forms are provided as Figures 6-1 and 6-2.

6.0 SHIPPING

The Photovac may be shipped as cargo or carried on as luggage providing there is no hydrogen fuel source or calibration gas cylinder accompanying the kit. If shipping or transporting the hydrogen fuel source, a Hazardous Airbill (such as the example in Figure 6-1) must be completed. When shipping or transporting the calibration gas, a separate Hazardous Airbill such as the one illustrated in Figure 6-2 must be prepared.

7.0 REFERENCES

MicroFID Handheld Flame Ionization Detector User's Manual, 1995.

FIGURE 6-1

EXAMPLE OF A HAZARDOUS AIRBILL FOR HYDROGEN

SHIPPER'S DECLARATION FOR DANGEROUS GOODS (Provide at least two copies to the airline)

Shipper
BROWN & ROOT ENVIRONMENTAL
SPRING RUN ROAD EXTENTION
CORAOPOLIS, PA 15108

Air Waybill No.
Page of Pages
Shipper's Reference Number (optional)

Consignee

FedEx. Federal Express

Two completed and signed copies of this Declaration must be handed to the operator.

TRANSPORT DETAILS

This shipment is within the limitations prescribed for: (delete non-applicable)

[redacted] | CARGO AIRCRAFT ONLY

Airport of Departure

Airport of Destination:

WARNING

Failure to comply in all respects with the applicable Dangerous Goods Regulations may be in breach of the applicable law, subject to legal penalties. This Declaration must not, in any circumstances, be completed and/or signed by a consolidator, a forwarder, or an IATA cargo agent.

Shipment type: (delete non-applicable)

NON-RADIOACTIVE | [redacted]

NATURE AND QUANTITY OF DANGEROUS GOODS

Dangerous Goods Identification							
Proper Shipping Name	Class or Division	UN or ID No.	Packing Group	Subsidiary Risk	Quantity and type of packaging	Packing Inst.	Authorization
HYDROGEN, COMPRESSED	2.1	UN 1049			1 PLASTIC BOX X KG	200	

Additional Handling Information

(412) 262-4583

Emergency Telephone Number

I hereby declare that the contents of this consignment are fully and accurately described above by the proper shipping name, and are classified, packaged, marked and labelled/placarded, and are in all respects in proper condition for transport according to applicable International and National Governmental Regulations.

Name/Title of Signatory
TOM PATTON, EQUIPMENT MGR.

Place and Date
PITTSBURGH, PA

Signature
(see warning above)

IF ACCEPTABLE FOR PASSENGER AIRCRAFT, THIS SHIPMENT CONTAINS RADIOACTIVE MATERIAL INTENDED FOR USE IN, OR INCIDENT TO, RESEARCH, MEDICAL DIAGNOSIS, OR TREATMENT.

FedEx M-1431 10M LOGOS# 2041750548

FIGURE 6-2

EXAMPLE OF A HAZARDOUS AIRBILL FOR METHANE IN AIR

SHIPPER'S DECLARATION FOR DANGEROUS GOODS (Provide at least two copies to the airline)

Shipper: Brown & Root Environmental
Spring Run Rd. Ext.
Coraopolis, PA 15108

Air Waybill No.
Page 1 of 1 Pages
Shipper's Reference Number (optional)

Consignee

FedEx. Federal Express

Two completed and signed copies of this Declaration must be handed to the operator.

WARNING

Failure to comply in all respects with the applicable Dangerous Goods Regulations may be in breach of the applicable law, subject to legal penalties. This Declaration must not, in any circumstances, be completed and/or signed by a consolidator, a forwarder, or an IATA cargo agent.

TRANSPORT DETAILS

This shipment is within the limitations prescribed for: (delete non-applicable)

PASSENGER AND CARGO AIRCRAFT	~~CARGO AIRCRAFT ONLY~~

Airport of Departure

Airport of Destination:

Shipment type: (delete non-applicable)

NON-RADIOACTIVE	~~RADIOACTIVE~~

NATURE AND QUANTITY OF DANGEROUS GOODS

Dangerous Goods Identification							
Proper Shipping Name	Class or Division	UN or ID No.	Packing Group	Subsidiary Risk	Quantity and type of packaging	Packing Inst.	Authorization
Compressed Gas N.O.S. (Methane in Air)	2.2	UN 1956			1 Fiberboard Box x ____ KG	200	

Additional Handling Information

Emergency Telephone Number (412) 262-4583

I hereby declare that the contents of this consignment are fully and accurately described above by the proper shipping name, and are classified, packaged, marked and labelled/placarded, and are in all respects in proper condition for transport according to applicable International and National Governmental Regulations.

Name/Title of Signatory: Tom Patton, Equip Mgr.
Place and Date: Pittsburgh, PA
Signature (see warning above)

IF ACCEPTABLE FOR PASSENGER AIRCRAFT, THIS SHIPMENT CONTAINS RADIOACTIVE MATERIAL INTENDED FOR USE IN, OR INCIDENT TO, RESEARCH, MEDICAL DIAGNOSIS, OR TREATMENT.

TABLE OF CONTENTS

SECTION		PAGE
1.0	PURPOSE	3
2.0	SCOPE	3
3.0	GLOSSARY	3
4.0	RESPONSIBILITIES	3
5.0	PROCEDURES	4
5.1	Principle of Operation	4
5.1.1	Displays	4
5.1.2	Keys	5
5.2	Default Display	6
5.3	Monitoring	6
5.3.1	Instrument Status	6
5.3.2	Alarms	6
5.4	STEL, TWA, MAX, and PEAK Operation	7
5.4.1	Short-term Exposure Limit (STEL) Mode	7
5.4.2	Time-weighted Average (TWA) Mode	7
5.4.3	MAX Mode	7
5.4.4	PEAK Mode	8
5.5	Set Functions	8
5.5.1	Pump	8
5.5.2	Clock	8
5.5.3	Calibration (Cal)	9
5.5.4	Library (Lib)	9
5.6	Preparing for Field Operation of the Photovac 2020	9
5.7	Maintenance and Calibration Schedule	10
5.7.1	Cleaning the UV Light Source Window	12
5.7.2	Cleaning the Ionization Chamber	12
5.8	Instrument Advantages	12
5.9	Limitations of the Photovac 2020 Photoionization Monitor	12
5.9.1	Variables Affecting Monitoring Data	13
6.0	TROUBLESHOOTING	13
6.1	Fault Messages	13
6.2	Specific Problems	15

TABLE OF CONTENTS

SECTION		PAGE
7.0	SHIPPING	20
8.0	REFERENCES	20

FIGURES

NUMBER		PAGE
5-1	DOCUMENTATION OF FIELD CALIBRATION	11
7-1	EXAMPLE OF A HAZARDOUS AIRBILL FOR ISOBUTYLENE	21

1.0 PURPOSE

To establish procedures for the use, maintenance, and calibration of the Photovac 2020 Photoionization Air Monitor.

2.0 SCOPE

Applies to each usage of the Photovac 2020 Photoionization Air Monitor by Brown & Root Environmental personnel.

3.0 GLOSSARY

Electron volt (eV) - A unit of energy equal to the energy acquired by an electron when it passes through a potential difference of 1 volt in a vacuum. It is equal to $1.602192 \pm 0.000007 \times 10^{-19}$ volts.

Intrinsically Safe (I.S.) - Based on wiring, configuration, design, operation, gasketing, construction, this instrument may be employed within locations in which flammable gases and/or vapors may exist.

Ionization Potential (I.P.) - The energy required to remove an electron from a molecule yielding a positively charged ion and a negatively charged free electron. The instrument measures this energy level.

Photoionization Detector (PID) - Photoionization detector employed as general reference to air monitors of this type. PIDs detection method employs ultraviolet (UV) radiation as an energy source. As air and contaminant are drawn through the ionization chamber the UV light source causes the contaminant with ionization potentials equal to or less than the UV source to break into positive and negatively charge ions. The created ions are subjected to an electrostatic field. The voltage difference is measured in proportion to the calibration reference and the concentration of the contaminant.

Ultraviolet Radiation (UV) - Ultraviolet radiation is the energy source employed by the instrument to ionize collected sample gas streams. The UV lamp source is required to be equal to or greater than the ionization potential of the substance drawn through the instrument in order to create separate ionized species.

4.0 RESPONSIBILITIES

Health and Safety Manager (HSM) - The HSM shall ensure that the user has been appropriately trained and certified in the usage of the Photovac 2020 instrument.

Equipment Manager - The Equipment Manager shall ensure all air monitoring instrumentation slated for field activities has been operationally checked out, fully charged, and calibrated prior to issuing any instrument for field service. Maintenance deficiencies identified by the Equipment Manager will require those instruments to be pulled from service until repairs can be facilitated.

Field Operations Leader (FOL)/Field Team Leader (FTL) - The FOL/FTL shall ensure all field team members employing the monitoring instruments as part of their assigned duties are adequately trained in the operation and limitations of this instrument. The FOL/FTL shall ensure that the air monitoring instruments are employed as directed by site guidance documents (i.e., Work Plan, Health and Safety Plan, etc.). Additionally, the FOL/FTL shall ensure that the appropriate documentation and recordkeeping requirements are fulfilled including Documentation of Calibration and Direct Reading Instrument Response Data Sheets for air monitoring activities.

Health and Safety Officer (HSO) - The HSO is responsible for determining air monitoring requirements for the site activities, and providing direction for air monitoring during specific site activities. This identification of types of air monitoring and direction for use are indicated within the Site-Specific Health and Safety Plan (HASP).

Site Safety Officer (SSO) - The SSO shall ensure the instruments identified are employed in the manner directed by the HSO, and that any action levels specified are observed for the application of engineering controls, personal protective equipment (PPE) use, and administrative controls. Additionally, he/she shall ensure the instruments are properly maintained and calibrated prior to use in the field. The SSO, during specific air monitoring applications including STEL and TWA mode measurements, will be responsible for the operation and application of this specialty air monitoring device.

5.0 PROCEDURES

5.1 Principle of Operation

The Photovac portable photoionizer detects the concentration of many organic (and a few inorganic). The basis for detection of this instrument is the ionization of components of captured gaseous streams. The incoming gas molecules are subjected to ultraviolet (UV) radiation, which is energetic enough to ionize many gaseous compounds. Molecules are transformed into charged-ion pairs, creating a current between two electrodes. Each molecule has a characteristic ionization potential, which is the energy required to remove an electron from the molecule, yielding a positively-charged ion and the free electron. The instrument measures this energy level.

This instrument measures the concentration of airborne photoionizable gases and vapors and automatically displays and records these concentrations. It does not distinguish between individual substances. Readings displayed represent the total concentration of all photoionizable chemicals present in the sample. This instrument is factory set to display concentration in units of ppm or mg/m^3.

The 2020 instrument is easy to operate. The meter display updates itself once per second. Concentrations are directly displayed on the readout.

2020 also performs short-term exposure limit (STEL), time-weighted average (TWA) and PEAK calculations. You can view any of these results, but only one mode may be viewed at a time.

2020 has 6 keys for alphanumeric entry and for accessing multiple functions. The keys are used to set up and calibrate 2020. They allow you to manipulate the concentration data in various ways.

All information entered with the keys and stored in 2020's memory is retained when the instrument is switched off. The clock and calendar continue to operate and do not need to be set each time 2020 is turned on.

5.1.1 Displays

The 2020 has a meter display for reporting detected concentration, and a display used to display status information and guide you through configuration options. All functions of the 2020 will be controlled or reported using one of these displays.

5.1.1.1 Meter Display

The meter display is a 4-digit display. It will always be used for reporting detected concentration. When the detector and pump are off, the meter display will be blank.

In order to accommodate the range of concentrations 2020 can detect, the meter reading will be reported using one of 2 resolutions. A resolution of 0.1 will be used for concentrations below 100 ppm, and a resolution of 1 will be used for concentrations above 100 ppm.

5.1.1.2 Status Display

The status display is a 2 line by 16 character display. The top line is used to display status information and prompts you for information. The bottom line is used for soft key names. Up to 3 names can be displayed for the 3 soft keys. If a name does not appear for a soft key, then the soft key has no associated function.

5.1.2 Keys

5.1.2.1 Fixed Keys

The three round keys below the soft keys each have a fixed function. The first key is the ON/OFF key, the middle key is the EXIT key, and the last key is the ENTER key.

The ON/OFF key is used to both turn power on to the 2020 as well as turn the power off. To turn on 2020, press the ON/OFF key. To turn the power off, press the ON/OFF key and hold it down for 2 seconds, and then release it. This is done to prevent accidental power off.

The EXIT key provides a way of returning to the default display. In the functional map, the soft keys allow you to advance and the EXIT key provides a way to go back. If you are at the initial entry of the menu, EXIT will return you to the default display.

The ENTER key has a context sensitive function. When you are operating or navigating through the function map, the ENTER key is used to exit the functions and return you to the default display. When entering data such as a name, number, date, or time, ENTER is used to confirm the entry.

5.1.2.2 Soft Keys

The three soft keys on 2020 are located directly below the status display. Each key has varying functions for configuring 2020, editing the data logger, and controlling the display. Since only three soft keys are available, each function is broken down into a path.

5.1.2.3 Entering Text With the Soft Keys

For all information that you must enter, the left, center, and right soft keys correspond to the up, down, and right arrow.

The up and down arrows are used to change the character highlighted by the cursor. The right arrow is used to advance the cursor to the next character on the right. When the cursor is advanced past the right most character, it wraps around to the first character again. To accept the changes, press the ENTER key. To ignore the change, press EXIT.

Formatting characters, such as the colon (:) in the time, the decimal (.) in a concentration, and the slash (/) in the date are skipped when advancing the cursor.

All inputs are an 8 character input, which is displayed on the right side of the top line of the status display. The prompt, describing the input, occupies the left half of the top line. The soft keys are defined on the bottom line of the status display.

5.2 Default Display

The meter display shows the detected concentration. The resolution of the display changes with the magnitude of the reading. A reading of 0 to 99.9 will be displayed with a resolution of 0.1 ppm or mg/m^3. A reading greater than 99.9 will be shown with a resolution of 1 ppm or mg/m^3. The meter will display concentrations up to 2000 ppm or 2(XX) mg/m^3.

The status display is used to display the instrument status, date, time, units, and active soft keys.

The default display provides the following information: instrument status, current detected concentration, time, date, and measurement units. The status display toggles between showing time and units and then the date.

When the display mode is MAX, the date and time correspond to the date and time the MAX concentration was recorded. In TWA mode, the time represents the number of hours and minutes during which the TWA has been accumulating. For PEAK and STEL monitoring, the date and time correspond to the current date and time.

5.3 Monitoring

5.3.1 Instrument Status

The instrument status is shown on the left of the first line of the status display and on the Table and Graph outputs. Each status has a priority assigned to it. If more than one status is in effect, then the status with the highest priority is displayed until the condition is corrected or until the option is turned off.

5.3.2 Alarms

While operating the instrument, any one of three alarm conditions can occur. To accurately identify the source of the alarm, each type of alarm has been given a unique status.

In addition to the status, 2020 also has an audible alarm and a visual alarm LED. To conserve power, the 2020 alternates between these two alarm indicators, rather than operating both concurrently. Different alarms are identified by the frequency at which the 2020 alternates as follows: PEAK alarm-5 times per second; STEL alarm-2.5 times per second; and TWA alarm-1.25 times per second.

The left soft key is used for acknowledging alarms, and is named "Ack." If no alarm exists, then the "Ack" key is not shown. To clear the alarm, press the "Ack" key. Once acknowledged, the alarm indicators are cleared. The alarm status will remain until the alarm condition clears.

2020 updates the peak concentration once every second. Following every update, the peak concentration is compared to the peak alarm level, and if exceeded, an alarm is triggered.

If 15 minute average exceeds the selected STEL, a STEL alarm is generated.

The TWA alarm is generated when the current average of concentration, since the TWA was last cleared, has exceeded the TWA exposure limit.

During calibration, all alarms are disabled. Once the calibration is complete the alarms are re-enabled.

5.4 STEL, TWA, MAX, and PEAK Operation

The 2020's meter display can be configured to show one of four values: STEL, TWA, PEAK, and MAX.

5.4.1 Short-term Exposure Limit (STEL) Mode

The Short-term Exposure Limit (STEL) mode displays the concentration as a 15 minute moving average. 2020 maintains 15 samples, each representing a one-minute averaging interval.

Once every minute, the oldest of the 15 samples is replaced with a new one minute average. This moving average provides a 15-minute average of the last 15 minutes with a one-minute update rate. Since the average is calculated using 15 one-minute averages, the meter display will only update once every minute.

STEL is set to zero each time the instrument is turned on. Since STEL is a 15-minute moving average, there is no need to clear or reset the STEL.

STEL calculations are always being performed by 2020. You can display the results of the calculations by selecting STEL as the Display mode.

5.4.2 Time-weighted Average (TWA) Mode

The TWA accumulator sums concentrations every second until 8 hours of data have been combined. If this value exceeds the TWA alarm setting, a TWA alarm is generated. The TWA is not calculated using a moving average. Once 8 hours of data have been summed, the accumulation stops. In order to reset the TWA accumulator, press the "Clr" key.

This sum will only be complete after 8 hours, so the meter displays the current sum divided by 8 hours. While you are in TWA mode, the time on the status display will show the number of minutes and hours of data that TWA has accumulated. When this reaches 8 hours 2020 stops accumulating data and the TWA is complete.

TWA calculations are always being performed by 2020. You can display the results of the calculations by selecting TWA as the Display mode.

5.4.3 MAX Mode

The MAX mode displays the maximum signal, with the date and time that it was recorded. 2020 continues to log data according to the selected averaging interval, but only the maximum detected concentration is displayed on the meter display.

The right soft key is used to clear the meter when displaying MAX. The "Clr" key only affects the reading that the meter is displaying. For example, if you display the MAX reading, and you press "Clr," only the MAX value is cleared. The TWA is still accumulating in the background.

5.4.4 PEAK Mode

The PEAK mode displays the current detected concentration. The reading is updated once a second. In the background, the 2020 data logger is sampling the concentration and measuring minimum, maximum, and average concentrations for the selected averaging interval. At the end of every interval, one entry is placed in the data logger until the data logger is full. Typical application concerning the use of this instrument is operated in this mode. Operation within the other specialized modes are the responsibility of the SSO.

5.5 Set Functions

Set functions are used to setup 2020. There are three functions which can be set on the 2020: Calibration, Pump and Clock.

5.5.1 Pump

The Pump function is used to control the pump. After selecting Set Pump, 2020 responds by displaying the new pump status.

The detector is also turned off when you turn the pump off. This prevents the detector from being damaged when there is no sample flowing through the detector.

When the pump and the detector are off, the meter display will be blank. Turn the pump and detector off when concentration measurements are not necessary, and 2020 will only be used for reviewing data or generating reports. By operating the instrument with the pump and detector off when you do not need them, you will conserve the lives of the battery and ultraviolet (UV) lamp.

1. Press the ENTER key. The top line of the status display changes to "Select?". The bottom line displays 3 soft key names: "Set," "Log," and "Disp."

2. Press the soft key below "Set."

3. The names of the soft keys change to reflect the Set options. The display now shows 3 devices which can be set: "Clock," "Pump," and "Cal." Press the "Pump" key.

4. The 2020 turns the pump off. If the pump was off, pressing "Pump" will turn the pump on.

5. A message will be displayed to show you the status of the pump. 2020 reverts back to the previous menu after a few seconds.

6. To return to the default display, press the ENTER key.

5.5.2 Clock

The Clock function is used to set both the current date and time.

1. Press the ENTER key.

2. Press the "Set" key.

3. When the names of the soft keys change, press the "Clock" key.

The up and down arrows are used to change the character underlined by the cursor. The right arrow is used to advance the cursor to the next character on the right. When the cursor is advanced past the right-most character, it wraps around to the first character again.

Formatting characters, such as the colon (:) in the time and the slash (/) in the date are skipped when advancing the cursor.

4. Use the "arrow keys" to enter the correct time. The time is formatted as Hour:Minute:Second.

5. Press the ENTER key to confirm the time and move to the date option.

6. When setting the date, the 2020 prompts you for the current date formatted as Year/Month/Day. Use the "arrow keys" to enter the correct date.

7. Press the ENTER key to confirm the date and return to the Set options. You can wait for the display to timeout or press ENTER to return to the default display.

5.5.3 Calibration (Cal)

Cal allows you to setup and calibrate 2020. You have three options under the Cal function: "Zero," Span," and "Mem."

A calibration memory consists of a name, a response factor, and PEAK, TWA, and STEL alarm levels.

The "Zero" and "Span" keys are covered in detail in the manufacturer's operations manual for the instrument.

To edit the calibration memory, select "Mem" and then "Chng." The 2020 prompts you with two new soft keys: "User" and "Lib."

5.5.4 Library (Lib)

Library selections simplify Cal Memory programming, and provide standard response factors for approximately 70 applications. "Lib" allows you to select an entry from a pre-programmed library. The name, response factor, and three alarm levels are all set from the library. To select a library entry to program the selected Cal Memory:

1. Select "Set," "Cal," "Mem," "Chng," and "Lib."

2. Use the "Next" and "Prev" keys to scroll through the list. See the manufacturer's manual Appendix 8.7 for a list of the library entries.

5.6 Preparing for Field Operation of the Photovac 2020

Turning 2020 On

1. Turn 2020 on by pressing the ON/OFF key.

2. 2020 will display the software version number. Wait for the 2020 to proceed to the default display.

3. Allow 10 minutes for the instrument to warm up and stabilize.

4. Press the Enter Key. The default display will provide 3 soft key selection "Set," "Log," and "Display."

5. Press "Set." From this option 3 other soft key selections will be offered: "Pump," "Clock," and "Cal."

6. Press "Cal." This will begin the calibration sequence. The first selection is to Zero the instrument.

7. Press Enter, zeroing will begin. (Note: When employing zero gas attach and activate zero gas supply at this time.)

8. The next selection offered will be Span. Press Enter at which time the concentration will be requested. The isobutylene calibration gas employed under general service will be marked on the side of the container. Use the soft keys to toggle into position and to log the concentration. Once the concentration is logged press "Enter." The direction or status display will indicate spanning. At this time hook up the span gas with a regulator to the Photovac 2020, and open it to supply enough flow to elevate the flow rate indicator to the green indicator line (1/8" from the rest position).

9. Once spanning is complete, the alarms which have been disabled during calibration will activate indicating that calibration is complete.

10. Document this calibration procedure using a Document of Calibration form as illustrated in Figure 5-1.

This instrument is ready for general purpose application.

Calibration is to be performed daily or prior to each use in accordance with Section 5.6 of this SOP.

5.7 Maintenance and Calibration Schedule

Function	Frequency
Routine Calibration	Prior to each use
Factory Inspection and Calibration	Once a year, or when malfunctioning
Wipe Down the Outer Casing of the Unit	After each use
Clean UV Light Source	Every 24 hours of operation
Sample Inlet Filter	Change on a weekly basis or as required by level of use
Battery charging	After each use
Clean ionization chamber	Monthly

FIGURE 5-1

DOCUMENTATION OF FIELD CALIBRATION

SITE NAME: _____ PROJECT NO.: _____

Date of Calibration	Instrument Name and Model	B & R Environmental Instrument I.D. Number	Person Performing Calibration	Instrument Settings/Readings		Calibration Standard (Lot Number)	Remarks/ Comments
				Pre-Calibration Setting	Post-Calibration Reading		

5.7.1 Cleaning the UV Light Source Window

1. Turn the FUNCTION switch to the OFF position. Use 2020 multi-tool and remove lamp housing cover.

2. Tilt the lamp housing with one hand over the opening, slide the lamp out of the housing.

3. The lamp window may now be cleaned with any of the following compounds using lens paper:

   a. 11.7 eV Lamp - Dry Aluminum Oxide Powder (3.0 micron powder)
   b. HPLC Grade Methanol - All other lamps

4. Following cleaning, reassemble by first sliding the lamp back into the lamp housing. Replace o-ring as necessary, reinstall lamp housing cover, tighten using 2020 multi-tool. (Do not over tighten).

5. Recalibrate as per Section 5.6.

5.7.2 Cleaning the Ionization Chamber

1. Turn the FUNCTION switch to the OFF position and remove the lamp housing cover and lamp as per Section 5.7.1.

2. Using a gentle jet of compressed air, gently blow out any dust or dirt.

3. Following cleaning, reassemble by first sliding the lamp back into the lamp housing. Replace o-ring as necessary, reinstall lamp housing cover, tighten using 2020 multi-tool. (Do not over tighten).

4. Recalibrate as per Section 5.6.

5.8 Instrument Advantages

The Photovac 2020 is easy to use in comparison to many other types of monitoring instrumentation. Its detection limit range is in the low parts-per-million range. Response time rapidly reaches 90 percent scale of the indicated concentration (less than 3 seconds for benzene). This instrument's automated performance covers multiple monitoring functions simultaneously, incorporating data logging capabilities.

5.9 Limitations of the Photovac 2020 Photoionization Monitor

- Since the 2020 is a nonspecific total gas/vapor detector, it cannot be used to identify unknown chemicals; it can only quantitate them in relationship to a calibration standard (relative response ratio).

- For appropriate application of the 2020, ionization potentials of suspected contaminants must be known.

- Because the types of compounds that the 2020 can potentially detect are only a fraction of the chemicals possibly present at a hazardous waste site or incident, a background or zero reading on this instrument does not necessarily signify the absence of air contaminants.

- The 2020 instrument can monitor only certain vapors and gases in air. Many nonvolatile liquids, toxic solids, particulates, and other toxic gases and vapors cannot be detected.

- PID's are generally not specific. Their response to different compounds is relative to the calibration gas used. This is referred to as relative response ratio. Instrument readings may be higher or lower than the true concentration. This can be an especially serious problem when monitoring for total contaminant concentrations if several different compounds are being detected at once.

- The 2020 is a small, portable instrument which cannot be expected to yield results as accurately as laboratory instruments.

5.9.1 Variables Affecting Monitoring Data

Monitoring hazardous waste site environment can pose a significant challenge in assessing airborne concentrations and the potential threats to site personnel. Several variables may influence both dispersion and the instrument's ability to detect actual concentrations. Some of the variables which may impact these conditions are as follows:

- Temperature - changes in temperature or pressure will influence volatization, and effect airborne concentrations. Additionally, an increase or decrease in temperature ranges may have an adverse effect on the instrument's ability to detect airborne concentrations.

- Humidity - excessive levels of humidity may interfere with the accuracy of monitoring results.

- Rainfall - through increased barometric pressure and water may influence dispersion pathways effecting airborne emissions.

- Electromagnetic interference - high voltage sources, generators, other electrical equipment may interfere with the operation and accuracy of direct-reading monitoring instruments.

6.0 TROUBLESHOOTING

6.1 Fault Messages

When the "Fault" status is displayed, 2020's operation is comprised.

Fault 1: Signal from zero gas is too high.

Cause: If another fault occurred while 2020 was setting its zero point, then this fault is displayed.

Action: Ensure no faults are occurring and calibrate 2020 again.

Cause: Contamination of sample line, sample line, sample probe or fittings before the detector.

Action: Clean or replace the sample line, sample probe or the inlet filter.

Cause: Span gas and zero air are mixed up.

Action: Ensure clean air is used to zero 2020. If you are using gas bags, mark the calibration and zero gas bags clearly.

Cause: Ambient air is contaminated.

Action: If you are unsure about the quality of ambient air, use a supply of commercial zero grade air to zero 2020.

Fault 2: Signal from span gas is too small.

Cause: Operator may have confused the span gas and zero air.

Action: Ensure clean air is used to zero 2020. If you are using gas bags, mark the calibration and zero gas bags clearly.

Action: Ensure the span gas is of a reliable concentration.

Cause: UV lamp window is dirty.

Note: Do not remove the detector lamp in a hazardous location.

Action: Clean the UV lamp window.

Cause: UV lamp is failing.

Note: Do not remove or replace the detector lamp in a hazardous location.

Action: Install a new UV lamp.

Cause: Incompatible application.

Action: The concentration and sample gas are incompatible for use with 2020.

Fault 3: UV lamp fault. UV lamp has not started.

Cause: UV lamp has not started immediately.

Action: This fault may be seen momentarily when 2020 is first turned on. Allow 30 to 60 seconds for the UV lamp to start and the fault to clear.

Cause: UV lamp serial number label is blocking the photocell.

Note: Do not remove or replace the detector lamp in a hazardous location.

Action: If you have a UV lamp with a white serial number label, it is possible that the label is blocking the photocell. Rotate the lamp approximately 90 degree and then try to start 2020 again. If the fault persists, replace the lamp.

Cause: UV lamp not installed.

Note: Do not remove or replace the detector lamp in a hazardous location.

Action: Install a UV lamp.

Cause: UV lamp has failed.

Note: Do not remove or replace the detector lamp in a hazardous location.

Action: Install a new UV lamp.

Cause: Electronic problem.

Action: If a new UV lamp still generates this fault, then contact the Photovac Service Department.

Fault 4: Pump current too low or too high.

Cause: If the pump sounds labored, then the pump is operating beyond normal operating parameters.

Action: Check for an obstruction in the sample line. Make sure sample line, sample probe or inlet filter are not plugged.

Note: Do not replace the inlet filter in a hazardous location.

Action: Replace the inlet filter.

Action: Ensure the sample outlet, located on the underside of 2020, is not obstructed.

Cause: UV lamp is too wide, causing flow to be restricted.

Note: Do not remove or replace the detector lamp in a hazardous location.

Action: If you have a UV lamp with a white serial number label, it is possible that the lamp is too wide for the lampholder. Contact the Equipment Manager.

Cause: 2020 has been exposed to a solvent that can pass through the inlet filter and liquid has been aspirated.

Action: Contact the Equipment Manager.

Cause: The pump has failed.

Action: Contact the Equipment Manager.

6.2 Specific Problems

Problem: Very low or no instrument response detected, yet compounds are known to be present.

Cause: 2020 has not been calibrated properly.

Action: Ensure the calibration gas is of a reliable concentration and then calibrate the instrument as outlined in Section 3.2 or 3.3 of the User's Manual.

After the instrument has been calibrated, sample the bag of calibration gas. A reading equivalent to the calibration gas should be displayed. If not, contact the Equipment Manager.

Note: Do not remove or recharge the battery pack in a hazardous location.

Action: Disconnect the battery charger before calibrating 2020.

Cause: Calibration Memories have not been programmed correctly.

Action: Program all the calibration memories you require for your application. You must use the correct calibration gas and concentration for each Cal Memory.

Cause: Response factor has been set to zero.

Action: Enter the correct response factor. Refer to Appendix 8.6 for a list of response factors. If the compound is not listed in Appendix 8.6 or you are measuring gas mixtures, then enter a value of 1.0. See User's Manual.

Cause: You are not using the correct Cal Memory.

Action: Select the correct Cal Memory for your application.

Note: It does not matter which Cal Memory is selected or which response factor is entered. 2020's response is not specific to any one compound. The reading displayed represents the total concentration of all ionizable compounds in the sample.

Cause: Detector is leaking. A decrease in sensitivity may be due to a leak in the detector.

Note: Do not remove or replace the detection lamp in a hazardous location.

Action: Ensure the UV lamp has been installed correctly.

Action: Ensure the lamp cover has been tightened down. Do not overtighten the cover.

Action: Ensure the o-ring seal on the lamp cover is positioned correctly.

Cause: UV lamp is too long, causing flow to be restricted.

Note: Do not remove or replace the detector lamp in a hazardous location.

Action: If you have a UV lamp with a white serial number label, it is possible that the lamp is too long for the lampholder. Replace the lamp and contact the Equipment Manager.

Cause: UV lamp is too wide, causing flow to be restricted.

Note: Do not remove or replace the detector lamp in a hazardous location.

Action: If you have a UV lamp with a white serial number label, it is possible that the lamp is too wide for the lampholder. Contact the Equipment Manager.

Cause: Sampling environment is extremely humid.

Action: Water vapor is not ionized by the PID, but it does scatter and absorb the light and results in a lower reading.

The 2020 detector has been designed to operate under high humidity conditions. Under extreme conditions you may notice decreased response due to humidity.

Cause: UV lamp is failing.

Note: Do not remove or replace the detector lamp in a hazardous location.

Cause: High concentration of non-ionizable compounds.

Action: Chemical compounds, such as methane, with IPs greater than the 10.6 eV scatter and absorb the UV light. Sensitivity may be decreased significantly.

Application with high backgrounds of such materials,, may be incompatible with 2020. Contact the Photovac Applications Group for more information.

Problem: Erroneously high readings.

Cause: Sampling environment is extremely humid.

Action: Water vapor may contain mineral salts which carry a charge. The water vapor becomes an electrolytic solution which becomes ionized when it enters the detector.

Atmospheric water in areas around the sea or stagnant water may produce a response in the absence of contaminants. The same effect may be seen when conducting ground water investigations in areas where the water is hard because it contains a significant concentration of minerals.

Cause: 2020 has not been calibrated properly.

Action: Ensure the calibration gas is of a reliable concentration and then calibrate the instrument as outlined in Section 5.6.

After the instrument has been calibrated, sample the bag of calibration gas. A reading equivalent to the calibration gas should be displayed. If not contact the Equipment Manager.

Cause: Cal Memories have not been programmed correctly.

Action: Program all the Cal Memories you require for your application. You must use the correct calibration gas and concentration for each Cal Memory. See Section 3.4, of the User's Manual.

Cause: You are not using the correct Cal Memory.

Action: Select the correct Cal Memory for your application. See Section 3.2.2 or 3.3.2, of the User's Manual.

Note: It does not matter which Cal Memory is selected or which response factor is entered. 2020's response is not specific to any one compound. The reading displayed represents the total concentration of all ionizable compounds in the sample.

Cause: Detector has been short circuited by foreign matter in the detector cell.

Note: Do not service 2020 in a hazardous location.

Action: Do not touch the wire grid inside the detector cell. Use a gentle jet of compressed air to remove any dust in the detector cell.

Warning: Do not insert any object, other than the UV lamp, into the lampholder.

Cause: There is an undetermined problem.

Action: Contact the Equipment Manager.

Problem: Date and time settings are not retained.

Cause: The battery pack has been removed before 2020 was turned off.

Note: Do not remove or recharge the battery pack in a hazardous location.

Action: Replace the battery pack and reset the time and date. Ensure 2020 has been turned off before removing the battery pack.

Cause: 2020 has not been used for 3 months or more and the internal battery (not the external battery pack) has discharged.

Note: Do not remove or recharge the battery pack in a hazardous location.

Action: Connect 2020 to the AC adapter and turn 2020 on. Turn the pump off. While 2020 is running the internal battery is charging. Leave the instrument running for approximately 24 hours.

Problem: Instrument status shows "Over."

Cause: High concentrations of gases and vapors will cause a rapid change in signal level. The detector and associated electronics may become temporarily saturated.

Action: Wait a few seconds for the status to return to normal. PIDs are designed to detect relatively low concentrations of gases and vapors. Exposure to very high concentrations may result in a very high or maximum response.

Cause: The detector has become saturated.

Action: Move 2020 to a location where it can sample clean air. Sample clean air until the reading stabilizes around 0.

Cause: Detector has been short circuited by foreign matter in the detector cell.

Note: Do not service 2020 in a hazardous location.

Action: Do not touch the wire grid inside the detector cell. Use a gentle jet of compressed air to remove any dust or dirt in the detector cell.

Warning: Do not insert any object, other than the UV lamp, into the lampholder.

Cause: There is an undetermined problem.

Action: Contact the Equipment Manager.

Problem: Display is blank.

Cause: Battery pack is critically low.

Note: Do not remove or recharge the battery pack in a hazardous location.

Action: Replace the battery pack or connect 2020 to the AC adapter.

Cause: The battery pack is not connected to the instrument correctly.

Action: Ensure the battery pack connector is securely attached to the connector on 2020.

Cause: There is an undetermined problem.

Action: Reset 2020. You must leave the instrument on while you disconnect the battery pack. This will reset the instrument. Reconnect the battery pack and close the battery hatch. Turn on 2020, set the time and date and program all the calibration memories that you are using.

Action: Contact the Equipment Manager.

Problem: Sample flow rate is less than 300 ml/min.

Cause: Inlet filter is plugged.

Note: Do not replace the inlet filter in a hazardous location.

Action: Replace inlet filter.

Cause: Inlet filter has not been installed properly.

Action: Ensure that the inlet filter has been installed correctly.

Cause: UV lamp is too long, causing flow to be restricted.

Note: Do not remove or replace the detector lamp in a hazardous location.

Action: If you have a UV lamp with a white serial number label, it is possible that the lamp is too long for the lampholder. Replace the lamp and contact the Equipment Manager.

Cause: UV lamp is too wide, causing flow to be restricted.

Action: If you have a UV lamp with a white serial number label, it is possible that the lamp is too wide for the lampholder. Contact the Equipment Manager.

Cause: 2020 has been exposed to a solvent that can pass through the inlet filter and liquid has been aspirated.

Action: Contact the Equipment Manager.

Cause: Sample outlet is obstructed.

Action: Ensure the sample outlet is? not obstructed in any way.

Cause: Pump has been damaged.

Action: Contact the Equipment Manager.

Problem: Liquid has been aspirated.

Cause: 2020 has been exposed to a solvent that can pass through the inlet filter.

Action: Contact the Equipment Manager.

Problem: Corrosive gases and vapors have been sampled.

Cause: 2020 has been exposed to corrosive gases and vapors.

Action: Corrosive gases and vapors can affect the electrodes within the detector as well as the lamp window. Prolonged exposure to corrosive materials may result in permanent fogging or etching of the window. If 2020 is exposed to corrosive material, contact the Equipment Manager.

7.0 SHIPPING

The Photovac may be shipped as cargo or carried on as luggage providing there is no calibration gas cylinder accompanying the kit. When shipping or transporting the calibration gas, a Hazardous Airbill including the information as stipulated in Figure 7-1 will be entered.

8.0 REFERENCES

Photovac 2020 Photoionization Monitor User's Manual, 1995.

FIGURE 7-1

EXAMPLE OF A HAZARDOUS AIRBILL FOR ISOBUTYLENE

SHIPPER'S DECLARATION FOR DANGEROUS GOODS (Provide at least two copies to the airline)

Shipper BROWN & ROOT ENVIRONMENTAL
SPRING RUN ROAD EXTENSION
CORAOPOLIS, PA 15108

Air Waybill No.
Page of Pages
Shipper's Reference Number (optional)

Consignee

FedEx. Federal Express

Two completed and signed copies of this Declaration must be handed to the operator.

TRANSPORT DETAILS

This shipment is within the limitations prescribed for: (delete non-applicable)

PASSENGER AND CARGO AIRCRAFT	~~CARGO AIRCRAFT ONLY~~

Airport of Departure

Airport of Destination:

WARNING

Failure to comply in all respects with the applicable Dangerous Goods Regulations may be in breach of the applicable law, subject to legal penalties. This Declaration must not, in any circumstances, be completed and/or signed by a consolidator, a forwarder, or an IATA cargo agent.

Shipment type: (delete non-applicable)

NON-RADIOACTIVE	~~RADIOACTIVE~~

NATURE AND QUANTITY OF DANGEROUS GOODS

Dangerous Goods Identification					Quantity and type of packaging	Packing Inst.	Authorization
Proper Shipping Name	Class or Division	UN or ID No.	Packing Group	Subsidiary Risk			
COMPRESSED GAS N.O.S. (AIR, ISOBUTYLENE	2.2	UN 1956			1 FIBER BOARD box x KG	200	

Additional Handling Information

Emergency Telephone Number (412) 262-4583

I hereby declare that the contents of this consignment are fully and accurately described above by the proper shipping name, and are classified, packaged, marked and labelled/placarded, and are in all respects in proper condition for transport according to applicable International and National Governmental Regulations.

Name/Title of Signatory
TOM PATTON _ EQUIPMENT MANAGER
Place and Date
PITTSBURGH, PA
Signature
(see warning above)

IF ACCEPTABLE FOR PASSENGER AIRCRAFT, THIS SHIPMENT CONTAINS RADIOACTIVE MATERIAL INTENDED FOR USE IN, OR INCIDENT TO, RESEARCH, MEDICAL DIAGNOSIS, OR TREATMENT.

FedEx M-1421 10/88 LOGICAL 2041730848

APPENDIX D

HEALTH AND SAFETY PLAN

Health and Safety Plan
for
Phase II Groundwater RCRA Facility Investigation Solid Waste Management Unit 30 (Landfarm) Naval Surface Warfare Center
Crane, Indiana

Southern Division Naval Facilities Engineering Command

Contract Number N62467-94-D-0888

Contract Task Order 0019

August 1999

HEALTH AND SAFETY PLAN
FOR
PHASE II GROUNDWATER RCRA FACILITY INVESTIGATION
SOLID WASTE MANAGEMENT UNIT 30
(LANDFARM)

NAVAL SURFACE WARFARE CENTER (NSWC)
CRANE, INDIANA

COMPREHENSIVE LONG-TERM
ENVIRONMENTAL ACTION NAVY (CLEAN) CONTRACT

Submitted to:
Southern Division
Naval Facilities Engineering Command
2155 Eagle Drive
North Charleston, South Carolina 29406

Submitted by:
Tetra Tech NUS
Foster Plaza 7
661 Andersen Drive
Pittsburgh, Pennsylvania 15220

CONTRACT NUMBER N62467-94-D-0888
CONTRACT TASK ORDER 0019

AUGUST 1999

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TABLE OF CONTENTS

SECTION **PAGE NO.**

1.0 INTRODUCTION 1-1
1.1 KEY PROJECT PERSONNEL AND ORGANIZATION 1-1
1.2 SITE INFORMATION AND PERSONNEL ASSIGNMENTS 1-3

2.0 EMERGENCY ACTION PLAN 2-1
2.1 INTRODUCTION 2-1
2.2 PRE-EMERGENCY PLANNING 2-1
2.3 EMERGENCY RECOGNITION AND PREVENTION 2-2
2.3.1 Recognition 2-2
2.3.2 Prevention 2-3
2.4 SAFE DISTANCES AND PLACES OF REFUGE 2-3
2.5 EVACUATION ROUTES AND PROCEDURES 2-3
2.6 DECONTAMINATION PROCEDURES / EMERGENCY MEDICAL TREATMENT 2-4
2.7 EMERGENCY ALERTING AND ACTION/RESPONSE PROCEDURES 2-4
2.8 PPE AND EMERGENCY EQUIPMENT 2-5
2.9 EMERGENCY CONTACTS 2-5
2.10 EMERGENCY ROUTE TO HOSPITAL 2-5

3.0 SITE BACKGROUND 3-1
3.1 SITE HISTORY 3-1
3.2 SPECIFIC SITES TO BE INVESTIGATED 3-1
3.2.1 Landfarm (SWMU 30) 3-2

4.0 SCOPE OF WORK 4-1

5.0 TASKS/HAZARDS/ASSOCIATED CONTROL MEASURES SUMMARIZATION 5-1

6.0 HAZARD ASSESSMENT 6-1
6.1 CHEMICAL HAZARDS 6-1
6.2 PHYSICAL HAZARDS 6-1

7.0 AIR MONITORING 7-1

8.0 TRAINING/MEDICAL SURVEILLANCE REQUIREMENTS 8-1
8.1 INTRODUCTORY/REFRESHER/SUPERVISORY TRAINING 8-1
8.1.1 Requirements for TtNUS Personnel 8-1
8.1.2 Requirements for Subcontractors 8-1
8.2 SITE-SPECIFIC TRAINING 8-1
8.3 MEDICAL SURVEILLANCE 8-3
8.3.1 Medical Surveillance Requirements For TtNUS Personnel 8-3
8.3.2 Medical Surveillance Requirements For Subcontractors 8-3
8.3.3 Requirements For All Field Personnel 8-5
8.4 SUBCONTRACTOR EXCEPTION 8-5

TABLE OF CONTENTS (Continued)

SECTION **PAGE NO.**

9.0 SITE CONTROL 9-1
9.1 EXCLUSION ZONE 9-1
9.2 CONTAMINATION REDUCTION ZONE 9-1
9.3 SUPPORT ZONE 9-1
9.4 SITE MAP 9-2
9.5 BUDDY SYSTEM 9-2
9.6 MATERIAL SAFETY DATA SHEET (MSDS) REQUIREMENTS 9-2
9.7 COMMUNICATION 9-2
9.8 SITE VISITORS 9-2
9.9 SITE SECURITY 9-4

10.0 SPILL CONTAINMENT PROGRAM 10-1
10.1 SCOPE AND APPLICATION 10-1
10.2 POTENTIAL SPILL AREAS 10-1
10.3 LEAK AND SPILL DETECTION 10-1
10.4 PERSONNEL TRAINING AND SPILL PREVENTION 10-2
10.5 SPILL PREVENTION AND CONTAINMENT EQUIPMENT 10-2
10.6 SPILL CONTROL PLAN 10-2

11.0 CONFINED SPACE ENTRY 11-1

12.0 MATERIALS AND DOCUMENTS 12-1

13.0 GLOSSARY 13-1

ATTACHMENTS

1 OSHA POSTER
2 MEDICAL DATA SHEET

TABLES

NUMBER		PAGE NO.
2-1	Emergency Reference	2-7
5-1	Task/Hazard Control Measures	5-3
6-1	Chemical, Physical, and Toxicological Data	6-2

FIGURES

NUMBER		PAGE NO.
2-1	Hospital Route Map SWMU 30 Landfarm Work Plan	2-9
2-2	Hospital Route Map RFI Phase II Release Assessment	2-11
8-1	Training Letter	8-2
8-2	Site-Specific Training Documentation	8-4
8-3	Subcontractor Medical Approval Form	8-6
8-4	Medical Surveillance Letter	8-8

1.0 INTRODUCTION

This Health and Safety Plan (HASP) is specifically written for site activities that are to be conducted at the Naval Surface Warfare Center Crane (NSWC Crane), located in Crane, Indiana. In addition to this HASP, a copy of the Brown & Root Environmental Health and Safety Guidance Manual must be present at the site during the performance of site activities. This guidance manual provides detailed information pertaining to the HASP as well as Brown & Root Environmental (TtNUS) standard operating procedures (SOPs). Both documents must be present at the site to comply with the requirements stipulated in the Occupational Safety and Health Administration (OSHA) standard 29 CFR 1910.120.

This HASP has been developed using the latest available information regarding known or suspected chemical contaminants and potential physical hazards associated with the proposed work and site activities. This HASP will be modified if new information becomes available. All changes to the HASP will be requested through the TtNUS Health and Safety Manager (HSM) and the Task Order Manager (TOM). It is the responsibility of the TOM to notify all affected personnel of all changes to this HASP. A Site Safety Follow-up Report will document all changes to this HASP.

The elements of this HASP are in compliance with the requirements established by OSHA 29 CFR 1910.120, "Hazardous Waste Operations and Emergency Response" (HAZWOPER) and sections of 29 CFR 1926 "Safety and Health Regulations For Construction." The information contained in this plan, as well as policies on conducting on-site operations, have been obtained from the TtNUS Health and Safety Program and NSWC Crane policies and procedures.

1.1 KEY PROJECT PERSONNEL AND ORGANIZATION

This section defines responsibility for site safety and health for TtNUS and subcontractor employees engaged in on-site activities. Personnel assigned to these positions will exercise the primary responsibility for all on-site health and safety. These persons will be the primary point of contact for any questions regarding the safety and health procedures and the selected control measures that are to be implemented for on-site activities.

- The TtNUS Task Order Manager (TOM) is responsible for the overall direction of health and safety for this project.

- The Project Health and Safety Officer (PHSO) is responsible for developing the HASP in accordance with applicable OSHA regulations. Specific responsibilities include:

  i. providing information regarding site contaminants and physical hazards associated with the site
  ii. establishing air monitoring and decontamination procedures
  iii. assigning personal protective equipment
  iv. determining emergency response procedures and emergency contacts
  v. stipulating training requirements and reviewing appropriate training and medical surveillance certificates
  vi. providing standard work practices to minimize potential injuries and exposures associated with hazardous waste work.

- The TtNUS Field Operations Leader (FOL) is responsible for implementation of the HASP with the assistance of an appointed Site Safety Officer (SSO). The FOL manages field activities, executes the work plan, and enforces safety procedures as applicable to the work plan.

- The Site Safety Officer (SSO) supports site activities by advising the FOL on all aspects of health and safety on-site. These duties may include:

  i. coordinates all health and safety activities with the FOL
  ii. selects, applies, inspects, and maintains personal protective equipment
  iii. establishes work zones and control points
  iv. implements air monitoring program for on-site activities
  v. verifies training and medical of on-site personnel status in relation to site activities
  vi. implements hazard communication and respiratory protection programs
  vii. coordinates emergency services.
  viii. provides site specific training for all on-site personnel

- Compliance with the requirements stipulated in this HASP is monitored by the SSO and coordinated through the CLEAN Health and Safety Manager.

1.2 SITE INFORMATION AND PERSONNEL ASSIGNMENTS

Site Name: NSWC Crane

Address: Crane, Indiana

Client Contact: Mr. Thomas Brent

Phone Number: (812) 854-6160

Alternate Contact: Ms. Chris Freeman

Phone Number: (812) 854-4423

Project Team:

TtNUS Personnel:	Discipline/Tasks Assigned:
Roger A. Clark, Ph.D.	Task Order Manager (TOM)
Matthew M. Soltis, CIH, CSP	Manager of Health and Safety (HSM)
Donald J. Westerhoff, CSP	Project Health and Safety Officer (PHSO)
TBD	Site Safety Officer (SSO)

Other Potential TtNUS Project Personnel:

Karen Smecker	Keith Simpson
Dave Yost	Terry Rojahn
Fred Ramser	Andy Kendrick
Tim Evans	

Non-TtNUS Personnel	Affiliation/Discipline/Tasks Assigned
TBD	
TBD	

Prepared by: Donald J. Westerhoff, CSP

Reviewed and Approved by:

Roger A. Clark, Ph.D.
Task Order Manager

Matthew M. Soltis CIH, CSP
CLEAN Health & Safety Manager

2.0 EMERGENCY ACTION PLAN

2.1 INTRODUCTION

This section has been developed as part of a preplanning effort to direct and guide field personnel in the event of an emergency. In the event of on-site emergencies, which cannot be handled by on-site personnel, site personnel will be evacuated to a safe place of refuge and the appropriate emergency response agencies will be notified. It has been determined that a majority of potential emergency situations would be better supported by outside emergency responders. Based on this determination, TtNUS and subcontractor personnel will not provide emergency response support beyond the capabilities of on-site response. Workers who are ill or who have suffered a non-serious injury may be transported by site personnel to nearby medical facilities, provided that such transport does not aggravate or further endanger the welfare of the injured/ill person. The emergency response agencies listed in this plan are capable of providing the most effective response, and as such, will be designated as the primary responders. These agencies are located within a reasonable distance from the area of site operations, which ensures adequate emergency response time. This emergency action plan conforms to the requirements of OSHA Standard 29 CFR 1910.38(a), as allowed in OSHA 29 CFR 1910.120(l)(1)(ii).

TtNUS will through necessary services include incidental response measures for incidents such as:

- Incipient stage fire fighting support and prevention
- Incipient spill control and containment measures and prevention
- Removal of personnel from emergency situations
- Provide initial medical support for injuries or illnesses requiring only first-aid level support
- Provide site control and security measures as necessary

2.2 PRE-EMERGENCY PLANNING

Through the initial hazard/risk assessment effort, injuries or illnesses resulting from exposure to chemical or physical hazards or fire are the most probable emergencies that could be encountered during site activities.

To minimize and eliminate these potential emergency situations, pre-emergency planning activities associated with this project include the following. The SSO and/or the FOL are responsible for:

- Coordinating with NSWC Crane Emergency Services personnel to ensure that TtNUS emergency action activities are compatible with existing facility emergency response procedures.

- Establishing and maintaining information at the project staging area (support zone) for easy access in the event of an emergency. This information will include the following:

  - Chemical Inventory (used on-site), with Material Safety Data Sheets.
  - On-site personnel medical records (medical data sheets).
  - A log book identifying personnel on site each day.

It will be the responsibility of the TtNUS FOL to ensure this information is available and present at the site.

- Identifying a chain of command for emergency action.

- Educating site workers to the hazards and control measures associated with planned activities at the site, and to provide early recognition and prevention where possible.

- Drill and practice incidental response measures periodically.

It is understood that the use of two-way communication devices (cellular phones and radios) must be approved by the NSWC Crane Safety Office and such equipment will not be used until official permission is granted.

2.3 EMERGENCY RECOGNITION AND PREVENTION

2.3.1 Recognition

Foreseeable emergency situations that may be encountered during site activities will generally be recognizable by visual observation. Visual observation is primarily relevant for physical hazards that may be associated with the proposed scope of work. Visual observation will also play a role in detecting some chemical overexposures. To adequately recognized exposures to site contaminants, site personnel must have a clear knowledge of signs and symptoms of exposure associated with the site contaminants. This information is provided in Table 6-1 of this HASP. Potential site hazards, the activities unto which they have been associated with, and the recommended control methods are discussed in detail in Section 5.0 and 6.0 of this HASP. Additionally, early recognition of emergency situations will be supported by

periodic site surveys to eliminate any situation predisposed to an emergency. The FOL, and the SSO will make up the site evaluation committee responsible for these periodic surveys. Site surveys will be conducted at least once a week during the initiation of this effort.

The above actions will provide early recognition for potential emergency situations. Should a incident occur, TtNUS will take defensive and offensive measure to control these situations. However, if the FOL and the SSO determine that an incident has progressed to a serious emergency situation, TtNUS will withdraw, and notify the appropriate response agencies.

2.3.2 Prevention

TtNUS and subcontractor personnel will minimize the potential for emergencies by ensuring compliance with the HASP, the Health and Safety Guidance Manual and applicable OSHA regulations.

2.4 SAFE DISTANCES AND PLACES OF REFUGE

In the event that the site must be evacuated, all personnel will immediately stop activities and report to the telephone communications point at the support zone. Telephone communication points and safe places of refuge will be identified prior to the commencement of site activities and will be conveyed to personnel as part of the daily safety meeting conducted each morning. During an evacuation, personnel reporting to the refuge location will remain there until directed otherwise by the TtNUS FOL. The FOL or the SSO will take a head count at this location to account for and to confirm the location of all site personnel. The site log book will be used to take the head count. Emergency response personnel will be immediately notified of any unaccounted personnel.

2.5 EVACUATION ROUTES AND PROCEDURES

An evacuation will be initiated whenever; severe weather is encountered; a fire or explosion occurs; readings on monitoring instrumentation indicate levels of contamination greater than instituted action levels; or if personnel show signs or symptoms of overexposure to potential site contaminants. In the event of an evacuation, personnel will proceed immediately to the designated place of refuge in the support zone, unless doing so would further jeopardize the welfare of workers. In such an event, personnel will proceed to a designated alternate location and remain until further notification from the TtNUS FOL.

Evacuation procedures will be discussed prior to the initiation of any work at the site. Evacuation routes from the site and safe places of refuge are dependent upon the location at which work is being performed

and the circumstances under which an evacuation is required. Additionally, site location and meteorological conditions (i.e., wind speed and direction) may dictate evacuation routes. As a result, assembly points will be selected and communicated to the workers relative to the site location where work is being performed.

2.6 DECONTAMINATION PROCEDURES / EMERGENCY MEDICAL TREATMENT

During an evacuation, decontamination procedures will be performed only if doing so does not further jeopardize the welfare of site workers. Decontamination will not be performed if the action which initiates an evacuation would further endanger the lives of workers if workers were to perform decontamination procedures. However, it is unlikely that an evacuation would occur at this site which would require workers to evacuate the site without first performing decontamination procedures.

2.7 EMERGENCY ALERTING AND ACTION/RESPONSE PROCEDURES

Since TtNUS personnel will be working in close proximity to each other, hand signals, voice commands, and air horns, will be sufficient to alert site personnel of an emergency. If site personnel will be working in remote locations or if site activities are conducted in separate sites simultaneously, two-way radios will be used to communicate between teams of workers. Use of cellular phones and radios must be approved by the NSWC Crane safety office.

If an emergency occurs on Base, the following procedures are to be initiated:

- Initiate an evacuation by hand signals, voice commands, air horn, or two-way radios. Report to the designated refuge assembly point in the support zone.

- Describe to the FOL (who will serve as the Incident Coordinator) what has occurred and as many details as possible. Once all personnel are evacuated, incipient response procedures will be enacted to control the situation.

In the event that site personnel cannot control the incident through offensive and defensive measures, the FOL and SSO will enact the emergency notification procedures to secure additional outside assistance in the following manner:

- On base call 854-3300 or 864-1333* or other emergency contacts (Table 2-1) and report the emergency. Give the emergency operator the location of the emergency, the type of emergency, the

number of injured, and a brief description of what occurred. Stay on the phone and follow the instructions given by the operator. The operator will then notify and dispatch the proper emergency response agencies.

*NOTE: On-base extensions 3300 and 1333 are the primary emergency phone numbers. From an NSWC Crane phone, on Base extensions must be preceded by "854," Off-base numbers can only be reached by dialing "990" or "991" first. Furthermore, all emergencies involving site activities should subsequently be reported to the Environmental Protection Department (x-3114/1132/6160).

- Conduct a head count of site personnel using the site logbook.

2.8 PPE AND EMERGENCY EQUIPMENT

A first-aid kit, eye wash units, stretcher, fire extinguishers (strategically placed), and fire blanket will be maintained on-site and shall be immediately available for use in the event of an emergency.

2.9 EMERGENCY CONTACTS

Prior to performing work at any of the sites, all personnel will be thoroughly briefed on the emergency procedures to be followed in the event of an accident. A mobile phone may be available on site. Table 2-1 provides a list of emergency contacts and their associated telephone numbers. This table must be posted on site where it is readily available to all site personnel. Telephone numbers for the Base Safety Office, the Base Emergency Preparedness Coordinator, and the Base Environmental Office will be obtained and incorporated into Table 2-1 prior to the commencment of site activities.

2.10 EMERGENCY ROUTE TO HOSPITAL

Directions to the Bloomington Hospital:*

Exit NSWC Crane on H-45 through the Bloomington gate. Follow Highway 45 North to Bloomington at Highway 45 and Highway 37. Continue going straight over the overpass (Bloomfield Road). Follow Bloomfield Road North; this road turns into 2nd Street. Follow 2nd Street, hospital will be on the right (601 West 2nd Street)

Directions to Bedford Medical Center:*

Exit the base on H-58, through the Bedford Gate. Head West on State Highway 158. State Highway 158 becomes 16th Street upon entering the City of Bedford. The medical center is on the right shortly after Plaza Drive.

***NOTE:** The Bedford Gate is open only from 0600 - 0830 and 1500 - 1800 hours, whereas the Bloomington Gate is open 24 hours.

TABLE 2-1

EMERGENCY REFERENCE
NSWC CRANE, CRANE, INDIANA

AGENCY	TELEPHONE
Base Emergency Number (Fire Department, Base Security, Ambulance)	dial 854-3300 or 854-1333
Base Environmental Office	(812) 854-6160
Bedford Ambulance	(812) 279-6545
Bloomington Hospital (Bloomington, IN) Hospital, Bedford Medical Center (Bedford, IN)	(812) 336-9515 (812) 275-1200
Poison Control Center	1-800-382-9097
National Response Center	1-800-424-8802
Base Contact Thomas Brent (Environmental Protection Department)	(812) 854-6160
Contract Task Order Manager, Roger A. Clark, Ph.D.	(412) 921-8415
TtNUS Office, Pittsburgh	1-800-245-2730 (412) 921-7090
CLEAN Health and Safety Manager, Matthew M. Soltis, CIH, CSP	(412) 921-8912
Project Health and Safety Officer, Donald J. Westerhoff, CSP	(412) 921-7281

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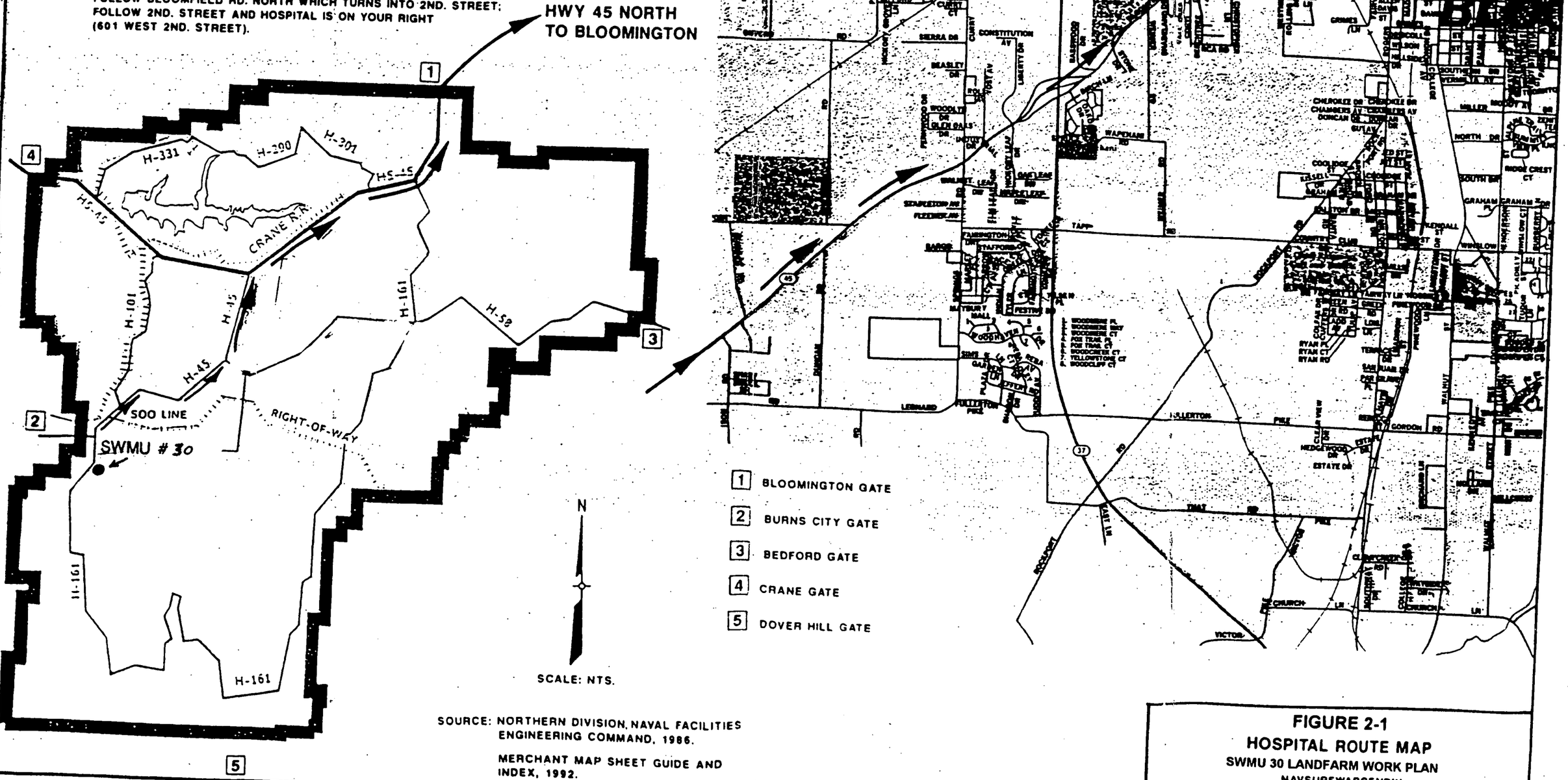
ROUTE TO BLOOMINGTON HOSPITAL

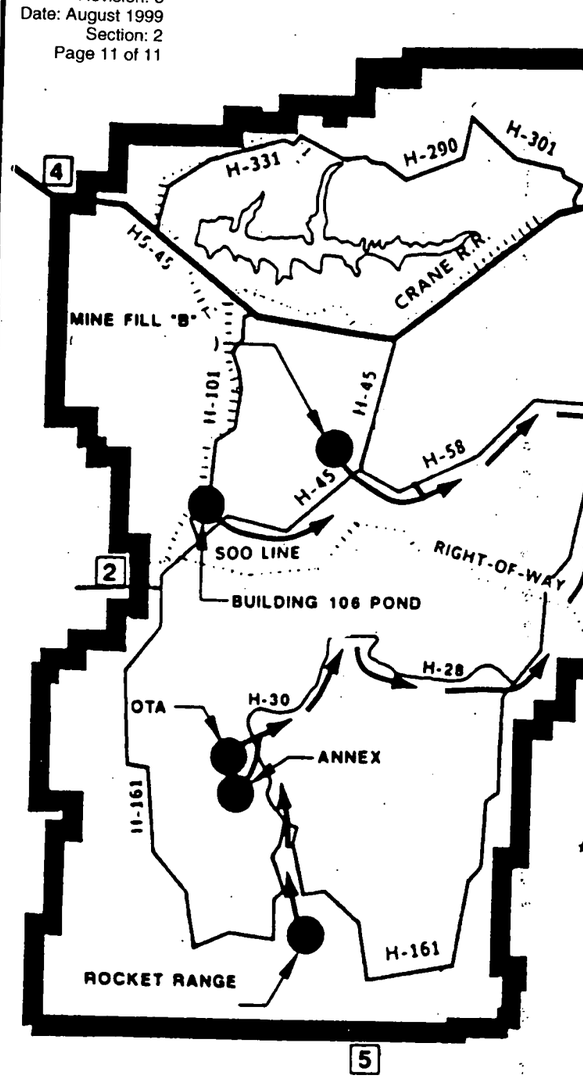
★ EXIT BASE ON H5-45 THROUGH THE BLOOMINGTON GATE; FOLLOW HWY 45 NORTH TO BLOOMINGTON AT HWY 45 AND HWY 37; CONTINUE GOING STRAIGHT OVER THE OVERPASS (BLOOMFIELD RD.); FOLLOW BLOOMFIELD RD. NORTH WHICH TURNS INTO 2ND. STREET; FOLLOW 2ND. STREET AND HOSPITAL IS ON YOUR RIGHT (601 WEST 2ND. STREET).



SOURCE: NORTHERN DIVISION, NAVAL FACILITIES ENGINEERING COMMAND, 1986.

MERCHANT MAP SHEET GUIDE AND INDEX, 1992.

FIGURE 2-1
HOSPITAL ROUTE MAP
SWMU 30 LANDFARM WORK PLAN
NAVSURFWARCENDIV
CRANE, INDIANA



ROUTE TO BEDFORD MEDICAL CENTER

★ EXIT BASE ON H-58, THROUGH THE BEDFORD GATE, HEADING WEST ON STATE HWY. 158. STATE HWY. 158 BECOMES 16TH STREET UPON ENTERING THE CITY OF BEDFORD.

1 BLOOMINGTON GATE

2 BURNS CITY GATE

3 BEDFORD GATE

4 CRANE GATE

5 DOVER HILL GATE

N

SCALE: NTS.

SOURCE: NORTHERN DIVISION NAVAL FACILITIES ENGINEERING COMMAND, 1986.

MERCHANT MAP SHEET GUIDE AND INDEX, 1992.

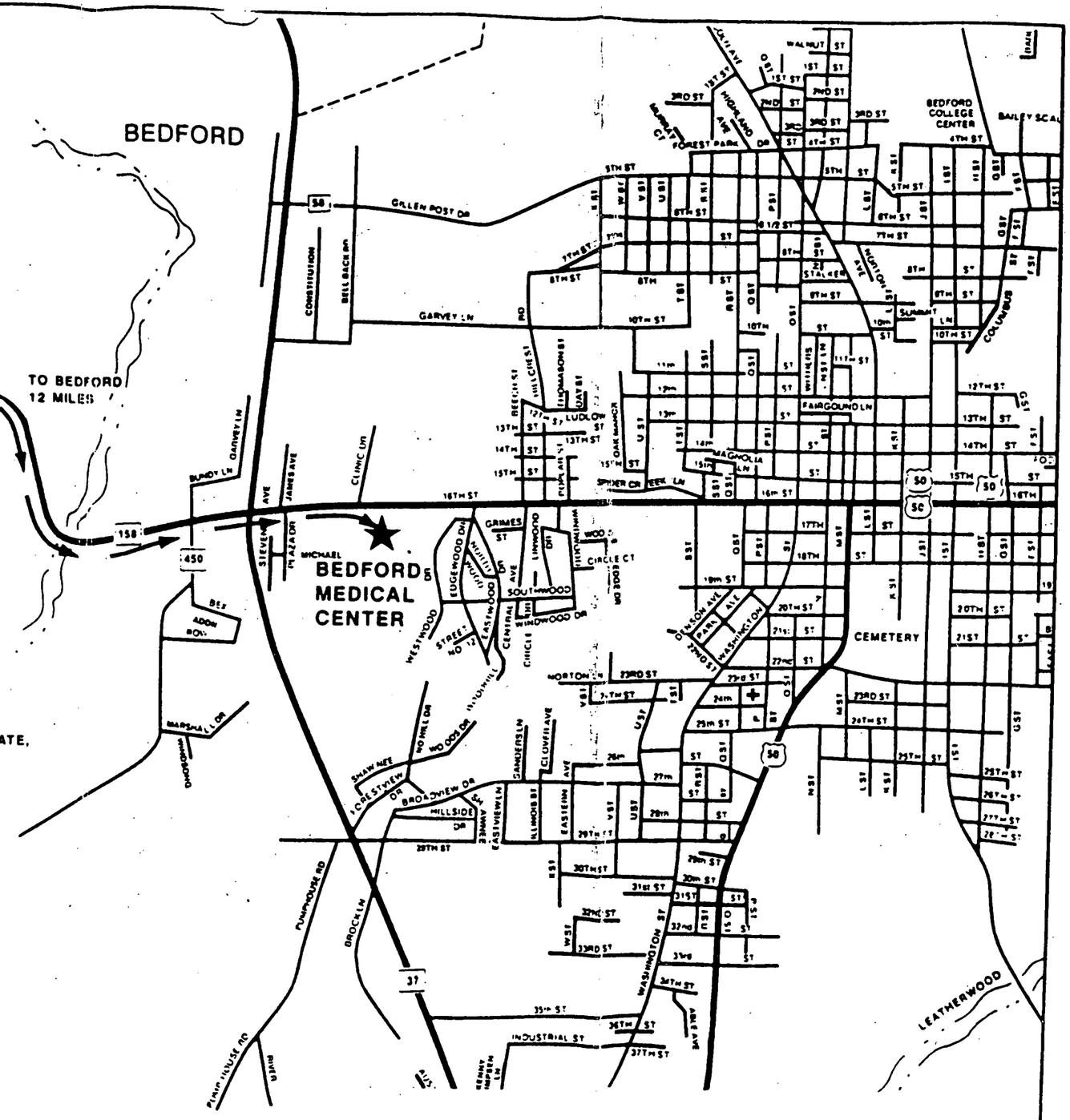


FIGURE 2-2
HOSPITAL ROUTE MAP

RFI PHASE II RELEASE ASSESSMENT
NAVSURFWARCENDIV
CRANE, INDIANA

3.0 SITE BACKGROUND

This section provides information pertaining to NSWC Crane and the sites that are to be investigated. This information will be revised if additional information becomes available or if additional sites are going to be investigated.

3.1 SITE HISTORY

The Naval Surface Warfare Center Crane (NSWC Crane) is located in Crane, Indiana approximately 75 miles southwest of Indianapolis and 71 miles northwest of Louisville, Kentucky. The facility encompasses more than 100 square miles (64,463 acres) in Daviess, Greene, Lawrence, and Martin Counties. It is located in a rural, sparsely populated area. The acreage surrounding the base is either wooded or farmed land. The facility, originally called Naval Ammunition Depot (NAD), Burns City, was opened in 1941 to serve as an inland ammunition production and storage center. The Depot's name was changed to NAD, Crane in 1943. In 1975, the name was changed to Naval Weapons Support Center, Crane and in 1992, the name was again changed to Naval Surface Warfare Center, Crane. Today NSWC Crane's mission is to "provide quality and responsive engineering, technical and material support to the Fleet for combat subsystems, equipment and components, microelectronic technology, microwave components, electronic warfare, acoustic sensors tests, engineering pyrotechnics, small arms, electronic module test and system command." Under the Single Service Management Program, a segment of the Center's mission is to provide support (including environmental protection) to the Crane Army Ammunition Activity (CAAA). The Army is tasked with the production and renovation of conventional ammunition and related items, the performance of manufacturing, engineering, and product quality assurance to support production; and the storage, shipment, demilitarization, and disposal of conventional ammunition and related components. Because of the nature of the Army's operations, CAAA contributes significant financial support for the environmental program through an Interservice Support Agreement.

3.2 SPECIFIC SITES TO BE INVESTIGATED

The Landfarm (SWMU 30) is the only site that is to be investigated as part of the scope of work addressed in this HASP. This HASP addresses activities that will be performed as part of the scope of work to characterize the nature and extent of contamination at SWMU 30.

3.2.1 Landfarm (SWMU 30)

This site is regulated under the state water program. Sludges from the main wastewater treatment plant are applied to the ground under specific permit conditions. No groundwater monitoring has been required by the state, the USEPA would like this media investigated. The primary objective of this CTO is to determine whether or not there is groundwater contamination migration and what its extent may be. Seven wells are in existence and need to be sampled.

4.0 SCOPE OF WORK

This section discusses the activities that are to be performed at the site. Table 5-1 of this HASP provides information related to each of the tasks that are to be performed as part of the scope of work conducted at the site. As new phases or tasks are to be performed at the site, Table 5-1 will be modified accordingly. If tasks other than those presented below are performed at the site, this section will be modified accordingly.

Site activities conducted during this phase of work shall include groundwater sampling of existing wells. Additional information regarding site activities is addressed in the Work Plan which includes a Quality Assurance Project Plan (QAPP) and a Sampling and Analyses Plan (SAP).

5.0 TASKS/HAZARDS/ASSOCIATED CONTROL MEASURES SUMMARIZATION

Table 5-1 of this section serves as the primary portion of the site specific HASP which identifies the tasks that are to be performed under the said HASP. The anticipated hazards, recommended control measures, air monitoring recommendations, required Personal Protective Equipment (PPE), and decontamination measures for each site task are discussed in detail. This table and the associated control measures shall be changed if the scope of work, contaminants of concern, or other conditions change. Through using this table, the FOL and field personnel can determine which hazards are associated with each task, what hazards are present at each site, and what associated control measures are necessary to minimize exposure or injuries related to those hazards. This table also assists the FOL in determining which PPE and decontamination procedures to use based on proper air monitoring techniques and site-specific conditions.

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TABLE 5-1
TASKS/HAZARDS/CONTROL MEASURES COMPENDIUM
NSWC CRANE, CRANE, INDIANA

Tasks/Operation/ Locations	Anticipated Hazards	Recommended Control Measures	Air Monitoring Type Action Levels	Personal Protective Equipment	Decontamination Procedures
Groundwater sampling of existing wells	*Chemical Hazards* 1) Primary site contaminants associated with the SWMU 30 include various inorganics such as chromium, cadmium, copper, lead, nickel, zinc, and cyanide. These contaminants may be bound to soils, sledges and particulates. The primary route of exposure to these contaminant is through ingestion or inhalation or airborne contaminant bound particulates. Analytical data does not indicate the presence of any volatile or semivolatile constituents. However, air monitoring activities will be performed as a precautionary measure to detect the presence of any volatile contaminants that may be present at the site. 2) The potential exists for minor exposure to hydrocarbons (indicated as BTEX) due to the use of gasoline or diesel powered equipment. 3) Cross contamination *Physical hazards* 1) Lifting (muscle strains and pulls) 2) Pinches and compressions 3) Slip, trips, and falls 4) Biological hazards (Insect/animal bites and stings, poisonous plants) 5) Ambient temperature extremes	*Chemical Hazards* 1) Employ real-time monitoring instrumentation, action levels, and identified PPE to control exposures to potentially contaminated water and soils. Minimize the generation of dust via dust suppression methods such as area wetting. 2) Avoid contact with fuels and minimize quantities used and stored on-site. Segregate fuels from combustibles and store in appropriate containers. 3) Decontaminate all equipment and supplies before moving to a new location. *Physical Hazards* 1) Use machinery or multiple personnel for heavy lifts. Use proper lifting techniques. 2) Use pinch bars or other equipment to remove hand from the point of operation. Do not operate equipment unless all appropriate safeguards are in place. 3) Preview work locations for unstable/uneven terrain. 4) Avoid nesting areas, employ repellents if necessary. Wear long pants and long sleeved shirts . Tape pant legs or tuck them into boots to minimize exposed areas of skin. If necessary employ Tyvek coveralls to further protect exposed areas of the body. Report potential hazards to the SSO. 5) Wear appropriate clothing for the anticipated weather conditions while maintaining the required level of protection. Institute work rest regimens in accordance with ACGIH recommendations as discussed in the Health and Safety Guidance Manual. Provide liquids to replace lost body fluids and maintain awareness of signs and symptoms of heat stress.	Based on previous analytical results, it is not anticipated that air monitoring instrumentation will detect the presence of any airborne volatile contaminants. However, monitoring instrumentation will be employed at each location in which sample extraction is being performed. All monitoring wells should be opened prior to sampling to allow venting. All monitoring will be performed using the following instrumentation: <u>Photoionization Detector w/ 11.7 eV UV lamp source</u> 1) Source monitoring will be conducted at regular intervals determined by the SSO. Positive sustained results which may impact filed crews will require the following actions: - Monitor the breathing zone of high risk employees. Any sustained readings in the breathing zone requires evacuation to a safe area until a determination of the source contaminant can be made. This determination can made through use of colormetric tube measurements or grab samples collected using Tedlar bags. If sustained readings are present in the workers breathing zone, contact the Brown & Root Environmental Project Heath and Safety Officer or the CLEAN Health and Safety Manager for additional air monitoring and PPE requirements. In the absence of any positive readings and airborne particulates, site personnel will perform site activities in Level D protection. Site activities may be upgraded to Level C protection if excessive dusts are generated during site activities or if further air monitoring (colormetric tube analysis or grab sampling) indicates the need for increased levels of protection (i.e., air purifying respiratory protection). However, other control measures such as area wetting procedures should be employed whenever possible to control the generation of dusts. Level B protection will be required if the identity of volatile contaminants cannot be determined or if further air monitoring (colormetric tube analysis or grab sampling) indicates the need for increased levels of protection (i.e., supplied air respiratory protection)..	It is anticipated that sampling activities will be performed in Level D Protection. **Level D protection** Hard-hat, safety glasses, steel-toe / shank boots, reflective vest (if in areas of vehicular traffic), impermeable boot covers, and earplugs or muffs. In addition to the above PPE, it is anticipated that most of the sampling may be accomplished through use of layered surgeon style inner nitrile gloves. Grossly contaminated or muddy sampling areas will require the use of nitrile outer gloves as well. Tyvek coveralls shall be required if the potential for contamination of work clothes exists or if poison ivy or if insect (ticks, chiggers, etc.) infestations exist. **Level C protection** Requires the use of Air-Purifying Respirators (APR) with appropriate combination cartridge filters (to be determined by the Project Health and Safety Officer or CLEAN Health and Safety Manager pending contaminant identification). High Efficiency Particulate Air (HEPA) filters will be used if airborne particulates are observed and cannot otherwise be controlled. This level of protection will be utilized whenever: - If colormetric tube analysis or grab sampling conducted as a result of elevated readings on the PID indicate the need for increased levels of protection. - Airborne particulates are generated and cannot be adequately controlled via area wetting methods. Chemical protective clothing will consist of impermeable boot covers, nitrile outer gloves ,surgical style inner gloves, Tyvek coveralls. If free phase product is encountered, or the potential for splashes saturation of work clothes exists, the use of a PVC or PE splash suit will be required. **Level B Protection** Supplied air respiratory protection consisting of either a Self Contained Breathing Apparatus (SCBA) or an air line respirator with escape SCBA. Chemical protective clothing will consist of impermeable boot covers, nitrile outer gloves, surgical style inner gloves, Tyvek coveralls. If the potential exists for saturation or work clothes, PVC or PE coated Tyvek will be substituted for regular Tyvek.	Personnel Decontamination will consist of a soap/water wash and rinse for outer protective equipment (e.g. boots, gloves, PVC splash suits, etc.). This function will take place at a satellite location. Disposable PPE will be bagged between sampling events. This procedure will consist of - Sample acquisition - Sample equipment will be bagged to be decontaminated at the central location - Clean the outside of the sample containers/label/bag - Soap/water wash and rinse of outer boots and gloves - Pack and ice sample transport container Level C and B protection requires a more extensive decontamination procedure which is outlined in Section 6.1.2 and 6.1.3 of Module 2 of the Health and Safety Guidance Manual All decontamination efforts should be followed by personnel performing personnel hygiene practices such as hand and face washing.

TASKS/HAZARDS/CONTROL MEASURES COMPENDIUM
NSWC CRANE, CRANE, INDIANA
PAGE 2

Tasks/Operation/ Locations	Anticipated Hazards	Recommended Control Measures	Air Monitoring	Personal Protective Equipment	Decontamination Procedures
Mobilization/ Demobilization	*Physical Hazards* 1) Lifting (muscle strains and pulls) 2) Pinches and compressions 3) Slip, trips, and falls 4) Moving machinery 5) Biological hazards (Insect/animal bites and stings, poisonous plants) 6) Vehicular and foot traffic 7) Contact with energized systems (e.g., powerlines)	1) Use machinery or multiple personnel for heavy lifts. Use proper lifting techniques. 2) Use pinch bars or other equipment to remove hands from the point of operation. 3) Preview work locations for unstable/uneven terrain. Barricade any excavations from access closer than two feet from the edge. 4) All equipment will be - Inspected in accordance with OSHA, and manufacturers design. - Operated by certified operators, and knowledgeable ground crew. - Establish safe zones of approach (boom + 3 feet). - Secure all loose articles to avoid possible entanglement. 5) Avoid nesting areas, use repellents. Report potential hazards to the SSO. 6) Identify all access routes hours of clearance, Base contacts personnel may approach under the badge clearance and permit system. 7) Inspect all areas prior to work for the potential to contact energized systems. All projecting devices shall be at least 20 feet from overhead power lines.	Not required Air monitoring will not be performed during this task given the low probability of any airborne concentrations of site contaminants being present.	**Level D Protection** - (Minimum Requirements) - Standard field attire (long sleeve shirt; long pants) - Safety shoes (steel- toe/shank) - Safety glasses - Hard-hat (when overhead hazards exists, or identified as a operation requirement) - Reflective vest for high traffic areas - Hearing protection for high noise areas, or as directed on an operation-by operation basis. - Work gloves	Not required Decontamination activities will not be required for this task since there is minimal potential for contamination of equipment or clothing during mobilization and demobilization efforts.

6.0 HAZARD ASSESSMENT

The following section provides information regarding the chemical and physical hazards associated with the NSWC Crane Site and the activities that are to be conducted as part of the scope of work. Table 6-1 provides information related to the chemical hazards that may be present at the site. Specifically, toxicological information, exposure limits, symptoms of exposure, physical properties, and air monitoring and sampling data are discussed in the table. It should be noted that the contaminants of concern may vary between tasks.

6.1 CHEMICAL HAZARDS

Primary site contaminants associated with SWMU 30 are related to sludges from the waste water treatment plant. Additionally, contamination from plating operations is also anticipated to be present at this site. Analytical results indicate the presence of various metals including chromium, copper, cadmium, barium, lead, nickel, and zinc. The presence of cyanide is also indicated in some of the analytical results. The analytical data also included analysis for TCLP volatiles and semivolatile organics. However, all sample results were below laboratory detection levels for constituents of these analyses.

6.2 PHYSICAL HAZARDS

The following is a list of physical hazards that may be encountered at the site or may present during the performance of site activities associated with the scope of work.

- Uneven or unstable terrain (slip, trip, and fall hazards)
- Strain/muscle pulls from manual lifting
- Inclement weather
- Ambient temperature extremes
- Natural hazards (exposure to poisonous or disease carrying animals and plants)

Each of these physical hazards is discussed in the Health and Safety Guidance Manual. Additionally, these physical hazards and the associated control measures are discussed in Table 5-1 of the site specific HASP.

TABLE 6-1

CHEMICAL, PHYSICAL, AND TOXICOLOGICAL DATA
NSWC CRANE, CRANE, INDIANA

Substance	CAS No.	Air Monitoring/Sampling Information		Exposure Limits	Warning Property Rating	Physical Properties	Health Hazard Information
Barium	7440-39-3 as Ba 10022-31-8 as Ba $(NO_3)_2$ 10361-37-2 as Ba Cl_2	Particulate form - This substance is unable to be detected by PID/FID.	Air sample using particulate filter; water desorption; AAS detection. Sampling and analytical protocol shall proceed in accordance with NIOSH Method #7056.	OSHA; NIOSH; ACGIH: 0.5 mg/m^3 IDLH: 50 mg/m^3	No identifiable warning properties to indicate presence and thereby detection. **Recommended APR Cartridge:** Suitable for dust and fume. Organic vapor acid gases with HEPA filter. **Recommended gloves:** This is in the particulate form. Therefore any glove suitable to prevent skin contact (Nitrile has been the one most widely used for the other substances).	**Boiling Pt:** 2984°F 1640°C (decomposes) **Melting Pt:** 1337°F 725°C **Solubility:** Varies between compounds 9/38% **Flash Pt:** Not available (Airborne dust may burn or explode when exposed to heat, flame, or incompatible chemicals) **LEL/LFL:** Not available **UEL/UFL:** Not available **Vapor Density:** Not available **Vapor Pressure:** 10 mmHg @ 1920°F 1049°C **Specific Gravity:** 3.5 **Incompatibilities:** Acids, oxidizers **Appearance and odor:** Silver to white, odorless	Overexposure to this substance results in the solubilization in the water or stomach acids. Symptoms include vomiting, colic, diarrhea (watery sometimes bloody), slow to irregular pulse, transient hypertension, convulsive tremors, and muscular paralysis resulting in stiffness immobility, leg cramps, twitching, and impairment of speech and swallowing. Overexposure to some compounds via inhalation may result in respiratory distress, dyspnea, and baritosis (a benign pneumoconiosis). Direct contact to the skin or eyes may result in irritation.
Cadmium	7440-43-9	Particulate Form - Unable to be easily detected by PID or FID.	Air sample using a mixed cellulose-ester filter / acid desorption and analysis by atomic absorption-flame. Sampling and analytical protocol shall proceed in accordance with NIOSH Method #7300 or #7048.	OSHA: 2 mg/m^3 (0.002 mg/m^3) ACGIH: 0.01 mg/m^3 (total particulate) 0.002 mg/m^3 (respirable particulate) IDLH: 9 mg/m3 (as cd)	The use of an air purifying, full face-piece respirator with a high efficiency particulate air filter for concentrations up to 0.25 mg/m^3. **Recommended Gloves:** This is in particulate form. Therefore any glove suitable to prevent skin contact.	**Boiling Pt:** 1412°F; 767°C **Melting Pt:** 610°F; 321°C **Solubility:** Insoluble **Flash Pt:** Not applicable (Airborne dust may burn or explode when exposed to heat, flame, or incompatible chemicals) **LEL/LFL:** Not applicable **UEL/UFL:** Not applicable **Vapor Density:** Not available **Vapor Pressure:** 1 mmHg @ 741°F; 394°C **Specific Gravity:** 8.65 @ 90°F; 32°C **Incompatibilities:** Strong oxidizers, elemental sulfur, selenium, tellurium, zinc, nitric acid, and hydrazoic acid **Appearance and Odor:** Metal: Silver-white, blue-tinged lustrous, odorless solid. Fume: yellow-brown, finely divided particulate dispersed in air.	Overexposure to this substance may result in irritation to the respiratory tract, dyspnea, tightness in the chest, coughing, possibly pulmonary edema. Overexposure to fumes causes symptoms characteristic of the flu (headaches, chills, muscle aches, nausea, vomiting, diarrhea). Chronic exposure may result in damage to the lungs, kidneys and liver. This substance has been identified as a confirmed animal; potential human carcinogen by IARC and NTP.

049910/P

TABLE 6-1

CHEMICAL, PHYSICAL, AND TOXICOLOGICAL DATA
NSWC CRANE, CRANE, INDIANA
PAGE 2

Substance		Air Monitoring/Sampling Information		Exposure Limits	Warning Property Rating	Physical Properties	Health Hazard Information
Chromium Compounds	7440-47-3 (Element)	Not detectable by PID. Not detectable by FID.	Air sample using mixed cellulose -ester filter; acid desorption and analysis by atomic absorption. Sampling and analytical protocol shall proceed in accordance with NIOSH Method #7024.	OSHA & NIOSH: (Chromium II, III) 0.5 mg/m^3 (Chromium VI) 0.1 mg/m^3 (Ceiling) ACGIH: 0.5 mg/m^3 (Chromium II, III compounds), 0.05 mg/m^3 (Chromium VI compounds) IDLH: 30 mg/m^3 (Chromium VI compounds)	The use of a air purifying, full face-piece respirator with a high efficiency particulate filter for concentrations up to 0.1 mg/m^3. **Recommended Gloves:** This is in particulate form. Therefore any glove suitable to prevent skin contact.	**Boiling Pt:** 4788°F; 2642°C **Melting Pt:** 3452°F; 1900°C **Solubility:** Insoluble **Flash Pt:** Not applicable (Airborne dust may burn or explode when exposed to heat, flame, or incompatible chemicals) **LEL/LFL:** Not applicable **UEL/UFL:** Not applicable **Vapor Density:** Not available **Vapor Pressure:** 0 mmHg **Specific Gravity:** 7.14 **Incompatibilities:** Strong oxidizers, peroxides, and alkalis **Appearance and Odor:** Appearance and odor vary depending upon the specific compound.	Health hazards are characterized normally through chronic exposure manifesting as histologic fibrosis of the lungs and ulceration of the nasal septum and skin. IARC, NTP and ACGIH list various chromium compounds as possessing carcinogenic properties.
Copper	7440-50-8 (Cu) 1317-38-0 (CuO)	Substance is not volatile. Unable to be detected by PID or FID.	Air sample using a mixed cellulose ester filter; inductively coupled plasma/atomic emission spectroscopy. Sampling and analytical protocol shall proceed in accordance with NIOSH Method #7300.	NIOSH; OSHA: 0.10 mg/m^3 ACGIH: 0.2 mg/m^3	The use of an air-purifying full-face respirator with a high efficiency particulate air filter. **Recommended gloves:** This is in the particulate form. Therefore any glove suitable to prevent skin contact (Nitrile has been the one most widely used for the other substances).	**Boiling Pt:** 4215°F; 2324°C **Melting Pt:** 1981°F; 1083°C **Solubility:** Insoluble **Flash Pt:** Not applicable (Airborne dust may burn or explode when exposed to heat, flame, or incompatible chemicals) **LEL/LFL:** Not applicable **UEL/UFL:** Not applicable **Vapor Density:** Not available **Vapor Pressure:** 1 mmHg @ 2962°F 1628°C **Specific Gravity:** 8.94 **Incompatibilities:** Oxidizers, alkalis, sodium azide, acetylene, bromates, chlorates, iodates, and acids. **Appearance and Odor:** Metal: Reddish, lustrous malleable, odorless solid. Fume: Finely divided black particulate dispersed in air.	Irritation to the nose, throat, and respiratory tract. Metallic taste Discoloration of skin (potential dermatitis) and hair. Chronic exposure may result in dermatitis and damage to the liver and kidneys. Overexposure to fumes causes symptoms characteristic of the flu (headaches, chills, muscle aches, nausea, vomiting, diarrhea). Ingestion may cause burning in the mouth, throat, and stomach. Metallic taste with colicky abdominal pain. Individuals with Wilson's disease are at greater risk of chronic exposure as a result of the bodies tendency to absorb and retain copper.

TABLE 6-1

CHEMICAL, PHYSICAL, AND TOXICOLOGICAL DATA
NSWC CRANE, CRANE, INDIANA
PAGE 3

Substance		Air Monitoring/Sampling Information		Exposure Limits	Warning Property Rating	Physical Properties	Health Hazard Information
Lead	7439-92-1	Particulate form - Unable to be detected by either PID or FID.	Air sample using a mixed cellulose ester filter; or HNO_3 or H_2O_2 desorption; or Atomic absorption detection. NIOSH Method #7082 or #7300.	OSHA: 0.05 mg/m^3 ACGIH: 0.15 mg/m^3 NIOSH: 0.10 mg/m^3 IDLH: 100 mg/m^3 as lead	The use of a air purifying, full-face respirator with high efficiency particulate air filter for up to 2.5 mg/m^3. **Recommended gloves:** This is in the particulate form. Therefore any glove suitable to prevent skin contact (Nitrile has been the one most widely used for the other substances).	**Boiling Pt:** 3164°F; 1740°C **Melting Pt:** 621°F; 327°C **Solubility:** Insoluble **Flash Pt:** Not applicable (Airborne dust may burn or explode when exposed to heat, flame, or incompatible chemicals) **LEL/LFL:** Not applicable **UEL/UFL:** Not applicable **Vapor Density:** Not available **Vapor Pressure:** 0 mmHg **Specific Gravity:** 11.34 **Incompatibilities:** Strong oxidizers, peroxides, sodium acetylide, zirconium, and acids **Appearance and Odor:** Metal: A heavy ductile, soft gray solid.	Overexposure to this substance via ingestion or inhalation may result in metallic taste in the mouth, dry throat, thirst, Gastrointestinal disorders (burning stomach pain, nausea, vomiting, possible diarrhea sometimes bloody or black, accompanied by severe bouts of colic), CNS effects (muscular weakness, pain, cramps, headaches, insomnia, depression, partial paralysis possibly coma and death. Extended exposure may result in damage to the kidneys, gingival lead line, brain, and anemia.
Nickel	7440-02-0	Particulate form - This substance is unable to be detected by PID/FID.	Air sample using particulate filter; acid desorption; ICP detection. Sampling and analytical protocol shall proceed in accordance with NIOSH Method #7300.	OSHA: as Ni metal and insoluble compounds 1 mg/m^3 NIOSH: 0.015 mg/m^3 ACGIH: 0.05 mg/m^3 IDLH: 10 mg/m^3	No identifiable warning properties to indicate presence and thereby detection. **Recommended APR Cartridge:** Suitable for dust and fume. Organic vapor acid gases with HEPA filter. **Recommended gloves:** This material is in the particulate form. Therefore any glove suitable to prevent skin contact (Nitrile has been the one most widely used for the other substances).	**Boiling Pt:** 4946°F; 2730°C **Melting Pt:** 2651°F; 1455°C **Solubility:** Insoluble acid **Flash Pt:** Not available(Airborne dust may burn or explode when exposed to heat, flame, or incompatible chemicals) **LEL/LFL:** Not available **UEL/UFL:** Not available **Vapor Density:** Not available **Vapor Pressure:** 1 mmHg @ 3290°F; 1810°C **Specific Gravity:** 8.90 **Incompatibilities:** Strong acids, halogens, sulfur, wood and other combustibles, nickel nitrate, and oxidizers **Appearance and odor:** Silvery white, hard, malleable ductile metal, odorless	Symptoms of overexposure to this product may include headaches, vertigo, delirium, extreme weakness, GI disturbance and pain including nausea vomiting and diarrhea, coughing, hyperpnea, cyanosis, weakness, allergic dermatitis, nickel itch, pulmonary asthma, chest pains tightness, dyspnea, dry cough, and conjunctivitis. This substances has been identified as a Human carcinogen by NTP and IARC.

TABLE 6-1

CHEMICAL, PHYSICAL, AND TOXICOLOGICAL DATA
NSWC CRANE, CRANE, INDIANA
PAGE 4

Substance		Air Monitoring/Sampling Information		Exposure Limits	Warning Property Rating	Physical Properties	Health Hazard Information
Zinc	7440-66-6	Particulate form - This substance is not detectable using a PID or FID.	Air sample using a particulate filter; acid desorption; AAS detection. Sampling and analytical protocol will proceed in accordance with NIOSH Method #7300.	OSHA: 10 mg/m^3 Total dust, 5 mg/m^3 Respirable fraction NIOSH: 5 mg/m^3 15 mg/m^3 (Ceiling) ACGIH: 10 mg/m^3	No identifiable warning properties to indicate presence and thereby detection. **Recommended APR Cartridge:** Suitable for dust and fume. Organic vapor acid gases with HEPA filter. **Recommended gloves:** This is in the particulate form. Therefore any glove suitable to prevent skin contact (Nitrile has been the one most widely used for the other substances).	**Boiling Pt:** 1666°F; 908°C **Melting Pt:** 788°F; 419.8°C **Solubility:** Insoluble **Flash Pt:** Not available (Airborne dust may burn or explode when exposed to heat, flame, or incompatible chemicals) **LEL/LFL:** Not available **UEL/UFL:** Not available **Vapor Density:** Not available **Vapor Pressure:** 0 mmHg **Specific Gravity:** 7.14 **Incompatibilities:** Strong acids, halogens, catalytic metals, combustibles, oxidizers, nitryl fluoride **Appearance and odor:** Bluish-white, lustrous metal, odorless	Inhalation of fumes may result in metal fume fever. This condition is characterized by metallic taste, dryness of the throat, coughing with generalized aching and flu-like symptoms. Effects through ingestion may include coughing, difficulty in breathing, and sweating. A human skin irritant. Irritation to the eyes may result from mechanical action.
Cyanides (as CN)	varies depending of compound	No information found.	Air sample using filter; analyze using specific ion electrode. Sampling and analytical protocol in accordance with established methodology.	OSHA; ACGIH; NIOSH: 5 mg/m^3 (ceiling) IDLH: 50 mg/m^3	Inadequate warning properties; recommend the use of supplied air respirators. **Recommended Gloves:** Butyl 1.00 hr.	**Boiling Pt:** 2725°F; 1496°C **Melting Pt:** 1047°F; 564°C **Solubility:** 58% **Flash Pt:** Not available **LEL/LFL:** Not available **UEL/UFL:** Not available **Vapor Density:** Not available **Vapor Pressure:** 0 mmHg **Specific Gravity:** 1.6 **Incompatibilities:** Strong oxidizers, acids, acid salts, chlorates, and nitrates **Appearance and odor:** KCN and NaCN are white granular or crystalline solids with a faint almond-like odor	Overexposure may result in chemical asphyxiation and death. Symptoms of exposure include weakness, headache, confusion, nausea, vomiting, increased respiratory rate, slow gasping respirations, irritation of the eyes and skin. Target organs are listed as Cardiovascular system, Central nervous system, liver, kidneys, and skin.

7.0 AIR MONITORING

Monitoring devices such as Direct Reading Instruments (DRIs), will be employed at the site to detect and evaluate the presence of site contaminants and other potentially harmful agents. The specific type of monitoring and the associated instruments, frequency of use, and applicable action levels are dependent upon the specific scope of work and the contaminants of concern. As a result, specific air monitoring measures and requirements have been established in Table 5-1 of this site specific HASP. Additionally, Section 1.0 of the TtNUS Health and Safety Guidance Manual contains detailed information regarding direct reading instrumentation, personal and area air sampling procedures, as well as general calibration procedures of various instruments.

8.0 TRAINING/MEDICAL SURVEILLANCE REQUIREMENTS

8.1 INTRODUCTORY/REFRESHER/SUPERVISORY TRAINING

8.1.1 Requirements for TtNUS Personnel

All TtNUS personnel must complete 40 hours of introductory hazardous waste site training in accordance with 29 CFR 1910.120(e) prior to performing work at NSWC Crane. Additionally, TtNUS personnel who have had introductory training more than 12 months prior to site work must have completed 8 hours of refresher training within the past 12 months before being cleared for site work. 8-hour Supervisory Training in accordance with 29 CFR 1910.120(e)(4) will be required for site supervisory personnel.

Documentation of TtNUS Health and Safety Training will be maintained at the project site. Copies of certificates or other official documentation will be used to fulfill this requirement.

At the request of the U.S. Navy, TtNUS will conduct a brief meeting daily to discuss operations planned for that day. At the end of the workday, a short meeting will be held to discuss the operations completed and any problems that were encountered.

8.1.2 Requirements for Subcontractors

All TtNUS subcontractor personnel must have completed introductory hazardous waste site training or equivalent work experience as defined in OSHA Standard 29 CFR 1910.120(e) and 8 hours of refresher training meeting the requirements of 29 CFR 1910.120(e)(8) prior to performing field work at NSWC Crane. TtNUS subcontractors must certify that each employee has had such training by sending TtNUS a letter, on company letterhead, containing the information in the example letter provided in Figure 8-1, and providing copies of all training certificates.

8.2 SITE-SPECIFIC TRAINING

TtNUS will provide site-specific training to all TtNUS employees and subcontractor personnel who will perform work on this project. Site-specific training will also be provided to all site visitors (DOD, EPA, etc.) who may enter the site to perform functions that may or may not be directly related to site operations. Site-specific training will include:

FIGURE 8-1

TRAINING LETTER

The following statements must be typed on company letterhead and signed by an officer of the company and accompany copies of associated training certificates:

LOGO
XYZ CORPORATION
555 E. 5th Street
Nowheresville, Kansas 55555

Month, day, year

Roger A. Clark, Ph.D.
Project Manager
Brown & Root Environmental
Foster Plaza VII 661 Andersen Drive
Pittsburgh, Pennsylvania 15220

Subject: HAZWOPER Training for NSWC Crane

Dear Dr. Clark:

As an officer of XYZ Corporation, I hereby state that I am aware of potential hazardous nature of the subject project. I also understand that is out responsibility to comply with all applicable occupational safety and health regulations including those stipulated in Title 29 of the Code of Federal Regulations (CFR), Parts 1900 through 1910 and Part 126.

I also understand that Title 29 CFR 1910.120 entitled "Hazardous Waste Operations and Emergency Response" requires appropriate level of training for certain employees engaged in hazardous waste operations. In this regard, I hereby state that the following employees have had 40 hours of introductory hazardous waste site training or equivalent work experience as requested by 29 CFR 1910.120(e) and have had 8 hours of refresher training as required by 29 CFR 1910.120(e)(8),

LIST FULL NAMES OF EMPLOYEES AND THEIR SOCIAL SECURITY NUMBERS HERE

Should you have any questions, please contact me at (555) 555-5555.

Sincerely,

(Name of Company Officer)

- Names of personnel and alternates responsible for site safety and health
- Safety, health and other hazards present on site
- Use of personal protective equipment
- Work practices to minimize risks from hazards
- Safe use of engineering controls and equipment
- Medical surveillance requirements
- Signs and symptoms of overexposure to site contaminants
- The contents of the site-specific health and safety plan including the contents of Table 5-1 and 6-1.
- Emergency response procedures (evacuation and assembly points)
- Spill response procedures
- Review the contents of relevant Material Safety Data Sheets

Site-specific documentation will be established through the use of Figure 8-2. All site personnel and visitors must sign this document upon receiving site-specific training prior to commencement of site activities.

8.3 MEDICAL SURVEILLANCE

8.3.1 Medical Surveillance Requirements For TtNUS Personnel

All TtNUS personnel participating in project field activities will have had a physical examination meeting the requirements of TtNUS' medical surveillance program and will be medically qualified to perform hazardous waste site work using respiratory protection.

Documentation for medical clearances will be maintained in the TtNUS Pittsburgh office and made available as necessary.

8.3.2 Medical Surveillance Requirements For Subcontractors

Subcontractor personnel are required to obtain a certificate of their ability to perform hazardous waste site work and to wear respiratory protection. The "Subcontractor Medical Approval Form" provided in Figure 8-3 of this HASP shall be used to satisfy this requirement providing it is properly completed and signed by a licensed physician.

FIGURE 8-2

SITE-SPECIFIC TRAINING DOCUMENTATION

My signature below indicates that I am aware of the potential hazardous nature of performing investigation activities at NSWC Crane, and that I have received site-specific training which included the elements presented below:

- Names of personnel and alternates responsible for site safety and health
- Safety, health and other hazards present on site
- Use of personal protective equipment
- Work practices to minimize risks from hazards
- Safe use of engineering controls and equipment
- Medical surveillance requirements
- Signs and symptoms of overexposure
- The contents of the health and safety plan including Table 5-1 and 6-1
- Emergency response procedures (evacuation and assembly points)
- Review contents of relevant Material Safety Data Sheets and Chemical Data Sheets / Hazard Lines
- Spill Response

I further state that I have been given the opportunity to ask questions and that all of my questions have been answered to my satisfaction.

I further state by the presence of my signature below the date of my training (introductory, refresher, and supervisory, as applicable), and my medical surveillance requirements are accurate and correct to the best of my knowledge.

Name	40-Hour General Site Worker Training (Date)	8-Hour Refresher Training (Date)	8-Hour Supervisory Training (Date)	Medical Surveillance	SIGNATURE

Subcontractors who have a company medical surveillance program meeting the requirements of paragraph (f) of OSHA 29 CFR 1910.120 can substitute "Subcontractor Medical Approval Form" with a letter, on company letterhead, containing all of the information in the example letter presented in Figure 8-4.

8.3.3 Requirements For All Field Personnel

Each field team member (including subcontractors and visitors entering the exclusion zone) shall be required to complete and submit a copy of the Medical Data Sheet found in the TtNUS Health and Safety Guidance Manual. This shall be provided to the SSO prior to participating in site activities. The purpose of this document is to provide site personnel and emergency responders with additional information that may be necessary in order to administer medical attention.

8.4 SUBCONTRACTOR EXCEPTION

Subcontractors who will not enter the exclusion zone during operation, and whose activities involve no potential for exposure to site contaminants, will not be required to meet the requirements for training/medical surveillance, other than site-specific training as stipulated in Section 8-2.

FIGURE 8-3

SUBCONTRACTOR MEDICAL APPROVAL FORM

For employees of_____

Company Name

Participant Name: _____ Date of Exam: _____

Part A

The above-named individual has:

1. Undergone a physical examination in accordance with OSHA Standard 29 CFR 1910.120, paragraph (f) and found to be medically -

( ) qualified to perform work at the NSWC Crane work site
( ) not qualified to perform work at the NSWC Crane work site

and,

2. Undergone a physical examination as per OSHA 29 CFR 1910.134(b)(10) and found to be medically -

( ) qualified to wear respiratory protection
( ) not qualified to wear respiratory protection

My evaluation has been based on the following information, as provided to me by the employer.

( ) A copy of OSHA Standard 29 CFR 1910.120 and appendices.
( ) A description of the employee's duties as they relate to the employee's exposures.
( ) A list of known/suspected contaminants and their concentrations (if known).
( ) A description of any personal protective equipment used or to be used.
( ) Information from previous medical examinations of the employee which is not readily available to the examining physician.

Part B

I, _____, have examined _____

Physician's Name (print) Participant's Name (print)

and have determined the following information:

FIGURE 8-3

SUBCONTRACTOR MEDICAL APPROVAL FORM
PAGE TWO

1. Results of the medical examination and tests (excluding finding or diagnoses unrelated to occupational exposure):

_____

_____

_____

2. Any detected medical conditions which would place the employee at increased risk of material impairment of the employee's health:

_____

_____

_____

3. Recommended limitations upon the employee's assigned work:

_____

_____

_____

I have informed this participant of the results of this medical examination and any medical conditions which require further examination of treatment.

Based on the information provided to me, and in view of the activities and hazard potentials involved at the NSWC Crane work site, this participant

( ) may

( ) may not

perform his/her assigned task.

Physician's Signature _____

Address _____

Phone Number _____

NOTE: Copies of test results are maintained and available at:

_____

Address

FIGURE 8-4

MEDICAL SURVEILLANCE LETTER

The following statements must be typed on company letterhead and signed by an officer of the company:

LOGO
XYZ CORPORATION
555 E. 5th Street
Nowheresville, Kansas 55555

Month, day, year

Roger A. Clark, Ph.D.
Project Manager
TtNUS Corp.
Foster Plaza VII 661 Andersen Drive
Pittsburgh, Pennsylvania 15220

Subject: Medical Surveillance for NSWC Crane

Dear Dr. Clark:

As an officer of XYZ Corporation, I hereby state that the persons listed below participate in a medical surveillance program meeting the requirements contained in paragraph (f) of Title 29 of the Code of Federal Regulations (CFR), Part 1910.120 entitled "Hazardous Waste Operations and Emergency Response" I further state that the persons listed below have had physical examinations under this program within the past 12 months and that they have been cleared, by a licensed physician, to perform hazardous waste site work and to wear positive and negative pressure respiratory protection. I also state that, to my knowledge, no person listed below has any medical restriction that would preclude him/her from working at the NSWC Crane Site.

LIST FULL NAMES OF EMPLOYEES AND THEIR SOCIAL SECURITY NUMBERS HERE

Should you have any questions, please contact me at (555) 555-5555.

Sincerely,

(Name of Company Officer)

9.0 SITE CONTROL

This Section outlines the means by which TtNUS will delineate work zones and use these work zones in conjunction with decontamination procedures to prevent the spread of contaminants into previously unaffected areas of the site. It is anticipated that a three zone approach will be used during work at this site; an exclusion zone, a contamination reduction zone, and a support zone. It is also anticipated that this control measure will be used to control access to the work areas, thereby restricting the general public to avoid the spread of contaminants, and to protect individuals who are not cleared to enter by way of training and/or medical surveillance qualifications.

9.1 EXCLUSION ZONE

The exclusion zone will be considered those areas of the site of known or suspected contamination. It is not anticipated that significant amounts of surface contamination are in the proposed work areas of this site. It is also anticipated that this will remain so until/unless contaminants are brought to the surface by intrusive activities. Furthermore, once intrusive activities have been completed and surface contamination has been removed, the potential for exposure is again diminished and the area can then be reclassified as part of the contamination reduction zone. Therefore, the exclusion zones for this project will be limited to those areas of the site where active work (groundwater sampling) is being performed plus a safety zone of 15 feet surrounding the point of operation. All exclusion zones will be delineated using barrier tape, cones and/or drive poles, and postings to inform and direct site personnel.

9.2 CONTAMINATION REDUCTION ZONE

The contamination reduction zone (CRZ) will be the buffer area between the exclusion zone and any of the site where contamination is not suspected. Personnel and equipment decontamination can take place in this area if a central decontamination location is not established for this project. This zone will also serve as a focal point in supporting exclusion zone activities. This area will be delineated using barrier tape, cones and/or drive poles, and postings to inform and direct site personnel.

9.3 SUPPORT ZONE

The support zone for this project will include a staging area where site vehicles will be parked, equipment will be unloaded, and where food and drink containers will be maintained. In all cases, the support zones will be established at areas of the site where exposure to site contaminants would not be expected during normal working conditions or foreseeable emergencies.

9.4 SITE MAP

Once the areas of contamination, access routes, topography, dispersion routes are determined, a site map will be generated and adjusted as site conditions change. These maps will be posted to illustrate up-to-date information of contaminants and adjustment of zones and access points.

9.5 BUDDY SYSTEM

Personnel engaged in on-site activities will practice the "buddy system" to insure the safety of all personnel involved in this operation.

9.6 MATERIAL SAFETY DATA SHEET (MSDS) REQUIREMENTS

TtNUS personnel will provide MSDS's for all chemicals brought on-site. The contents of these documents will be reviewed by the SSO with the user(s) of the chemical substances prior to any actual use or application or the substances on-site. The MSDS's will be maintained in a central location (i.e., temporary office) and will be available for anyone to review upon request.

9.7 COMMUNICATION

It is anticipated that site personnel will be working in close proximity during proposed field activities. In the event that site personnel are in isolated areas or are separated by significant distances, a supported means of communication between field crews will be utilized. Two-way radio communication devices, if needed, will be submitted for NSWC Crane approval.

External communications will be done so utilizing the telephones at predetermined and approved locations or through cellular phones. External communication will primarily be used for the purpose of resource and emergency resource communications. Prior to the commencement of site activities, the FOL will determine and arrange for telephone communications, if it is determined a cellular means will not be used.

9.8 SITE VISITORS

Site visitors for the purpose of this document are identified as representing the following groups of individuals:

- Personnel invited to observe or participate in operations by TtNUS.
- Regulatory personnel (i.e., DOD, EPA, OSHA, etc.)
- Southern Division Navy personnel
- Other authorized visitors

All non DOD personnel working on this project are required to gain initial access to the base by coordinating with our Task Order Manager or designee and following established base access procedures.

Once access to the base is obtained, all personnel who require site (areas of ongoing operations) access will be required to obtain permission from the FOL and the Base Contact. Upon gaining access to the site, all site visitors wishing to observe operations in progress will be required to meet the minimum requirements as stipulated below.

- All site visitors will be routed to the FOL, who will sign them into the field logbook. Information to be recorded in the logbook will include the individuals name (proper identification required), who they represent, and the purpose for the visit.

- All site visitors will be required to produce the necessary information supporting clearance on to the site. This includes information attesting to applicable training (40-hours of HAZWOPER training required for all Southern Division Navy Personnel), and medical surveillance as stipulated in Section 8.3, of this document. In addition, to enter the sites operational zones during planned activities, all visitors will be required to first go through site-specific training covering the topics stipulated in Section 8.2 of this HASP.

Once the site visitors have completed the above items they will be permitted to enter the site and applicable operational areas. All visitors are required to observe the protective equipment and site restrictions in effect at the site. Any and all visitors not meeting the requirements as stipulated in this plan for site clearance will not be permitted to enter the site operational zones during planned activities. Any incidence of unauthorized site visitation will cause all on-site activities to be terminated until that visitor can be removed. Removal of unauthorized visitors will be accomplished with support form the Base Contact, if necessary. At a minimum, the Base Contact will be notified of any unauthorized visitors.

9.9 SITE SECURITY

Site security will be accomplished using field personnel. This is a multiple person operation, involving multiple operational zones. TtNUS personnel will retain complete control over active operational zones.

As this activity will take place at a Navy facility, the first line of security will take place at the base gate restricting the general public. The second line of security will take place at the work site referring interested parties to the FOL and Base Contact.

The Base Contact will serve as the focal point for base personnel and interested parties and will serve as the final line of security and the primary enforcement contact.

10.0 SPILL CONTAINMENT PROGRAM

10.1 SCOPE AND APPLICATION

It is not anticipated that bulk hazardous materials (over 55-gallons) will be handled at any given time as part of the scope of work. Small (less than 5 gallons) of generator fuel, either gasoline or diesel fuel, may be used and stored on-site and constitute a minor spill hazard. It is also not anticipated that spillage of any stored materials would constitute a danger to human health or the environment. However, it is possible that as the job progresses, the potential for accumulating purge water and decontamination fluids in the central staging area exists. Once these fluids are characterized they can be removed from the staging area and disposed of. As these fluids remain uncharacterized while in the staging area, and given the potential presence of minor quantities of fuel on-site, a spill containment program will be developed and instituted as part of this HASP.

10.2 POTENTIAL SPILL AREAS

Potential spill areas will be monitored in an ongoing attempt to prevent and control further potential contamination of the environment. Fuels will be handled and transferred where necessary to accommodate operation of the generators. There are limited areas vulnerable to other spill hazards, including areas that may be used for central staging and decontamination activities. Additionally, areas designated for handling, loading, and unloading of potentially contaminated waters present limited potential for leaks or spills. At this junction, it is anticipated that all purge water and decontamination fluids generated as a result of this scope of work, will be containerized, labeled, and staged to await chemical analyses. The results of these analyses will determine appropriate disposal methods. During the turn around for the chemical analyses there exists a possibility to accumulate multiple drums of fluids.

10.3 LEAK AND SPILL DETECTION

To establish an early detection of potential spills or leaks, a periodic walk around of the mobile and fixed holding tank will be conducted during working hours to visually determine that containers are not leaking. Fuel storage containers will be inspected periodically and during each use for signs of leakage or potential leakage. If a leak is detected in the holding tank, the contents of the tank will be transferred into a new holding tank using a submersible pump. If a leak is detected in a fuel storage container, the contents of the container will be transferred into a similar container approved for fuel storage. Leaked material will be collected and contained using absorbents such as Oil-dry, vermiculite, or sand, which will be stored at the

staging area in a drum conspicuously marked. This used material, too, will be containerized for disposal pending analyses. All inspections will be documented in the Project Logbook.

All Investigation-Derived Waste (IDW) including primarily purge water generated during sampling activities will be containerized into a 300- and/or 2,100-gallon plastic holding tank.

10.4 PERSONNEL TRAINING AND SPILL PREVENTION

All personnel will be instructed on the procedures for spill prevention, containment, and collection of hazardous materials in the site-specific training. The FOL and/or the SSO will serve as the Spill Response Coordinator for this operation should the need arise.

10.5 SPILL PREVENTION AND CONTAINMENT EQUIPMENT

The following represents the minimum equipment which will be maintained at the staging area at all times for the purpose of supporting this Spill Prevention/Containment Program.

- Sand, clean fill, vermiculite, or other noncombustible absorbent (oil-dry)
- Drums (55-gallon U.S. DOT 17-E or 17-H) and an empty 5-gal. container approved to store fuels
- Shovels, rakes, and brooms
- Hand operated drum pump with hose
- Labels
- Drum Patch kit

10.6 SPILL CONTROL PLAN

This section describes the procedures the TtNUS field crew members will employ upon the detection of a spill or leak.

1) Notify the SSO or FOL immediately upon the detection of a leak or spill. The SSO or FOL will then immediately notify the Base Contact at x-3114 or 6160.

2) Employ the personnel protective equipment stored at the staging area. Take immediate actions to stop the leak or spill by plugging or patching the container or raising the leak to the highest point. Spread the absorbent material in the area of the spill covering completely.

3) Transfer the material to a new container, collect and containerize the absorbent material. Label the new container appropriately. Await analyses for treatment or disposal options.

It is not anticipated that a spill will occur in which the field crews cannot handle. Should this occur notification of appropriate emergency response agencies will be carried out by the FOL or SSO.

11.0 CONFINED SPACE ENTRY

Personnel under the provisions of this HASP are not allowed, under any circumstances, to enter confined spaces. A confined space is defined as an area which has one or more of the following characteristics:

- Is large enough and so configured that an employee can bodily enter and perform assigned work.
- Has limited or restricted means for entry or exit (for example, tanks, vessels, silos, storage bins, hoppers, vaults, and pits are spaces that may have limited means of entry).
- Is not designed for continuous employee occupancy.
- Contains or has a potential to contain a hazardous atmosphere.
- Contains a material that has the potential to engulf an entrant.
- Has an internal configuration such that an entrant could be trapped or asphyxiated by inwardly converging walls or by a floor which slopes downward and tapers to a smaller cross-section.
- Contains any other recognized, serious, safety or health hazard.

For further information on confined space consult the Health and Safety Guidance Manual or call the Manager, Health Sciences. If confined space operations are to be performed as part of the scope of work, detailed procedures and training requirements will be addressed in an addendum or the site specific health and safety plan.

12.0 MATERIALS AND DOCUMENTS

The TtNUS Filed Operations Leader (FOL) shall ensure the following materials/documents are taken to the project site and utilized as required.

- Incident Reports
- Supervisor's Accident Report
- Medical Data Sheets
- Material Safety Data Sheets for decon solutions and other substances brought to the site
- Follow-Up Reports (to be completed by the Field Team Leader)
- OSHA Job Safety and Health Poster (posted at the site)
- Training/Medical Surveillance Documentation Form (Blank)
- OSHA 29 CFR 1910.120 (HAZWOP) training certificates
- First Aid Supply Usage Form
- Emergency Reference Form (Section 2.0, extra copy for posting)
- Health and Safety Guidance Manual

13.0 GLOSSARY

ACGIH	American Conference of Governmental Industrial Hygienists
APR	Air Purifying Respirators
ASP	Associate Safety Professional
B&R	Brown & Root Environmental
CFR	Code of Federal Regulations
CIH	Certified Industrial Hygienist
CLEAN	Comprehensive Long-Term Environmental Action Navy
CNS	Central Nervous System
CQP	Construction Quality Plan
CSP	Certified Safety Professional
CTO	Contract Task Order
DRI	Direct Reading Instruments
eV	electron Volts
FID	Flame Ionization Detector
FOL	Field Operations Leader
HSGM	Health and Safety Guidance Manual
HASP	Health and Safety Plan
HAZWOPER	Hazardous Waste Operations and Emergency Response
HEPA	High Efficiency Particulate Air
HSM	Health and Safety Manager
IDLH	Immediately Dangerous to Life and Health
N/A	Not Available
NIOSH	National Institute Occupational Safety and Health
NSWC	Naval Surface Warfare Center
OSHA	Occupational Safety and Health Administration (U.S. Department of Labor)
PEL	Permissible Exposure Limit
PHSO	Project Health and Safety Manager
PID	Photo Ionization Detector
PM	Project Manager
PPE	Personal Protective Equipment
PPM	Parts Per Million
PVC	Poly Vinyl Chloride
SAP	Sampling and Analyses Plan

SCBA	Self Contained Breathing Apparatus
SSO	Site Safety Officer
STEL	Short Term Exposure Limit
SWMU	Solid Waste Management Unit
TOM	Task Order Manager
TWA	Time Weighted Average
UV	Ultra Violet
WP	Work Plan

ATTACHMENT 1
OSHA POSTER

JOB SAFETY & HEALTH PROTECTION

The Occupational Safety and Health Act of 1970 provides job safety and health protection for workers by promoting safe and healthful working conditions throughout the Nation. Requirements of the Act include the following:

Employers

All employers must furnish to employees employment and a place of employment free from recognized hazards that are causing or are likely to cause death or serious harm to employees. Employers must comply with occupational safety and health standards issued under the Act.

Employees

Employees must comply with all occupational safety and health standards, rules, regulations and orders issued under the Act that apply to their own actions and conduct on the job.

The Occupational Safety and Health Administration (OSHA) of the U.S. Department of Labor has the primary responsibility for administering the Act. OSHA issues occupational safety and health standards, and its Compliance Safety and Health Officers conduct jobsite inspections to help ensure compliance with the Act.

Inspection

The Act requires that a representative of the employer and a representative authorized by the employees be given an opportunity to accompany the OSHA inspector for the purpose of aiding the inspection.

Where there is no authorized employee representative, the OSHA Compliance Officer must consult with a reasonable number of employees concerning safety and health conditions in the workplace.

Complaint

Employees or their representatives have the right to file a complaint with the nearest OSHA office requesting an inspection if they believe unsafe or unhealthful conditions exist in their workplace. OSHA will withhold, on request, names of employees complaining.

The Act provides that employees may not be discharged or discriminated against in any way for filing safety and health complaints or for otherwise exercising their rights under the Act.

Employees who believe they have been discriminated against may file a complaint with their nearest OSHA office within 30 days of the alleged discrimination.

Citation

If upon inspection OSHA believes an employer has violated the Act, a citation alleging such violations will be issued to the employer. Each citation will specify a time period within which the alleged violation mu be corrected

The OSHA citation must be prominently displayed at or near the plac of alleged violation for three days, or until it is corrected, whichever is later, to warn employees of dangers that may exist there.

Proposed Penalty

The Act provides for mandatory penalties against employers of up to $1,000 for each serious violation and for optional penalties of up to $1,000 for each nonserious violation. Penalties of up to $1,000 per da may be proposed for failure to correct violations within the proposed t period. Also, any employer who willfully or repeatedly violates the Act be assessed penalties of up to $10,000 for each such violation.

Criminal penalties are also provided for in the Act. Any willful violat resulting in death of an employee, upon conviction, is punishable by a of not more than $10,000, or by imprisonment for not more than six months, or by both. Conviction of an employer after a first conviction doubles these maximum penalties.

Voluntary Activity

While providing penalties for violations, the Act also encourages efforts labor and management, before an OSHA inspection, to reduce workplac hazards voluntarily and to develop and improve safety and health progr in all workplaces and industries. OSHA's Voluntary Protection Program recognize outstanding efforts of this nature.

Such voluntary action should initially focus on the identification and elimination of hazards that could cause death, injury, or illness to employees and supervisors. There are many public and private organizations that can provide information and assistance in this effort, requested. Also, your local OSHA office can provide considerable help advice on solving safety and health problems or can refer you to other sources for help such as training.

Consultation

Free consultative assistance, without citation or penalty, is available to employers, on request, through OSHA supported programs in most Sta departments of labor or health

More Information

Additional information and copies of the Act, specific OSHA safety and health standards, and other applicable regulations may be obtained from your employer or from the nearest OSHA Regional Office in the following locations:

Atlanta, Georgia
Boston, Massachusetts
Chicago, Illinois
Dallas, Texas
Denver, Colorado
Kansas City, Missouri
New York, New York
Philadelphia, Pennsylvania
San Francisco, California
Seattle, Washington

Telephone numbers for these offices, and additional area office locations, are listed in the telephone directory under the United States Department of Labor in the United States Government listing.

Washington, D.C.
1985
OSHA 2203

William E. Brock, Secretary of Labor

U.S. Department of Labo
Occupational Safety and Health Administrat

Under provisions of Title 29, Code of Federal Regulations, Part 1903.2(a)(1) employers must post this notice (or a facsimile) in a conspicuous place where notices to employees are customarily posted.

ATTACHMENT 2
MEDICAL DATA SHEET

MEDICAL DATA SHEET

This brief Medical Data Sheet will be completed by all on-site personnel and will be kept in the command post during the conduct of site operations. This data sheet will accompany any personnel when medical assistance is needed or if transport to hospital facilities is required.

Project _____

Name _____ Home Telephone _____

Address _____

Age _____ Height _____ Weight _____

Name of Next Kin _____

Drug or other Allergies _____

Particular Sensitivities _____

Do You Wear Contacts? _____

Provide a Checklist of Previous Illnesses or Exposure to Hazardous Chemicals _____

_____

_____

What medications are you presently using? _____

_____

_____

Do you have any medical restrictions? _____

_____

_____

Name, Address, and Phone Number of personal physician: _____

_____

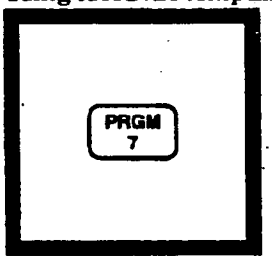
_____

I am the individual described above. I have read and understand this HASP.

_____ _____

Signature Date

Using Accuvac Ampuls



1. Enter the stored program number for hexavalent chromium (Cr^{6+})- AccuVac Ampuls.

Press: **PRGM**

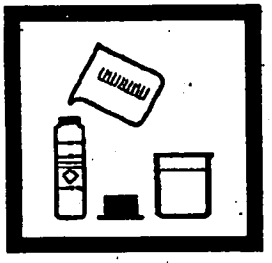
The display will show:

PRGM ?

2. Press: **14 ENTER**

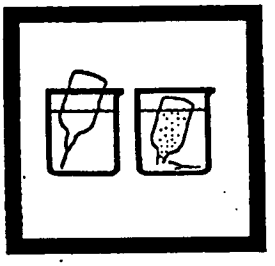
The display will show mg/L, Cr6 and the **ZERO** icon.

Note: For alternate forms (CrO_4, Cr_2O_7), press the **CONC** *key.*



3. Fill a sample cell with at least 10 mL of sample (the blank). Collect at least 40 mL of sample in a 50-mL beaker.

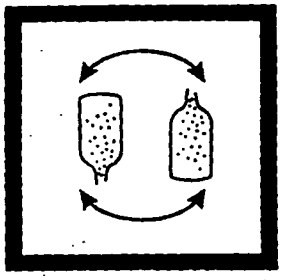
Note: For turbid samples, add the contents of one Acid Reagent Powder Pillow to 10 mL of the blank. This ensures turbidity dissolved by the acid in the ChromaVer 3 Chromium Reagent is also dissolved in the blank.



4. Fill a ChromaVer 3 Reagent AccuVac Ampul (the prepared sample) with sample.

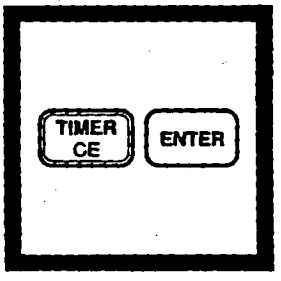
Note: Keep the tip immersed while the ampul fills completely.

Note: ChromaVer 3 should be white to tan in color. Replace if it is brown or green.



5. Quickly invert the ampul several times to mix. Wipe off any liquid or fingerprints.

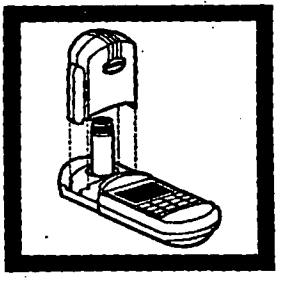
Note: A purple color will form if hexavalent chromium is present.



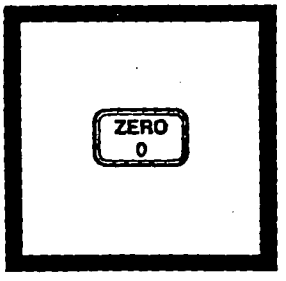
6. Press:

TIMER ENTER

A five-minute reaction period will begin.



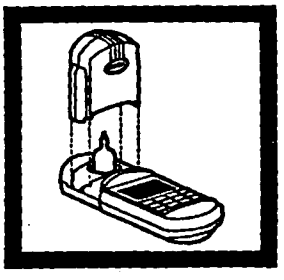
7. When the timer beeps place the blank into the cell holder.



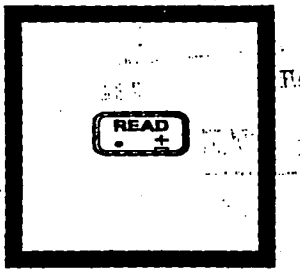
8. Press: **ZERO**

The cursor will move to the right, then the display will show:

0.00 mg/L Cr6



9. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



10. Press: **READ**

The cursor will move to the right, then the result in mg/L hexavalent chromium will be displayed.

Note: Standard Adjust may be performed using a prepared standard (see Standard Adjust in Section 1).

APPENDIX F

SLUDGE SAMPLING ADDENDUM

PHASE II GROUND WATER RCRA FACILITY INVESTIGATION
SOLID WASTE MANAGEMENT UNIT 30 (LANDFARM)
QUALITY ASSURANCE PROJECT PLAN ADDENDUM FOR SOIL/SLUDGE SAMPLING AND RISK SCREENING

Five rounds of groundwater data were collected at the Landfarm between October 1999 and October 2000. Results of these data have shown that no statistically significant impacts to groundwater have occurred from this SWMU.

Based on the results of groundwater data, Tetra Tech NUS (TtNUS) prepared the Draft Phase II RCRA Facility Investigation Report for Solid Waste Management Unit (SWMU) #30 (Landfarm) dated May 2001. The Navy requested no further action at this SWMU for all media. The U.S. EPA responded to this request on October 30, 2001, recommending that the Navy proceed with drafting a risk screening document for this SWMU as part of an official no further action request (as was done for previous NSWC Crane SWMUs such as the Lithium Battery Site). As requested by the U.S. EPA, this risk screening should be conducted using the Technical Guidance on Corrective Action Activity, Naval Surface Warfare Center Crane, Indiana dated August 1999. The U.S. EPA also recommended that the Navy collect and analyze soil/sludge samples from the Landfarm in order to identify any potential risks that may be present in the soil/sludge exposure pathway. They further suggested that samples should be collected from the surface soil (0 to 1 foot); tested for total metals; and these results compared to the Basewide Background Soil Investigation levels and risk-based screening levels for soils (human health and ecological). They further added that this would determine what, if any, institutional and/or engineering controls might be necessary for the SWMU should soil/sludge sampling show elevated levels of constituents.

A conference call was conducted on November 8, 2001 to discuss the proposed additional activities for the Landfarm. The following persons were present during the conference call:

Bill Gates (Remedial Project Manager-SOUTHDIV)
Christine Freeman (NSWC Crane)
Peter Ramanauskas (Project Manager-U.S. EPA Region 5)
Roger Clark (Task Order Manager- TtNUS)
LeeAnn Sinagoga (Department Manager Risk Assessment-TtNUS)
Mark Francis (Deputy Task Order Manager- TtNUS)

The following summarizes EPA's recommendations from this conference call:

- No additional risk screening is required for groundwater.
- Prepare a streamlined soil sampling Work Plan Addendum referencing previously approved NSWC Crane QAPPs.
- List the types, number, locations and depths of samples to be collected.
- If possible, make visual distinction between sludge and soil in the field.
- Analyze only for metals (F006) and any other metals related to F006 including cyanide.
- Recommended sampling depths of surface (0 to 1-foot bgs) and shallow subsurface (1 to 4 feet bgs).
- Incorporate grid and/or random sampling methods.
- Compare results to Risk Based Screening Levels and Background Values.
- Use both industrial and residential values for comparison.
- May want to perform intermediate risk assessment, which would fall between a quantitative and a qualitative screening.
- The assessment will need to also address ecological factors.

Based on the above-listed recommendations, the following activities are proposed to address these comments.

SAMPLING RATIONALE

This section describes the sampling rationale to be incorporated for the soil/sludge sampling at the Landfarm. In an attempt to expedite the fieldwork at this SWMU, similar field collection and analysis procedures that were recently conducted at an adjacent SWMU will be incorporated where possible. Fieldwork at an adjacent SWMU was recently conducted at the Old Rifle Range (SWMU 7) that is also located at NSWC Crane. The field activities at the Old Rifle Range (ORR) consist of collection and analysis of surface and shallow subsurface soil samples. This work is being conducted under a recently U.S. EPA-approved Quality Assurance Project Plan (QAPP) dated October 2000. Where possible, field

procedures including sampling methodologies referenced in the ORR QAPP will used in the current Work Plan Addendum for the Landfarm.

SOIL SAMPLING LOCATIONS, ANALYSES, AND RATIONALE

This section presents sampling locations, QA samples to be collected, analyses to be performed, and rationale for the sampling and analytical program. Details regarding the equipment and procedures for collecting, preserving, packaging, and shipping the samples are included in Section 4.0 and related SOPs in Appendix B of the QAPP for the ORR at Crane (TtNUS, October 2000).

Wastewater treatment plant sludges were applied at SWMU 30 (Landfarm) from November 1988 through March 1995. Although laboratory analyses were performed on these sludges, the only available data are from January 1990 through July 1992 (refer to Tables1-1A & 1-1B of the Landfarm Work Plan, TtNUS, August 1999). The primary constituents of concern are several metals such as those cited as the reason that some wastewater treatment plant sludges from electroplating operations are an F006 listed hazardous waste (e.g., cadmium, chromium, and nickel). [These metals have been detected in sludge samples (1990, 1991) for SWMU 30.] Based on existing site information, wastewater treatment plant sludges were not preferentially applied to any particular sub-area of SWMU 30. The following surface and subsurface soil sampling activities are intended to determine whether significant quantities of waste materials (metals) have accumulated in the soils as a result of the sludge application.

Soil sampling locations at SWMU 30 will be selected as follows. SWMU 30 will be divided into 12 sub-areas as shown on the attached figure (each sub-area will represent approximately 9,000 square feet). Each sub-area will be divided further into a 10-by-10 array of subcells (i.e., 100 sub-cells approximately 90 square feet each). Five subcells within each sub-area will be selected as borehole sampling locations (the borehole will be advanced at the approximate center point of the sub-cell). Sub-cells to be sampled will be randomly selected using a uniform random number generator [Microsoft Excel 97 SR-2 (h)]. This soil sampling design constitutes a stratified random design [as was used in the evaluation of SWMU 1/12 (Mustard Gas Burial Ground) during a recent NSWC Crane investigation]. Samples collected from each of the five subcells in each sub-area will be composited and the composite soil sample will be analyzed as described below. Samples will be collected at the three depth-intervals described below. Thus, three composite soil samples will be submitted to the laboratory from each sub-area [i.e., a total of 36 composite soil samples (i.e., 12 sub-areas; 3 composite samples from each sub-area) will be submitted for analysis]. Additionally, no more than 6 borings (18 samples) will advanced just outside the assumed limits of SWMU 30 to confirm the lateral extent of the SWMU and to further investigate potential contaminant migration from the SWMU.

Because of the shallow depths and unconsolidated nature of the samples to be collected, subsurface soil sampling may be performed using a hand auger or DPT, depending on site conditions. Based on historical accounts of the sludge application activities, the sludges were highly liquid and were poured directly onto the ground surface. At the request of the U.S. EPA, an attempt will be made during sampling to differentiate between sludge and native soil, although it is assumed that an obvious visual distinction between the two will not be feasible. Excess soil cuttings from the borings will be placed back in the borehole. Details regarding soil sampling equipment and procedures are included in the previously approved QAPP for the Old Rifle Range (ORR) at Crane (TtNUS, October 2000).

Three soil samples will be collected from each borehole location from the following nominal depth intervals: 0 to 6 inches, 6 inches to 2 feet bgs, and 2 feet to 4 feet bgs. Bedrock is not anticipated to occur at shallow depths underlying the Landfarm, although if bedrock is shallower than the bottom of a depth interval, sampling will stop at the bedrock. Surface soils will be defined as soils 0 to 2 feet bgs, and subsurface soils will be defined as soils greater than 2 feet bgs. Soil samples will be collected even if the borehole advances into a saturated zone. The top of the water table underlying the Landfarm averages over 25 feet bgs and saturated conditions are not anticipated.

Although subsurface soils will be collected (2 to 4 feet bgs); contamination is most likely to be present in the surface soils (versus subsurface soils) given the fact that the sludges were applied to the surface soils. Initially, samples from the 0 to 6 inch soil interval and the 2 to 4 foot bgs soil interval only will be analyzed for target analytes. The soil sample from the 6 inch to 2 foot bgs interval will be analyzed if chemical concentrations in the 0 to 6 inch depth interval exceeds the following screening levels (and background):

- U.S. EPA Region 9 Preliminary Remediation Goals for exposure to soils (residential land use)
- IDEM - Tier I Default Closure Levels for soils for Residential Land Use
- U.S. EPA Soil Screening Levels for Soil Ingestion, Transfer from Soils to Air, and Migration from Soils to Groundwater
- U.S. EPA Region 5 Ecological Data Quality Levels

A maximum of 54 samples (i.e., 36 sub-area samples and 18 boundary samples) will be collected and analyzed. Additional soil samples may be collected and analyzed in a Phase 2 investigation if:

- The statistical analysis (specific statistical methodologies to be determined following receipt of soil data) of the Phase 1 data indicates that exposure point concentrations based on the Phase 1 data are highly uncertain (e.g., the variability in chemical concentrations noted in the Phase I samples indicate an inadequate number of samples have been collected), and/or

- The vertical or horizontal extent of contamination has not been bounded AND the risk assessment of target analyte concentrations in the Phase I soil samples exceed risk benchmarks acceptable to the Navy and the U.S. EPA.

All of the soil samples will be analyzed for target analyte list metals and cyanide, hexavalent chromium and pH. The analytical detection limit requirements (Table 1), analytical methods and corresponding laboratory SOPs (Table 2), quality control samples collection frequency (Table 3), matrix spike and laboratory duplicate control limits (Table 4), laboratory control sample control limits (Table 5), and bottleware, preservation, and holding time requirements (Table 6) are specified in the attached Tables 1 through 6.

TABLE 1

DETCTION LIMITS VERSUS RISK CRITERIA
NSWC CRANE INDIANA

Parameter	CAS NUMBER	Aqueous Matrix			Solid Matrix		
		Laboratory MDL/IDL[1] (ug/L)	Laboratory RL[1] (ug/L)	Risk-Based Target Level [2] (ug/L)	Laboratory MDL/IDL[1] (mg/kg)	Laboratory RL[1] (mg/kg)	Risk-Based Soil Target Level [2] (mg/kg)
METALS (SW-846 Method 6020 ICP/MS)							
Antimony	7440-36-0	0.1	1.0	6	0.05	1	0.1423
Arsenic	7440-38-2	0.1	10	0.045	0.05	1	0.39
Barium	7440-39-3	0.1	1.0	2000	0.05	1	1.04
Beryllium	7440-41-7	0.1	1.0	4	0.05	1	0.1
Cadmium	7440-43-9	0.1	10	0.66	0.05	1	0.00222
Chromium (total)	7440-47-3	0.1	5.0	11	0.05	1	2
Cobalt	7440-48-4	0.1	3.0	5	0.05	1	0.14033
Copper	7440-50-8	0.1	2.0	5	0.05	1	2.96
Lead	7439-92-1	0.1	1.0	1.3	0.05	1	0.05373
Mercury (SW-846 Method 7470A/7471A)	7439-97-6	0.1	0.2[4]	0.0013	0.02	0.2	0.073
Nickel	7440-02-0	0.1	10	29	0.05	1	7
Selenium	7782-49-2	0.3	1.0	5	0.15	1	0.02765
Silver	7440-22-4	0.1	30	1	0.05	1	2
Thallium	7440-28-0	0.1	10	0.56	0.05	1	0.04
Vanadium	7440-62-2	0.1	2	19	0.05	1	1.59
Zinc	7440-66-6	0.4	10	58.9	0.2	1	6.62
MISCELLANEOUS METALS (SW-846 METHOD 6010B Trace) [5]							
Aluminum	7429-90-5	81	200	87	8.1	20	76000
Calcium	7440-70-2	150	5000	--	15	500	--
Iron	7439-89-6	13	100	300	1.3	10	23000
Magnesium	7439-95-4	7.6	5000	--	0.76	500	--
Manganese	7439-96-5	0.2	15	50	0.02	1.5	1800
Potassium	7440-09-7	16	5000	--	1.6	500	--
Sodium	7440-23-5	76	5000	--	7.6	500	--
MISCELLANEOUS PARAMETERS							
Cyanide (SW-846 Method 9012A)		2.4	10	5.2	0.2	1	1.3
Hexavalent Chromium (SW-846 7196A)		1	5	11	1.2	2.5	0.4

MDL = method detection limit
IDL = instrument detection limit
RL = reporting limit
µg/L = micrograms per liter.
mg/kg = milligrams per kilogram.

TABLE 1

DETCTION LIMITS VERSUS RISK CRITERIA
NSWC CRANE INDIANA

Parameter	CAS NUMBER	Aqueous Matrix			Solid Matrix		
		Laboratory MDL/IDL[1] (ug/L)	Laboratory RL[1] (ug/L)	Risk-Based Target Level [2] (ug/L)	Laboratory MDL/IDL[1] (mg/kg)	Laboratory RL[1] (mg/kg)	Risk-Based Soil Target Level [2] (mg/kg)

Shaded cells indicate laboratory MDLs that exceed a risk-based target level for the project.

Shaded cells indicate laboratory RLs that exceed a risk-based target level for the project.

* Asterisks indicate those chemicals for which the laboratory RL exceeds the risk-based target level for the project.

1 Method Detection Limits (MDLs) (all parameters except metals), instrument detection limits (IDLs) (metals only), and reporting limits as provided by Laucks Testing Laboratories, Inc. The values may change prior to the SWMU investigations as laboratory MDLs and IDLs are updated.

2 Value is based on the lowest human health or ecological risk-based criteria as presented in Appendix B, Tables B-1 (aqueous) and B-2 (solids).

3 Risk-based target level is not provided because human and ecological risk-based criteria are not available for this parameter.

4 Laucks Testing Laboratories is confident that it can reliably report to this PQL, even though this value is less than two times the MDL and/or IDL.

TABLE 2

SUMMARY OF ANALYTICAL PROCEDURES
SOLID SAMPLES
NSWC CRANE, CRANE, INDIANA

Analytical Parameter	Preparation Method	Analytical Method	Preparation/Analytical SOP(s) [1]
Metals (As, Ba, Be, Cd, Cr [total], Co, Cu, Pb, Ni, Se, Ag, Tl, V, Zn)	SW-846 Method 3050B (**no** HCl)	SW-846 Method 6020	LTL-7012 or LTL-7015/ LTL-7202
Sb	SW-846 Method 3050B (**no** H_2O_2)	SW-846 Method 6020	LTL-7012 or LTL-7015/ LTL-7202
Metals (Al, Ca, Fe, Mg, Mn, K, Na)	SW-846 Method 3050B	SW-846 Method 6010B	LTL-7012 or LTL-7015/ LTL-7105
Mercury	SW-846 Method 7471A	SW-846 Method 7471A	LTL-7501
Hexavalent chromium	SW-3060A	SW-846 7196A	LTL7014/LTL-7401
Cyanide	SW-846 9012B	SW-846 9012B	LTL-9104
pH	No prep method required	SW-846 9045C	LTL-9113

1 Laboratory SOPs are attached.

TABLE 3

NON-CALIBRATION QC SAMPLE COLLECTION FREQUENCIES, ACCEPTANCE LIMITS AND CORRECTIVE ACTIONS NSWC CRANE, CRANE, INDIANA

QC Sample Type	Collection Frequency	Acceptance Limits	Corrective Action
Field Duplicate	1 per 10 investigative samples collected.	Aqueous = 20% RPD Soil/Sediment = 50% RPD	Qualify data according to data validation requirements.
Equipment Rinsate Blank	1 per 10 investigative samples collected, with a minimum of one per day of sampling, per non-disposable sampling device/instrument. For pre-cleaned, dedicated, and/or disposable equipment (i.e., disposable plastic trowels, etc.), one rinsate blank will be collected and analyzed at a frequency of one per lot or "batch blank" for a specific equipment type.	< RL (soil and water)	Identify source of contamination, if possible. Qualify data according to validation criteria. Qualify use of data if contamination appears to have adversely affected its usability.
Laboratory Control Sample	1 per 20 environmental samples per matrix	See Table 5	Laboratory action taken per LTL-1008. TtNUS action taken per validation protocols.
Laboratory Method Blank	1 per 20 environmental samples or per preparation batch, whichever is more frequent	< RL (soil and water)	Laboratory action taken per LTL-1008. TtNUS action taken per validation protocols.
Matrix Spike*	1 per 20 environmental samples	See Table 4	Laboratory action taken per LTL-1008. TtNUS action taken per validation protocols.
Matrix Spike Duplicate*	1 per 20 environmental samples analyzed for organic target analytes	See Table 4	Laboratory action taken per LTL-1008. TtNUS action taken per validation protocols.
Temperature Blank	One blank per sample cooler.	4 ± 2 °C	Laboratory action taken per LTL-1008. TtNUS action taken per validation protocols.

* Matrix spike and matrix spike duplicates are not analyzed in the field, but additional sample material must be collected in the field to ensure that the laboratory has enough material for spiking and duplicate analysis.

TABLE 4

QUALITY CONTROL LIMITS[1]
MATRIX SPIKE AND LABORATORY DUPLICATE SAMPLES
METALS ANALYSES
NSWC CRANE, INDIANA

Chemical	Solid Matrix		Aqueous Matrix	
	Accuracy[2] (%R)	Precision[3] (RPD)	Accuracy[2] (%R)	Precision[3] (RPD)
APPENDIX IX METALS BY SW-846 METHOD 6020 (UNLESS OTHERWISE NOTED)				
Antimony	75-125	50	75-125	30
Arsenic	75-125	50	75-125	30
Barium	75-125	50	75-125	30
Beryllium	75-125	50	75-125	30
Cadmium	75-125	50	75-125	30
Chromium (total)	75-125	50	75-125	30
Cobalt	75-125	50	75-125	30
Copper	75-125	50	75-125	30
Lead	75-125	50	75-125	30
Mercury (SW-846 Method 7470A)	75-125	50	75-125	30
Nickel	75-125	50	75-125	30
Selenium	75-125	50	75-125	30
Silver	75-125	50	75-125	30
Thallium	75-125	50	75-125	30
Vanadium	75-125	50	75-125	30
Zinc	75-125	50	75-125	30
MISCELLANEOUS METALS BY SW-846 METHOD 6010B				
Aluminum	75-125	50	75-125	30
Calcium	75-125	50	75-125	30
Iron	75-125	50	75-125	30
Magnesium	75-125	50	75-125	30
Manganese	75-125	50	75-125	30
Potassium	75-125	50	75-125	30
Sodium	75-125	50	75-125	30
Micsellaneous				
Hexavalent Chromium	50-150	30	50-148	30
Cyanide	50-150	30	64-135	11

1 In-house QC limits provided by Laucks Testing Laboratories, Inc.
2 These acceptance limits apply to spikes that augment the native sample analyte concentration by at least 25 percent.
3 These acceptance limits apply to original and duplicate sample concentrations >5x RL. If one or both of the results is <5x RL, the acceptance criterion is ± RL. If one of the results is a non-detect, the reported %RPD will be 200%.

%R = Percent Recovery RPD = Relative Percent Difference

TABLE 5

QUALITY CONTROL LIMITS[1]
LABORATORY CONTROL SAMPLES
METALS ANALYSES
NSWC CRANE, INDIANA

Chemical	Solid Matrix Accuracy (%R)[2]	Aqueous Matrix Accuracy (%R)[2]
METALS BY SW-846 METHOD 6020 (UNLESS OTHERWISE NOTED)		
Antimony	75-125	75-125
Arsenic	80-120	80-120
Barium	80-120	80-120
Beryllium	80-120	80-120
Cadmium	80-120	80-120
Chromium (total)	80-120	80-120
Cobalt	80-120	80-120
Copper	80-120	80-120
Lead	80-120	80-120
Mercury (SW-846 Method 7470A)	80-120	80-120
Nickel	80-120	80-120
Selenium	80-120	80-120
Silver	75-125	75-125
Thallium	80-120	80-120
Vanadium	80-120	80-120
Zinc	80-120	80-120
MISCELLANEOUS METALS BY SW-846 METHOD 6010B		
Aluminum	80-120	80-120
Calcium	80-120	80-120
Iron	80-120	80-120
Magnesium	80-120	80-120
Manganese	80-120	80-120
Potassium	80-120	80-120
Sodium	80-120	80-120
Miscellaneous		
Hexavalent Chromium	50-150	73-130
Cyanide	50-150	85-115

1 In-house QC limits provided by Laucks Testing Laboratories, Inc.

2 As noted in the laboratory SOPs, the laboratory may use a purchased standard reference material (SRM) in place of a LCS. If an SRM is used, the acceptance limits provided by the supplier of the SRM may be used unless the SRM limits are wider than the acceptance limits provided in this table. If the SRM limits are wider, the laboratory must use the acceptance limits provided in this table.

%R = Percent Recovery

TABLE 6

SUMMARY OF SAMPLE ANALYSES, CONTAINER TYPES AND VOLUMES, PRESERVATION REQUIREMENTS, HOLDING TIMES AND ANALYTICAL LABORATORY NSWC CRANE, CRANE, INDIANA

Parameter	Sample Container	Container Volume	Preservation	Maximum Holding Time[1]	Analytical Laboratory
Soil					
Metals	Wide-mouth jar, Teflon-lined plastic cap	8 ounce	Cool to 4 °C	Within 180 days; mercury within 28 days	Laucks
pH				As soon as possible	Laucks
Hexavalent Chromium				28 day to analysis	Laucks
Cyanide				14 days to analysis	Laucks
Aqueous Blanks					
Metals plus tin (total)	Polyethylene, plastic cap w/ plastic liner	1000 mL	Cool to 4 °C, HNO_3 to pH <2	Within 180 days; mercury within 28 days	Laucks
Hexavalent Chromium	Polyethylene, plastic cap w/ plastic liner	500 mL	Cool to 4	24 hours to analysis	Laucks
Cyanide	Polyethylene, plastic cap w/ plastic liner	500 mL or 1000 mL	NaOH	14 days to analysis	Laucks

1 All holding times are from date of collection.

2 Includes Unified Soil Classification, grain size, and bulk density

Laboratory SOPs

LABORATORY STANDARD OPERATING PROCEDURES (SOPs)
TABLE OF CONTENTS

SOP NUMBER	REVISION	TITLE
LAUCKS TESTING LABORATORIES		
LTL-1008	6	QC Corrective Actions
LTL-7012	1	Microwave Assisted Acid Digestion of Sediments, Sludges, Soils, and Oils Using SW846 Method 3015
LTL-7014	0	Alkaline Digestion for Hexavalent Chromium By SW-846 Method 3060A
LTL-7015	1	Acid Digestion of Sediments, Sludges, and Soils Using SW846 Method 3050B
LTL-7105	2	Method Protocols for the Analysis of Metals by ICP Using SW846 6010B
LTL-7202	4	Metals Analysis Using Inductively Coupled Plasma – Mass Spectrometry (ICP/MS) SW846 Method 6020
LTL-7401	2	The Colorimetric Determination of Hexavalent Chromium by SW-846 7196A
LTL-7501	2	Operation of the PE FIMS 400 for the Analysis of Hg for EPA Methods SW-846 7470/7471, 245.2/245.5, and CLP ILM04.1
LTL-9104	2	Preparation and Analysis of Total and Amenable Cyanide according to EPA Methods 335.1, 335.3, and SW-846 Method 9012A
LTL-9113	2	pH in Soil and Waste Samples Using SW-846 Method 9045C

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-1008

Title: **QC Corrective Action**

Revision history:

Number	Date
1 (LTL-0008)	12/12/86
2 (LTL-0008)	5/19/87
3 (LTL-0008)	6/29/89
4	3/3/96
5	6/22/96
6	6/16/00

Written by: Harry Romberg
Harry Romberg, Quality Assurance Officer

Date: 06-19-2000

Approved by: Kathy Kreps
Kathy Kreps, Laboratory Director

Date: 06-19-00

Controlled Document

No. 20 Assigned to: Tetra

Table of Contents

1. INTRODUCTION AND SCOPE **3**

1.1 PURPOSE 3
1.2 SCOPE 3

2. DEFINITION OF TERMS **3**

2.1 THIS SECTION DEFINES TERMS AND ACRONYMS AS THEY ARE USED IN THIS SOP 3

3. OUT-OF-CONTROL EVENT PROCEDURE **4**

3.1 IDENTIFYING AN OUT-OF-CONTROL EVENT 4
3.2 RESPONDING TO AN OUT-OF-CONTROL EVENT 5
3.3 CORRECTIVE ACTIONS 7
3.4 DOCUMENTING AN OUT-OF-CONTROL EVENT 8

APPENDIX 1 ..... **10**

CORRECTIVE ACTION FORM 10

APPENDIX 2 ..... **12**

QC_DB REPORT FORM 12

APPENDIX 3 ..... **14**

OUT-OF-CONTROL EVENT FORM 14

APPENDIX 4 ..... **16**

EXAMPLE SAMPLE RECEIPT FORMS 16

APPENDIX 5 ..... **19**

EXAMPLE LOGSHEET (COLD STORAGE) 19

1. Introduction and Scope

1.1 Purpose

1.1.1 The purpose of this SOP is to establish a system to identify, document and resolve out-of-control events.

1.2 Scope

1.2.1 An out-of-control event may be recognized by any member of Laucks. When they occur, the analyst, supervisor and Quality Assurance work jointly to solve and correct the problem. Out-of-control events are documented using an Out-of-Control-Event form or a Corrective Action form, or in a few selected instances, on a logsheet with space specifically for such actions. Corrective action resulting from an audit is also dealt with using its own Audit Response form but this action is elucidated in an SOP specific to that process.

2. Definition of Terms

2.1 This section defines terms and acronyms as they are used in this SOP.

2.1.1 **Corrective Action**: Action taken by an individual(s) to correct a problem as evidenced by either the failure of QC criteria or a more general problem which could affect performance of an analysis, the quality of service or other activity undertaken by the laboratory.

2.1.2 **Out-of-control event**: Any occurrence or condition failing to meet Laucks QC criteria or has the potential to impact data quality.

2.1.3 **QA/QC**: Quality Assurance/Quality Control

2.1.4 **QAP:** Quality Assurance Plan – The laboratory document governing overall Quality Assurance (QA) policy and guidelines. This document discusses physical assets as well as administrative aspects of the laboratory's QA program.

2.1.5 **Reagent blank**: a measured volume of reagents used in a method.

2.1.6 **RSD:** Relative Standard Deviation - The ratio of the standard deviation of a set of values to the mean of the set of values. A measure of the similarity of the values one to another.

2.1.7 **Method blank or Preparation Blank**: a reagent blank that undergoes a preparation (digestion, extraction, distillation, etc.) step prior to analysis.

2.1.8 **RPD**: Relative Percent Difference

2.1.9 **LCS**: Laboratory Control Sample

3. OUT-OF-CONTROL EVENT PROCEDURE

3.1 Identifying an Out-Of-Control Event

3.1.1 The following is a list of examples of out-of-control events. This is not a complete list of all possible out-of-control events and many of those listed may be different for different methods. Specific criteria are given in analytical SOPs or in other QA documents. If there is doubt about whether a situation is out-of-control and must be responded to, consult with Quality Assurance.

3.1.1.1 GC/MS instrument tune criteria failing to meet criteria

3.1.1.2 Initial calibration linearity, depending upon the method used for calibration, correlation coefficient <0.995 (<0.990 for some fuels analyses) or percent RSD failing to meet method specifications.

3.1.1.3 Daily and continuing calibration verification or calibration blanks outside acceptable ranges as defined in their respective SOPs.

3.1.1.4 **NOTE:** If any of the above instances (3.1.1.1-3.1.1.3) occurs, analysis is stopped. No sample analysis can occur until the event is back in control. A corrective action form does not need to be filled out for these instances if identified at the analyst level and corrected before any data are affected. Since even sample data with non-compliant QC are sometimes used to support other results even if the non-compliant data itself is not reportable, once actual samples or sample-specific QC have been analyzed, the above (3.1.1.1 – 3.1.1.3) constitute out-of-control events and must be formally addressed using the appropriate forms.

3.1.1.5 Matrix spike, surrogate spike or blank spike recoveries outside acceptable ranges.

3.1.1.6 Unacceptable RPD value for MS/MSD or duplicate samples.

3.1.1.7 Unacceptable values for LCS's and QC samples.

3.1.1.8 A method (reagent) blank containing interference or a target analyte at a concentration greater than or equal to ½ the method **reporting** limit.

3.1.1.9 **Note:** Samples which contain target analyte levels which are greater than 20 times the blank or which contain none of the offending analyte may be considered acceptable.

3.1.1.10 A sample received, prepared or analyzed past holding time.

3.1.1.11 A sample depleted before all required analyses are completed.

3.1.1.12 An extract blown down to dryness, spilled or otherwise compromised.

3.1.1.13 Contaminated reagents and glassware.

3.1.1.14 Equipment malfunction or instrument failure, such as cold storage unit temperature outside acceptable ranges and the loss of data acquisition.

3.1.1.15 Record keeping omissions, errors, and deviations from the record keeping standard operating procedures are also out-of-control situations

3.2 Responding to an Out-Of-Control Event

3.2.1 When an out-of-control event is recognized, each individual involved with the analysis in question has an interactive role and responsibility, these are as follows:

3.2.2 Analyst:

3.2.2.1 Must be able to recognize QC failure and immediately take the proper action or, if unsure of the appropriate response, notify the supervisor and work with the supervisor and Quality Assurance to solve the problem; also maintains QC charts.

3.2.2.2 The analyst is also responsible for performing the following steps to correct the problem:

3.2.2.3 Examine all calculations for correctness

3.2.2.4 Examine bench sheets for correctness

3.2.2.5 Check instrumentation and operating conditions to preclude the possibility of malfunctions or operator error

3.2.2.6 Verify integrity of spiking solution, laboratory control sample, or calibration standard

3.2.2.7 Re-analyze the sample

3.2.2.8 Take other actions as noted in the specific analytical SOP.

3.2.2.9 If these steps do not yield acceptable results, consult the supervisor.

3.2.3 Supervisor:

3.2.3.1 Must review all analytical and QC data for reasonableness, accuracy and clerical errors; also responsible for QC charts. Some of the above duties may be assigned to others, with supervisory oversight, if those others have been trained to observe the conditions which would initiate further investigation.

3.2.3.2 In an out-of-control event, the supervisor works with the analyst and Quality Assurance to solve the problem and prevents the reporting of suspect data by stopping work on the analysis in question and insuring that analyses for all suspect results are repeated, if possible, after the source of the error is determined and remedied.

3.2.3.3 If corrective actions do not yield results which meet specifications, it may be determined that sufficient action has been taken. The supervisor and QA will approve of such decisions and if it is determined that the data quality could be impacted, the supervisor will ensure that appropriate comments are reported with the data to the client.

3.2.4 Quality Assurance:

3.2.4.1 The Quality Assurance Officer or designee will work with supervisory personnel and/or analysts to solve out-of-control situations which are not routinely corrected at the bench.

3.2.4.2 In the event that an out-of-control situation occurs that is unnoticed at the bench or supervisory level, such as performance failure on a blind QC sample, Quality Assurance will notify the supervisor, help identify and solve the problem where

applicable, insure the work is stopped on the analysis and no suspect data is reported.

3.2.4.3 Finally the Quality Assurance Officer or designee must review and approve all corrective action reports which cannot be resolved. If corrective actions do not yield results which meet specifications, it may be determined that sufficient action has been taken. The supervisor and QA will approve of such decisions.

3.2.4.4 If it is determined that the data quality could be impacted, the supervisor will ensure that appropriate comments are reported with the data to the client and QA will review said comments.

3.2.5 Project Manager:

3.2.5.1 The Project Manager is responsible for notifying the client of out-of-control events, such as missed holding times, raised reporting limits, matrix interferences, etc. which cannot be resolved without potential impact on either the data quality, the agreed upon or routinely reported results, or the timely and expected delivery date. It is not necessary to contact the client for events which are correctable and do not impact the final data quality, holding times or turn-around unless specifically requested by the client.

3.3 Corrective Actions

3.3.1 Appropriate corrective action depends on the type of analysis, the extent of the discrepancy, and whether the event is determinant or not. The corrective action to be taken for analytical QC failures is usually described in the specific analytical method but may also be determined by either the supervisor, Quality Assurance Officer, or by both in conference, if necessary.

3.3.1.1 Some items may not necessitate direct intervention of QA where standard practices are in place for some events, where the SOP or project or program QAP itself dictates the corrective action and where the action taken is the most conservative response practical. These types of events may be considered to have automatic QA approval and may not even require the completion of any related out-of-control event forms.

3.3.2 A corrective action can be as extensive as replacing a complete lot of contaminated extraction solvent, re-extracting and re-analyzing a complete batch of samples, due to reagent blank contamination; or as simple as recalculating a series of results because a

wrong dilution factor was applied. Again, the appropriate corrective action must be determined on a case by case basis.

3.3.3 Data cannot be released until the system is in control or the QC failure can be attributed to a cause other than method performance. In the case where the out-of-control event is due to matrix problems in the sample and the system remained out of control, the data is flagged, narrative comments usually made in the report, and supporting documentation is released to the client.

3.3.4 Corrective actions are considered adequate when the problem has been resolved and data can be reported or other actions taken from an in-control condition. Alternatively, it may be determined that the action taken was, as a minimum, all that was required by the method or that no further action was reasonable or possible that would improve the data. In these cases, the final decision must be approved by the supervisor and QA.

3.4 Documenting an Out-Of-Control Event

3.4.1 This is accomplished by completing one of the following

- A Corrective Action (CA) Form (See Appendix 1)
- A QC_DB Report Form (for Inorganics analytical QC only, see Appendix 2)
- An Out-Of-Control Event (OOCE) Form (lab use only, see Appendix 3)
- A Sample Receipt Form (for sample receipt events, see Appendix 4)
- An Audit Finding Report Form (QA use only, not shown here, see audit SOP)
- or logged onto a form which itself includes corrective actions (example, Cold Storage Logsheet, see Appendix 5).

3.4.2 CA forms are general and are for documenting corrective action taken to correct problems not associated with a particular analytical event. Such events are usually more general, such as PE sample failure, questioning a general practice even if it did not necessarily precipitate an error because it could lead to problems (i.e. failing to properly document standards or misuse of a run log, etc.)

3.4.3 Out-Of-Control Event (OOCE) Forms are filled out by **technical** laboratory staff only and are designed for documenting analytical QC failures and associated corrective actions. Where other forms, such as the Inorganics QC_DB Report Form, are used to document that the QC parameters were checked, any failures of QC and the decision to perform corrective action or continue data processing must be documented on the OOCE form. The checklist may then be attached to the OOCE form for final data submission.

Note: It is not necessary for analytical staff to document actions which were taken prior to processing samples or which do not in any way affect reported data.

3.4.4 Audit Finding Reports are responded to by the assigned individual and signed off by QA or a designated individual (see the audit SOP).

3.4.5 All OOCE and Corrective Action Forms shall be filled in completely by the person observing the event. Actions taken may be filled in by either the initiating person or the person actually performing the corrective action. The descriptions of the event and any corrective actions taken should be detailed and specific. The OOCE form provides check boxes for most analytical events.

Note: Holding time violations due to laboratory error are annotated on the OOCE form. Holding time violations occurring due to receipt of samples beyond the criteria are documented on the sample receipt form only.

3.4.6 If the corrective action taken and annotated on the OOCE Form resolves the problem and allows data to be reported which is in control, the action is complete and only needs to be signed by the individual taking action and the individual initiating the action.

3.4.7 If the corrective action taken and annotated on the OOCE Form does not resolve the event and it is determined that no further action can or will be taken, the form must be signed by the analyst, supervisor, and QA.

3.4.8 Originals of all OOCE forms must be turned into QA. Copies must be included in each SDG or workorder in validatable packages and in the first workorder in the "samples affected" column for non-validatable data packages.

3.4.9 Any corrective actions taken which could either impact data directly, help to explain analytical decisions that were made in order to resolve analytical discrepancies, or which would help in the interpretation of the final data package must also be narrated in the final report. OOCE forms must be turned in with the data and the supervisor creating the narrative comment for that area will comment on any decisions resulting from failed QC which could impact data validity or interpretation.

Appendix 1

Corrective Action Form

Laucks Testing Laboratories
Corrective Action Report

1) Problem Description:

Response tasked to: _____ on _____

By: _____ Response Requested By _____

2) Cause:

3) Action Taken:

Completed by _____ on _____

☐ Corrective actions will be reviewed 30 days after completion to verify problem has been corrected.

☐ No further action necessary

Reviewed by: _____ on _____

1) Person initiating corrective action fill out Part 1 and may fill out Part 2 if they are aware of the cause
2) Original goes to person tasked with a response; one copy goes to QA Officer and another kept by person initiating corrective action
3) Person tasked completes response in Part 2 (if not previously completed) and Part 3, signs response, and returns original to person initiating action
4) Person initiating action determines if action corrects the problem and signs "Reviewed by." If action was insufficient, return to the person charged with responding without signing.
5) Completed original goes to QA Officer

Appendix 2

QC_DB Report Form

Laucks Testing Laboratories

QC_DB Report Form

Analyst_____

Checker_____

Test Code_____

		QC Exceeds Control Limit √ if yes	Corrective Action Approved By ____
PBlk	B_____00_____	☐	
MS/MSD	K_____00_____	☐	
SRM	R_____00_____	☐	
Blk Spk	S_____00_____	☐	
MS/Dup	M_____00_____	☐	
Duplicate	D_____00_____	☐	
BS/BSD	L_____00_____	☐	

This report validates the following work orders

Appendix 3

Out-Of-Control Event Form

OUT OF CONTROL EVENT FORM

No: _____

Date Recognized: _____
Date Occurred: _____
Method: _____
Initiated By: _____
Analyst: _____

- ☐ GC/MS VOA
- ☐ GC/MS ABN
- ☐ GC VOA
- ☐ GC non-VOA
- ☐ HPLC
- ☐ Metals
- ☐ Wet Chemistry
- ☐ Extractions
- ☐ Data Management

Samples Affected (Workorder & Sample Numbers.)

Type of Event: (*check all that apply*)

___ Holding time missed (*describe below*)
___ Blank ≥ MDL___ RL___ CRQ/DL___
___ Spike Recoveries do not meet criteria
___ Duplicate RPDs do not meet criteria
___ MS/MSD Results do not meet criteria ___ %Rec ___ RPD
___ BS/BSD Results do not meet criteria ___ %Rec ___ RPD
___ Analytical Spike recoveries do not meet criteria
___ Standard Additions do not meet criteria
___ LCS or Blank Spike Recoveries do not meet criteria
___ Surrogate Recoveries do not meet criteria
___ Calibration Corr. Coefficient does not meet criteria
___ Calibration Verification does not meet criteria ___ Init ___ Cont.
___ Recovery ___ Retention time ___ %D
___ Tuning fails criteria
___ ISTD fails criteria
___ Calculation/Transcription error
___ Other (explain)

Corrective Action: (*check all that apply*)

___ Repeat Calibration
___ Made new standards
___ Reanalyzed, Date: _____
___ Sample(s) Redigested/Reextracted Date: _____
___ Results Recalculated
___ Cleaned System
___ Ran Standard Additions
___ Notified Client _____
___ Other (Please explain)

Check One:

___ Original Results Reported
___ Rerun Results Reported

Notified:

___ QA
___ Client Services

Action taken By: _____ Date: _____ Reviewed by Initiator: _____ Date: _____
___ Out of Control Event Corrected By: _____

___ Corrective Actions Not Successful (signatures required) **DATA MUST BE FLAGGED AND/OR NARRATED**

Analyst: _____ Date: _____
Supervisor: _____ Date: _____
QA Department: _____ Date: _____

Distribution:
Original to QA
Copy to workorder / SDG file for all validatable packages and to first workorder on list for non-validatable data.

Appendix 4

Example Sample Receipt Forms

Laucks Testing Laboratories, Inc.
NON-CLP SAMPLE RECEIPT LOG

Initial once samples are checked in_____

DATE RECEIVED:_____
TIME RECEIVED:_____
CLIENT NAME:_____
SDG #_____
COC #_____

SAMPLE LOG-IN DATE:_____
WORKORDER #:_____
CLIENT PROJECT:_____
AIRBILL ATTACHED?:(#)_____
RECEIVED BY:_____

Non-Conformance: (Check applicable item(s)) _____ Client IDs affected:

- ☐ (1) Not enough sample sent for proper analysis. #s affected:_____
- ☐ (2) Sample Bottle received broken and/or cap not intact._____
- ☐ (3) Custody seal: Absent____ Present/Intact_____ Present/Broken _____
- ☐ (4) Any temperature out of compliance:_____
- ☐ (5) Sample received outside of holding time._____
- ☐ (6) Sample not properly preserved. pH = ___. Wrong preservative used._____
- ☐ (7) Illegible sample numbers or label missing from bottles._____
- ☐ (8) Identification on bottle same as identification on paperwork: yes:_____no:_____
- ☐ (9) Incomplete instructions received with sample(s), i.e.,
- ☐ no Request for Analysis, no Chain-of-Custody._____
- ☐ (10) Samples received in improper container._____
- ☐ (11) Samples held in field before receipt by Lab. Days (specify)_____
- ☐ (12) Air Bubble(s) in ___of ____ samples for volatiles analysis._____
- ☐ (13) Other _____

CORRECTIVE ACTION: (Check applicable item(s))

Correction action taken by:

		Inititals	Date
☐	(1) Client informed verbally (Client Services).	_____	
☐	(2) Client informed by memo/letter/fax (Client Services).	_____	_____
☐	(3) Sample processed "as received" (Sample Entry).	_____	_____
☐	(4) Re-sampling requested of client (Client Services).	_____	_____
☐	(5) Samples placed "on hold" until further notice (Sample Entry/Client Services).	_____	_____
☐	(6) NOTE IN NARRATIVE. See temperature/pH login sheet. (Sample Entry).	_____	_____
☐	(7) Other (Specify)_____	_____	_____

* When complete (within 24 hours of nonconformance) forward to QA. Original to be forwarded to initiator to be included in transmittal file.

Comments:

_____

_____

_____

_____

_____

Laucks Testing Laboratories, Inc.
SAMPLE RECEIPT LOG (2) In-house

P:\PM\Holly\docs\fax\pjmemo.doc12/22/94

DATE RECEIVED:_____ WORKORDER #:_____
CLIENT NAME:_____
RECEIVED BY:_____

Non-Conformance:

☐ Other

_____

_____

_____

_____

_____

_____

_____

_____

_____

_____

_____

_____

_____

CORRECTIVE ACTION: (Check applicable item(s))

		Correction action taken by: Inititals	Date
☐	(1) Client informed verbally (Client Services).	_____	
☐	(2) Client informed by memo/letter/fax (Client Services).	_____	_____
☐	(3) Sample processed "as received" (Sample Entry).	_____	_____
☐	(4) Re-sampling requested of client (Client Services).	_____	_____
☐	(5) Samples placed "on hold" until further notice (Sample Entry/Client Services).	_____	_____

* When complete (within 24 hours of nonconformance) forward to QA. Original to be forwarded to initiator to be included in transmittal file.

Comments:

_____

_____

_____

_____

_____

Appendix 5

Example Logsheet (Cold Storage)

Cold Storage Temperature Log

Laucks Testing Laboratories, Inc.

Cold Storage ID #:_____ Thermometer ID:_____

Location:_____ Year: **2000** Correction Factor (*add this number when* ***recording*** *the thermometer reading)*:_____°C

Day	Month: Time	Temp.	Initials	Actions	Month: Time	Temp.	Initials	Actions	Month: Time	Temp.	Initials	Actions
1												
2												
3												
4												
5												
6												
7												
8												
9												
10												
11												
12												
13												
14												
15												
16												
17												
18												
19												
20												
21												
22												
23												
24												
25												
26												
27												
28												
29												
30												
31												

Q:\TEMPLATS\LTLFORMS\REFERFRM.DOC 12/21/99

Record Time and Temperature in the proper blocks and initial the entry each day of normal laboratory operation.
If refrigerator temperatures exceed 4°C±2°C or if freezer temperatures are warmer than -10°C, corrective action must be taken.
Corrective action includes 1) Adjust the temperature of the thermostat 2) Defrost the refrigerator or freezer
3) Contact the appropriate laboratory maintenance personnel, the departmental supervisor, and/or the QA Officer
4) One of the above may decide that professional maintenance is necessary or even that the cold storage unit must be disposed of.

Any and all [illegible]ns **MUST** be recorded on this log sheet. If there is insufficient room, mark on the back [illegible] page with the date the action occurred.
Samples **M**[illegible] **NOT** be stored in units which are not maintaining the proper temperature.

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP: LTL-7012

Title: **Microwave Assisted Acid Digestion of Sediments, Sludge, Soils, and Oils Using SW846 Method 3051**

Revision history:

Number	Date
0.	6/04/96
1.	8/21/98

UNCONTROLLED

Revised by: _____ Date: 08-21-98
Ming-Hwa Hwang, Metals Supervisor

Reviewed by: Harry Romberg Date: 8-21-98
Harry Romberg, QA Officer

Approved by: Kathy Kreps Date: 8-24-98
Kathy Kreps, Laboratory Director

Table of Contents

1. INTRODUCTION AND SCOPE 3

2. SUMMARY OF METHOD 3

3. SAFETY PROCEDURES 4

4. EQUIPMENT 4

5. REAGENTS 4

6. MICROWAVE SAMPLE DIGESTION PROCEDURE 5

7. QUALITY CONTROL 6

8. REFERENCES 7

APPENDIX A, SAMPLE LTL-7012 DIGESTION LOGS 8

APPENDIX B, FLOWCHART FOR LTL-7012 10

APPENDIX C, QUALITY CONTROL SOLUTIONS 12

1. Introduction and Scope

1.1 This method is restricted to use by, or under the supervision of analysts experienced in the technique described. Each analyst performing this method must have demonstrated the ability to perform the described analysis.

1.2 This digestion procedure is used for the preparation of sediments, sludge, soils, and oils for analysis by flame atomic absorption spectroscopy (FLAA), graphite furnace absorption spectroscopy (GFAA), inductively coupled argon plasma spectroscopy (ICP), or inductively coupled argon plasma mass spectrometry (ICP-MS). The procedure is a hot acid leach for determination of available metals.

1.3 Samples prepared by Method 3051 using nitric acid digestion may be analyzed by FLAA, GFAA, ICP or ICP-MS for the following:

Aluminum	Cadmium	Iron	Molybdenum	Sodium
Antimony	Calcium	Lead	Nickel	Strontium
Arsenic	Chromium	Magnesium	Potassium	Thallium
Boron	Cobalt	Manganese	Selenium	Vanadium
Barium	Copper	Mercury	Silver	Zinc
Beryllium				

1.4 This method is provided as an alternate to LTL-7011. It is intended to provide a rapid multielement acid leach digestion prior to analysis by Flame Atomic Absorption(FLAA), Graphite Furnace Atomic Absorption(GFAA), Inductively Coupled Plasma Emission Spectroscopy, or Inductively Coupled Plasma Mass Spectroscopy(ICP-MS). If a decomposition including Hydrochloric Acid is required for certain elements, it is recommended that LTL-7011 be used. The parameters which are quoted in this procedure are those recommended by the manufacturer of the microwave digestion system used, in order to be in compliance with SW-846 protocols.

2. Summary of Method

2.1 A representative sample of up to 0.5g is digested in a Teflon vessel with 10 mL of HNO_3 in a microwave unit. After cooling, the vessel contents are either filtered or allowed to settle and diluted to volume and analyzed by an appropriate method.

3. Safety Procedures

3.1 These procedures involve the use of strong and/or hot acid solutions. The analyst must wear eye protection, a lab coat, and gloves to protect against burns.

3.2 Samples and spiking solutions may contain high levels of toxic metals and other unknown constituents. The analyst must take every precaution to avoid contact with these potentially hazardous materials and should wash hands thoroughly before eating or leaving the laboratory.

3.3 **Very reactive or volatile materials may create high pressures when heated, causing the vessels to vent with potential loss of sample and analytes. Samples that contain carbonates or other carbon dioxide generating compounds may generate enough pressure to vent the vessel. If this situation is anticipated the analyst may wish to use a smaller sample or an alternative digestion method.**

4. Equipment

- MDS-2100 Microwave Digestion system, CEM
- 120 mL Teflon® vessels and caps, carousel
- 50 or 100 mL graduated cylinder, acid washed
- Glass funnels, acid-washed
- Filter paper, Whatman No. 41 or equivalent
- Sample digestate bottles, acid washed
- Analytical balance, capable of weighing ± 0.01 g

Note: All glassware used for this digestion must be prepared by the method stated in Inorganics Glassware Cleaning Procedures, LTL-7003.

All Teflon® vessels and caps are to be thoroughly rinsed with deionized water prior to digestions and shaken to remove any significant droplets in the vessels.

The microwave is to be calibrated according to the procedures stated in SW846 method 3051. The MDS-2100 microwave, being designed with temperature control, does not require the calibration outlined in method 3051.

5. Reagents

5.1 Reagent Water - Reagent water will be interference free. All references to water in the method refer to reagent water unless otherwise specified.

5.2 Nitric acid (concentrated), HNO_3, ACS Reagent grade or better. Acid should be analyzed to determine levels of impurities. If the method blank is less than reporting limit then the acid can be used.

6. Microwave Sample Digestion Procedure

See Appendix A for digestion log
See Appendix B for flowchart
See Appendix C for quality control solutions

6.1 Weigh a well mixed sample directly into the Teflon digestion vessel to the nearest 0.01g. For soils, sludge and sediments, use no more than 0.50g. For oils, use no more than 0.25g.

6.2 Add 10 mL of concentrated nitric acid to each vessel and cap hand tight. If a vigorous reaction occurs with the addition of the acid, wait for the reaction to subside before capping. For each microwave digestion run, the pressure control lid is to accompany the sample that seems to have the most carbon. Weigh each capped vessel to the nearest 0.01 g.

6.3 Microwave

6.3.1 MDS-2100 Microwave Digestion system, CEM

6.3.2 Place the carousel in the unit and attach the pressure control line to the digestion vessel with the double ported lid. Run the heating program SW-3051 (temperature).

Program SW-3051 (temperature) as follows:

Stage	(1)	(2)	(3)	(4)	(5)
%POWER	100	0	0	0	0
PSI	70	0	0	0	0
RAMP	05:00	0000	0000	0000	0000
HOLD	04:30	0000	0000	0000	0000
TEMP	175	000	000	000	000

6.4 At the end of the microwave program, allow the vessels to cool in the unit for a minimum of 5 minutes before removal to avoid possible injury if a vessel vents immediately after microwave heating. Once they are at room temperature, weigh and record the weight of each vessel assembly. If the weight of the sample plus acid has decreased by more than 10%, discard and prepare the sample again.

6.5 Uncap the vessels in a fume hood due to the likely presence of nitrogen oxide. Dilute sample to 50 mL in a graduated cylinder and transfer the samples to an acid-cleaned polyethylene bottle. If only one graduated cylinder is used to volume all samples, the blank is to be the final sample diluted to assure the absence of sample carry-over. If the digested sample contains particulate which may clog nebulizers or interfere with injection of the sample into an instrument, the sample may be allowed to settle or be filtered.

6.6 The prepared samples are then transferred to the metals analysis department. The digestion log is to reflect the time at the start and finish of the digest. In order to maintain a strict chain of custody, the time and date when the digestates are relinquished to the analysis department, as well as the initials of the analyst accepting the digest, are recorded on the digestion log

7. Quality Control

7.1 In each batch or SDG consisting of no more than 20 samples, a preparation blank is created. The blank should be labeled on the digestate bottle in the following way: B, date, M (microwave), S (soil) and the sequence number of the digestion. Example B061598MS01.

7.2 Spiked samples should be employed to determine accuracy. In each batch or SDG consisting of no more than 20 samples, either matrix spike / sample duplicate (MS/Dup) or matrix spike /matrix spike duplicate (MS/MSD) samples will be prepared. For SW3051-specified quality control measures, MS/Dup samples will be prepared in the following way. Dispense a sample in triplicate. Two will be analyzed exactly as any other sample. The third must be spiked with the appropriate spike solutions, determined by the analyst. All glassware and digestate bottles must be marked appropriately. If MS/MSD samples are required instead of the SW846-specified MS/Dup, both the second and third aliquot must be spiked.

7.3 Determine the analytes required for analysis by consulting sample work order information. The analyst must record the specific spiking solution(s) used, the volume(s) of the solution(s), and the corresponding Metals Standard Logbook number(s) on the digestion log as illustrated in Appendix C

7.4 In each batch or SDG consisting of no more than 20 samples, prepare a Lab Control Sample (LCSS) in exactly the same manner as a sample. All spikes and standard reference materials must be recorded by the analyst on the digestion log. The record must state the specific spiking solution used, the volume of the solution, and it's Metals Standard Logbook number, and the lot number of any reference materials.

8. References

8.1 USEPA, Test Methods for Evaluating Solid Waste Physical/Chemical Methods, USEPA SW846, most recent version, Method 3051.

Appendix A, Sample LTL-7012 Digestion Logs

Laucks Testing Laboratories, INC.	START DATE TIME	FINISH DATE TIME	METHOD	ANALYST	ANALYSIS
SDG #:			SW846 3051		Microwave Digestion
MATRIX: SOIL					

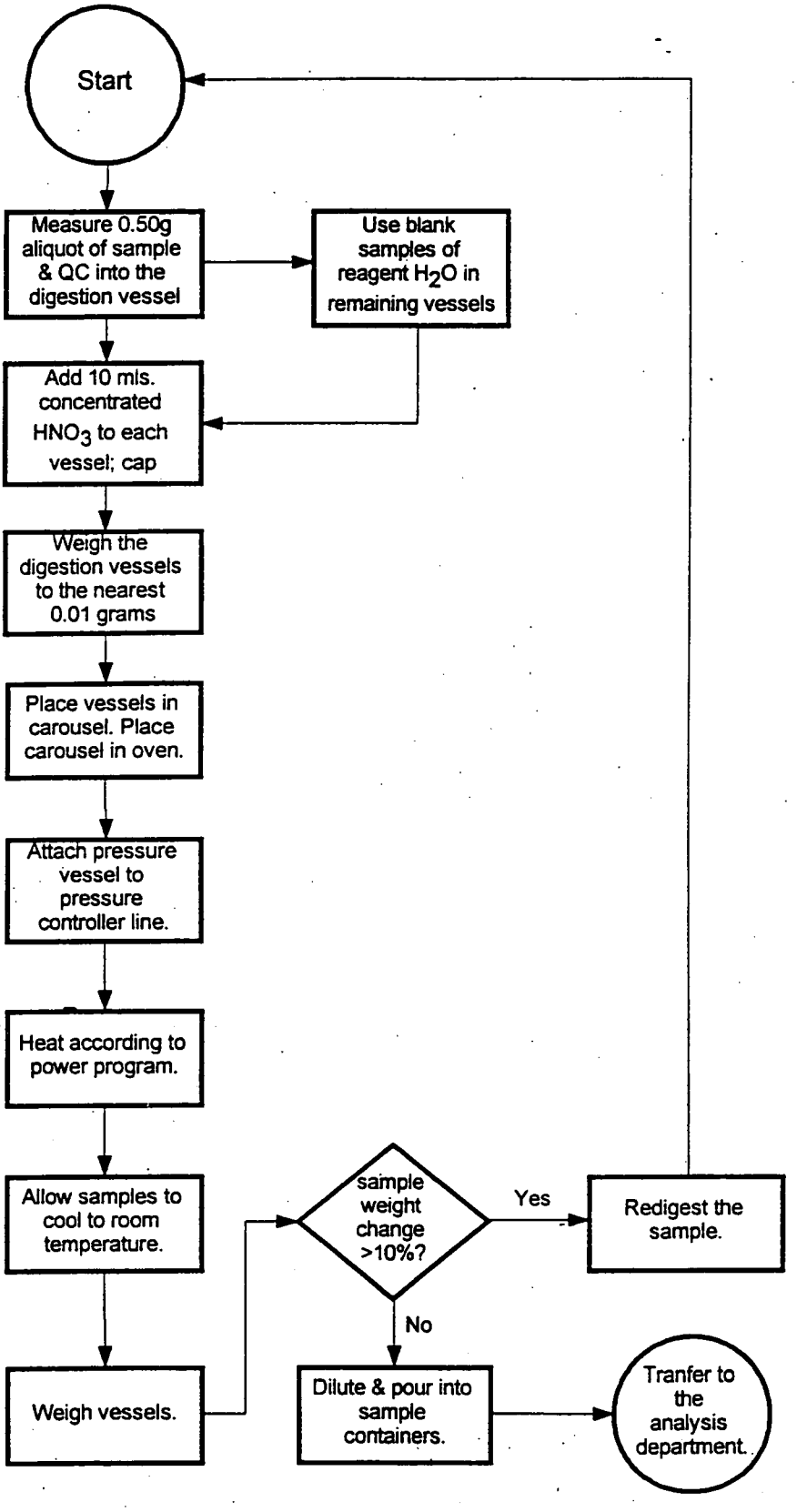
Bomb #	Sample ID	Samp (g)	Final Vol (mL)	Color		Text	Artifacts	Bomb & Sample (g)	Final Bomb (g)	Δ (g)	Δ >0.5 g?
				Int	Fin						

Chain of Custody	Digest Relinquished Date / Time : Analyst	Digest Accepted :Analyst	Digest Disposed :Date
	/ :		

COLOR: 1 = Red, 2 = Blue, 3 = Yellow, 4 = Green, 5 = Orange, 6 = Violet, 7 = White, 8 = Brown, 9 = Gray, 10 = Black, 11 = Colorless.

TEXTURE: F = Fine (Powdery), M = Medium (Sandy), C = Coarse (Rocky).

Appendix B, Flowchart for LTL-7012


Start
Measure 0.50g aliquot of sample & QC into the digestion vessel
Use blank samples of reagent H_2O in remaining vessels
Add 10 mls. concentrated HNO_3 to each vessel; cap
Weigh the digestion vessels to the nearest 0.01 grams
Place vessels in carousel. Place carousel in oven.
Attach pressure vessel to pressure controller line.
Heat according to power program.
Allow samples to cool to room temperature.
Weigh vessels.
sample weight change >10%?
Yes
Redigest the sample.
No
Dilute & pour into sample containers.
Tranfer to the analysis department.

Appendix C, Quality Control Solutions

ICP Spikes

- The CLP spike solution for ICP digest was made by diluting 10 mLs of CLPP-SPK-1™, 10 mLs of CLPP-SPK-2™, and 10 mLs of CLPP-SPK-3™ to 100 mLs with Type II water.

- Add 500 µLs of CLP spike solution to soil samples prior to digestion.

ICP Analytes

Analyte	Concentration in Digest (µg/L)	Analyte	Concentration in Digest (µg/L)
Ag	50	Fe	1000
Al	2000	Mn	500
As	2000	Ni	500
Ba	2000	Pb	500
Be	50	Se	2000
Cd	50	Sb	500
Co	500	Tl	2000
Cr	200	V	500
Cu	250	Zn	500

Table based on a final volume of 50 mL for soil digests.

GRAPHITE FURNACE SPIKES

- Add the analytes listed in the table below to a 100 mL volumetric flask which contains ~50 mL of Type II water and 5 mL of HNO_3.

Analyte	Volume	Stock Concentration
As	400 µL	1000 mg/L
Pb	200 µL	1000 mg/L
Se	100 µL	1000 mg/L
Tl	500 µL	1000 mg/L

- Dilute to 100 mL with Type II water. This spike solution now contains:

4.0 mg/L	Arsenic
2.0 mg/L	Lead
1.0 mg/L	Selenium
5.0 mg/L	Thallium

- Add 500 µL of Spike Solution to soil samples prior to digestion.

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-7014

Title: **Alkaline Digestion For Hexavalent Chromium By SW 846 Method 3060A**

Revision history:

Number	Date
0	06/03/97

Controlled Document
No. 20 Assigned to: Tetra

Written by: Bill Lundberg
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Date: 6/4/97

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TABLE OF CONTENTS

1. INTRODUCTION AND SCOPE 3

1.1 METHOD DESCRIPTION 3
1.2 SAMPLE COLLECTION, SAMPLE STORAGE, HOLDING TIMES 3
1.3 DEFINITION OF TERMS 4

2. EQUIPMENT LIST AND STANDARDS 5

2.1 APPARATUS 5
2.2 STANDARDS 5
2.3 REAGENTS 6

3. SAFETY PRECAUTIONS AND WASTE DISPOSAL 6

3.1 SAFETY PRECAUTIONS 6
3.2 WASTE DISPOSAL 7

4. CALIBRATION AND QUALITY CONTROL 7

4.1 METHOD DETECTION LIMIT STUDY 7
4.2 METHOD BLANKS 7
4.3 METHOD BLANK SPIKES 8
4.4 PRE DIGESTION MATRIX SPIKE 8
4.5 POST DIGESTION SPIKE 9
4.6 SAMPLE DUPLICATE 10

5. OPERATION PROCEDURES 11

5.1 SAMPLE ANALYSIS 11
5.2 ANALYTICAL OPERATION 11
5.3 COMPOUND QUANTIFICATION 12

6. REPORTS 13

6.1 DATA PACKET ORGANIZATION 13
6.2 QUALITY CONTROL REPORTS 13
6.3 SAMPLE RESULT REPORTS 13

APPENDIX I 14

QC SUMMARY TABLE 14

APPENDIX II 15

EH/PH PHASE DIAGRAM 15

APPENDIX III 16

FLOW CHARTS 16

1. Introduction and Scope

1.1 Method Description

1.1.1 Method 3060A is an alkaline digestion procedure for extracting hexavalent chromium, Cr(VI), from soluble, adsorbed, and precipitated forms of chromium compounds in soils, sludges, sediments, and some industrial waste materials. To quantify total CR(VI) in a solid matrix, three criteria must be satisfied: (a) the extracting solution must solubilize all forms of Cr(VI), (b) the conditions of the extraction must not induce reduction of native Cr(VI) to Cr(III), and (c) the method must not cause oxidation of native Cr(III) contained in the sample to Cr(VI). Method 3060A meets these criteria for a wide spectrum of solid matrices. Under the alkaline conditions of the extraction, minimal reduction of Cr(VI) or oxidation of native Cr(III) occurs. The addition of Mg^{2+} in a phosphate buffer to the alkaline solution has been shown to suppress oxidation if observed. The accuracy of the extraction procedure is assessed using spike recovery data for soluble and insoluble forms of Cr(VI) (e.g., $K_2Cr_2O_7$ and $PbCrO_4$), coupled with measurement of ancillary soil properties, indicative of the potential for the soil to maintain a Cr(VI) spike during digestion, such as oxidation-reduction potential (ORP), pH, organic matter content, ferrous iron, and sulfides. Recovery of an insoluble Cr(VI) spike can be used to assess the first two criteria, and method-induced oxidation is minimal except in soils high in Mn and amended with soluble Cr(III) salts or freshly precipitated $Cr(OH)_3$. The sample is digested using 0.28M Na_2CO_3/0.5M NaOH solution and heating at 90-95°C for 60 minutes to dissolve the Cr(VI) and stabilize it against reduction to Cr(III). After digestion the Cr(VI) is quantitated using SW 846 7196A.

1.1.2 This method is restricted to use by, or under the supervision of analysts experienced in the technique described. Each analyst performing this method must have demonstrated the ability to perform the described analysis.

1.2 Sample Collection, Sample Storage, Holding Times

1.2.1 Samples should be collected using devices and placed in containers that do not contain stainless steel (e.g., plastic or glass). A 16 oz glass jar will be required due to the possible need to analyze the sample for other parameters should the matrix spike (MS) exceed limits.

1.2.2 Samples should be stored field-moist at 4°C ± 2°C until analysis.

1.2.3 Hexavalent chromium has been shown (interlaboratory studies) to be quantitatively stable in field-moist soil samples for at least one month from sample collection. In addition, Cr

(VI) has also been shown (interlaboratory studies) to be stable in the alkaline digestate for up to 96 hours after extraction from soil.

1.3 Definition of Terms

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

1.3.2 Batch Identifier - A number given to each preparation or analysis group which uniquely identifies that batch. This number is generally the blank ID for preparation batches and an analysis number which is similar to the blank ID, only preceded by an "A" rather than a "B" for inorganic batches. The preparation batch IDs are discussed in other documentation. The batch identifier for the second run of soils for Cr(VI) analyzed on June 2, 1997 would be A060297_CR6_S02.

1.3.3 Blank spike - A background free matrix (clean sand for soils/sediments) to which a known amount of Cr(VI) is added each time samples are prepared. Blank spikes are required on all HAZWRAP and NFESC work. Note that an LCS or SRM will substitute as a blank spike for most inorganic analyses. At this time there is no known Cr(VI) LCS. In the context of this SOP, a blank spike is the same as a QC check standard. See also QC check standard.

1.3.4 DIW - Deionized water - Lab reagent water. This water should be free of virtually all analytes.

1.3.5 IDL - Instrument detection limit. The lowest concentration of a target analyte that will yield a signal:noise ratio of least 3x. Used as a starting point for selecting MDL study spiking levels.

1.3.6 MDL - Method detection limit - The lowest concentration a sample which will yield a positive result that is greater zero at a known level of confidence. MDLs are empirically determined by Laucks.

1.3.7 MDL standard - Method detection limit standard - A standard prepared so that the concentrations of the target analytes are no greater than 4x the empirically determined MDLs. This standard is used to verify that the instrument or system is capable of detecting the target analytes on an ongoing basis.

1.3.8 QC check standard - Quality control check standard. Referred to in this SOP as a blank spike. A QC check standard is used to determine whether the analytical system is in control if MS/MSD recoveries are out of control. See also blank spike.

2. Equipment List and Standards

2.1 Apparatus

2.1.1 Beakers: borosilicate glassware, 250-mL, with watch glass covers.

2.1.2 Graduated Cylinder: 100-mL.

2.1.3 Volumetric Flasks: Class A glassware, 1000-mL and 100-mL with stoppers.

2.1.4 Filtration Apparatus.

2.1.5 Filter membranes (0.45 μm). Preferably cellulosic or polycarbonate membranes.

2.1.6 Heating Device - capable of maintaining the digestion solution at 90 - 95°C with continuous auto stirring capability or equivalent.

2.1.7 Volumetric pipettes: Class A glassware, assorted sizes, as necessary.

2.1.8 Calibrated pH meter.

2.1.9 Calibrated balance.

2.1.10 Thermometer (NIST-Certified or equivalent) or other appropriate temperature sensing device.

2.2 Standards

2.2.1 Potassium Dichromate, $K_2Cr_2O_7$, spiking solution, 100 mg/L Cr (VI). Dissolve 0.2829 g of dried (105°C) $K_2Cr_2O_7$ in distilled deionized water in a 1 liter volumetric flask and dilute to the mark. Store at 20-25°C in a tightly sealed container for up to six months.

2.2.2 The Blank Spike, and MS are prepared by adding 1.0 mL of the 100 mg/L standard to their respective beakers.

2.2.3 Lead Chromate: $PbCrO_4$, analytical reagent grade. The insoluble matrix spike is prepared by adding 10-20 mg $PbCrO_4$ to a separate aliquot. Store under dry conditions at 20-25°C in a tightly sealed container.

2.3 Reagents

2.3.1 Nitric acid: HNO_3 concentrated, analytical reagent grade or spectrograde quality. Store at 20-25°C in the dark. Discard if the solution has a yellow tinge; this is indicative of photoreduction of NO_3^- to NO_2^-

2.3.2 Sodium carbonate: Na_2CO_3, anhydrous, analytical reagent grade. Store at 20-25°C in a tightly sealed container.

2.3.3 Sodium hydroxide: NaOH, analytical reagent grade. Store at 20-25°C in tightly sealed container.

2.3.4 Magnesium Chloride: $MgCl_2$ (anhydrous), analytical reagent grade. 392.18 mg $MgCl_2$ is equivalent to 100 mg Mg^{2+}. Store at 20-25°C in a tightly sealed container.

2.3.5 Phosphate Buffer: 0.5M K_2HP0_4/0.5M KH_2PO_4 buffer at pH 7: Dissolve 87.09 g analytical reagent grade K_2HP0_4 and 68.04 g analytical reagent grade KH_2PO_4 in 700 mL of deionized water. Transfer to a 1L volumetric flask and dilute to volume.

2.3.6 Digestion solution: Dissolve 20.0 ± 0.05 g NaOH and 30.0 ± 0.05 g Na_2CO_3 in deionized water in a one-liter volumetric flask and dilute to the mark. Store the solution in a tightly capped polyethylene bottle at 20-25°C and prepare fresh monthly. The pH of the digestion solution must be checked before using. The pH must be 11.5 or greater; if not, discard.

3. Safety precautions and Waste Disposal

3.1 Safety Precautions

3.1.1 All standards, samples and sample solutions should be handled as if they are hazardous substances.

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

3.1.3 Electrical shock - All instruments present the possibility of electrical shock. The operator should take all precautions including ensuring that all instruments are operated with fully

grounded power outlets, turning off the instrument and disconnecting the instrument from the electrical power supply before working on any electrical components, etc.

3.2 Waste Disposal

3.2.1 The waste generated by this digestion are not hazardous and may discarded down the sink, while diluting with tap water. Waste segregation and disposal from the point of collection is further covered in the Laucks SOP on Waste Segregation and Disposal.

4. Calibration and Quality Control

4.1 Method Detection Limit Study

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

4.2 Method Blanks

4.2.1 Method blanks are used to verify contamination free reagents and apparatus. They are prepared with every set of samples prepared at the same time or at least one blank every 20 samples which ever is more frequent. Any analyte response above the detection limit is reported. Method blank control limits are that contamination should not exceed the Reporting Limit or 10% of the concentration of the lowest sample, whichever is greater.

4.2.2 Corrective action

4.2.2.1 Corrective action may necessitate re-preparation and re-analysis of the sample set. For example if an analyte were found in the blank but not in any of the associated samples then sample group may not require re-analysis. In addition, if sample levels exceed 10 times the blank, the level of contamination may be considered insignificant. In any case, if re-preparation and re-analysis is not being undertaken, the analyst must first discuss the issue with the Quality Assurance Officer. It is the laboratory's responsibility to ensure that method interferences caused by contaminants in acids, solvents, reagents, glassware, and other sample processing hardware leading to discrete artifacts and/or elevated baselines in the analytical run be minimized. In the extreme case of chronic contamination, blanks may have to be analyzed from each stage of the sample processing to determine the contamination source so it can be

eliminated. In all cases where blank contamination exceeds the control limit, a narrative comment must be made which documents the corrective actions taken.

4.3 Method Blank Spikes

4.3.1 A method blank spike follows the same protocol as with the matrix spike analysis except that the spiking solution is added to a method blank solution instead of an actual sample. A method blank with added analytes is a method blank spike. A method blank spike is the same as a QC check standard. A blank spike OR a standard reference material (SRM) must be analyzed. The SRM is the preferred material and the blank spike should only be analyzed where an SRM does not exist or is not practical for routine use.

4.3.1.1 Corrective action

4.3.1.2 Recovery must be within the certified acceptance range or a recovery range of 80 to 120% or the sample batch must be reanalyzed.

4.4 Pre digestion Matrix Spike

Both soluble and insoluble pre-digestion matrix spikes must be analyzed at a frequency of one per batch of ≤ 20 field samples. The soluble matrix spike should be spiked with 1.0 mL of the spiking solution prepared in 2.2.1 (equivalent to 40 mg/kg Cr(VI)) or at twice the sample concentration, whichever is greater. The insoluble matrix spike is prepared by adding 10-20 mg of $PbCrO_4$ (2.2.3) to a separate sample aliquot. It is used to evaluate the dissolution during the digestion process. Both matrix spikes are then carried through the digestion process. More frequent matrix spikes must be analyzed if the soil characteristics within the analytical batch appear to have significant variability based on visual observation.

4.4.1 A sample is chosen at random from the samples to be analyzed, and an aliquot of spiking solution is added to this sample prior to preparation. The analyst should attempt to avoid selecting samples which are identified by the client as blanks. As the purpose of the matrix spike is to test the system under "typical" conditions, the analyst may also avoid selecting the most difficult sample of the batch for spiking. It is not always required that a matrix spike analysis be performed with each preparation/analysis batch, however, the minimum frequency for MS analysis is 1 each per 20 samples per matrix. This will be best accomplished by running one with every batch for many analyses. This matrix spike sample is used to evaluate the matrix effect of the sample upon recovery of the analytes. The recovery of spike analytes is calculated as follows:

$$\text{recovery} = \frac{(SS - S) * 100}{SA}$$

where:

SS = concentration in spiked sample
S = native concentration in unspiked sample
SA = spiked added, the amount of spiking material actually added to the sample calculated on the sample basis

4.4.2 The recovery criteria are defined by SW 846 as 75% - 125%.

4.4.3 Corrective action

4.4.3.1 If the matrix spike recoveries are not within these recovery limits, the entire batch must be redigested/reanalyzed. If upon reanalysis the matrix spike is not within the recovery limits, but the LCS is within criteria specified in 4.3.1.2, information such as pH, Fe^{+2}, ORP, S_2 and TOC should be carefully evaluated, as the Cr(VI) data may be valid for use despite the perceived "QC failure." The information discussed below is provided to interpret ancillary parameter data in conjunction with data on spike recoveries.

When pre-digestion matrix spike recoveries for Cr(VI) are less than acceptance range minimum criterion (75%), this is indicative of highly reducing samples (e.g., anoxic sediments) with no measurable native Cr(VI) in the unspiked sample (assuming the criteria in 4.3.1.2 are met). Such a result indicates that the combined and interacting influences of ORP, pH and reducing agents (e.g., organic acids, Fe^{+2} and sulfides) caused reduction of Cr(VI) spikes. Oxidation-reduction potentials below the bold diagonal line on Fig. 2 of SW 846 Method 3060A (Eh/pH Phase Diagram, located in Appendix 2 of this SOP) indicates a reducing soil for Cr(VI). The downward slope to the right indicates that the Eh value, at which Cr(VI) is expected to be reduced, decreases with increasing pH. The solubility and quantity of organic constituents will influence reduction of Cr(VI). The presence of H_2S or other strong odors indicate a reducing environment for Cr(VI). In general, acidic conditions accelerate reduction of Cr(VI) in soils, and alkaline conditions tend to stabilize Cr(VI) against reduction. If spike recoveries are not within the recovery limits, the reductive nature of the sample <u>must</u> be documented.

4.5 Post Digestion Spike

4.5.1.1 One post-digestion Cr(VI) matrix spike must be analyzed per batch. The post-digestion matrix spike concentration should be equivalent to 40 mg/kg or twice the

sample concentration observed in the unspiked aliquot of the test sample, whichever is greater. Dilute the sample aliquot to a minimum extent, if necessary, so that the absorbance reading for both the unspiked sample aliquot and spiked aliquot are within the initial calibration curve. A guideline for the post-digestion matrix spike recovery is 85-115% recovery. If not achieved, consider the corrective actions/guidance on data use specified in 4.4.3.1. These digestates may contain soluble reducing agents for Cr(VI), such as fulvic acids.

4.6 Sample Duplicate

4.6.1 Criteria

4.6.1.1 Sample duplicates are required. At least one duplicate sample per 20 samples per matrix is required when matrix spikes are being performed. RPD values are calculated in a manner similar to MS/MSD RPDs:

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

where:
S1 = measured concentration in the initial analysis
S2 = measured concentration in the duplicate analysis

4.6.1.2 Duplicate samples must have a Relative Percent Difference (RPD) of ≤ 20%, if both the original and the duplicate are ≥ four times the laboratory reporting limit. A control limit of ± the laboratory reporting limit is used when either the original or the duplicate sample is < four times the laboratory reporting limit.

4.6.2 Corrective action

4.6.2.1 In general, reanalysis of the samples should occur if duplicate values fail to meet these criteria. Extenuating circumstances or special considerations should be discussed with the Quality Assurance Officer.

4.6.2.2 If a trend in out of control RPD values is observed, the methods used must be examined to determine the source of variance. Once this source is identified, the method must be changed so that samples can be analyzed with a predictable reproducibility. Generally, if recoveries are in control and no analyte of interest was detected in any of the samples, no immediate action will be taken on that sample set.

If integrity of reported sample values is in doubt, re-analysis may be called for. Corrective actions should be discussed with the Quality Assurance Officer.

5. Operation procedures

5.1 Sample Analysis

5.1.1 Analysis sequence

5.1.1.1 A typical batch will consist of:

Prep Blank
Blank Spike
Sample
Sample Duplicate
Sample Spike-Soluble
Sample Spike-Insoluble
Sample Spike Soluble post digestion
Up to 19 more samples

5.2 Analytical Operation

5.2.1 Adjust the temperature setting of each heating device used in the alkaline digestion by preparing and monitoring a temperature blank (a 250 mL beaker filled with 50 mL digestion solution (2.3.6)). Maintain a solution temperature of 90 - 95°C as measured with a NIST-calibrated thermometer or equivalent.

5.2.2 Place 2.5 ± 0.10 g of the as received sample into a clean and labeled 250 mL beaker. The sample should be mixed thoroughly before the aliquot is removed.

5.2.3 Add 50 mL of digestion solution (2.3.6) to each sample. Add ≅ 400 mg of $MgCl_2$ (2.3.4) and 0.5 mL of 1.0 M phosphate buffer (2.3.5). Cover all samples with watch glasses. The Mg^{+2} is used to suppress oxidation of certain forms of Cr(III) (such as soluble forms) that can be oxidized to Cr(VI) during the procedure.

5.2.4 Stir the samples continuously (unheated) for at least five minutes using a stirring bar.

5.2.5 Heat the samples and maintain a temperature range of 90-95°C with constant stirring for 60 minutes at temperature.

5.2.6 Gradually cool each solution to room temperature and transfer it quantitatively to the filtration apparatus with distilled deionized water rinses and filter through a 0.45 µm membrane filter. Rinse the inside of the filter flask and filter pad with deionized water and transfer the filtrate and the rinses to a clean 250-mL beaker. NOTE: The remaining solid after filtration of the matrix spike should be saved for possible use in assessing low Cr(VI) matrix spike recoveries. Store the filtered solid at 4 ± 2°C.

5.2.7 Place a magnetic stirring bar into the sample digest beaker, place the vessel on a stirrer, and, with constant stirring, slowly add concentrated nitric acid solution to the beaker dropwise. Adjust the pH of the solution to 7.5 ± 0.5 and monitor the pH with a pH meter. If the pH of the digest should drop below 7.0, discard the solution and redigest. If overshooting pH 7.5 ± 0.5 occurs repeatedly, prepare a diluted nitric acid solution and repeat digestion procedure. CAUTION: CO_2 will be evolved. This step should be performed in a fume hood.

5.2.8 Remove the stirring bar and rinse, collecting the rinsate in the beaker. Transfer quantitatively the contents of the beaker to a 100 mL volumetric flask and adjust the sample volume to 100 mL (to the mark on the volumetric flask) with deionized water. Mix well.

5.2.9 The sample digestates are now ready to be analyzed. Determine the Cr(VI) concentration in mg/kg by SW-846 Method 7196A (colorimetrically by UV-Vis spectrometry)

5.3 Compound Quantification

5.3.1 The quantitation of Cr(VI) follows LTL-7401 with the following exceptions:

The calibration curve is prepared to go from 0.05-0.50 mg/L.

A 1 cm cell is used

5.3.2 The output from the UV-Vis is calculated in mg/L in 100 mL of solution. To calculate the concentration in the soil:

$$\text{Cr(VI), mg/Kg db} = \frac{A*100*\text{dilution}}{\text{Sa wt,g}*\frac{TS}{100}}$$

where A= concentration in mg/L in the digest

5.3.3 Any sample exceeding the linear range of the calibration curve should be diluted and reanalyzed.

5.3.4 If a sample displays a reading at the instrument that is at odds with the analyst's observation of the color of the sample, an aliquot of the sample should be prepared at the same dilution as the sample and analyzed just like a sample, except that instead of the color reagent, an aliquot of acetone only should be used. This is the turbidity correction for that sample and its value should be subtracted from that of the colorized sample in order to compute the final result.

6. Reports

6.1 Data Packet Organization

6.1.1 The bench sheet generated during the digestion should list the sample ID, analyst, test, date, temperatures, weights, ID of the standard, and any other pertinent information.

6.2 Quality Control Reports

6.2.1 All results for quality control tests are entered into the lab data base using the QC_DB program. Printouts of all data entered must be included in the data packet. The routine minimum is a method blank report, and an MS/MSD or MS/duplicate report. Many analyses will also require an SRM, blank spike or other report.

6.3 Sample Result Reports

6.3.1 Data Qualifying Flags

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

CODE	Definition
U	: The analyte of interest was not detected, to the limit of detection indicated.

Appendix I

QC Summary Table

Laucks Testing Laboratories

Method 3060A/7196A QA Requirements and Corrective Actions

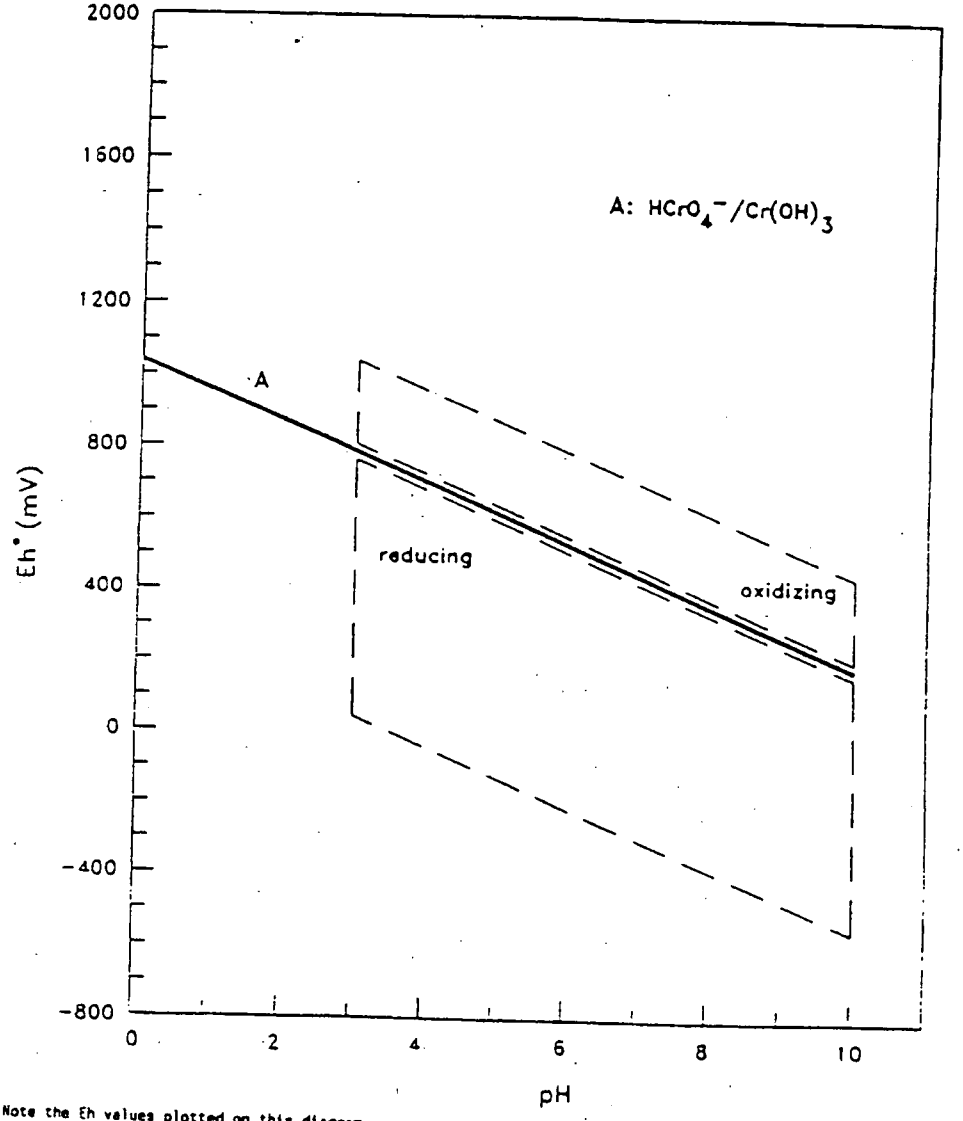
QA Element	Method Criterion	Laucks Criterion	Frequency	Corrective Action	Documentation
Method Blank	<MDL, or 10% of lowest sample	<RL, or 10% of lowest sample	1/20, minimum	Redigest	QC_DB report
Matrix Spike Recovery	75-125	75-125	1/20, minimum	Redigest	QC_DB report
Duplicate % Difference	≤20%, or ± RL	≤20%, or ± RL	1/20, minimum	Redigest	QC_DB report
Blank Spike Recovery	80-120%	80-120%	1/20, minimum	Redigest	QC_DB report
Post digestion Matrix Spike recovery	85-115%	85-115%	1/20, minimum	Redigest	QC_DB report

See text for more specifics on the corrective actions. Results may be reported if the samples are determined to cause matrix effects. However, this entails additional determinations of pH, oxidation-reduction potential and reducing agents (TOC, sulfides, ferrous iron).

Appendix II
Eh/pH Phase Diagram

FIGURE 2
Eh/pH Phase Diagram

The dashed lines define Eh-pH boundaries commonly encountered in soils and sediments.



Note the Eh values plotted on this diagram are corrected for the reference electrode voltage: 244 mV units must be added to the measured value when a separate calomel electrode is used, or 199 mV units must be added if a combination platinum electrode is used.

Appendix III

Flow Charts

FIGURE 1

QUALITY CONTROL FLOW CHART

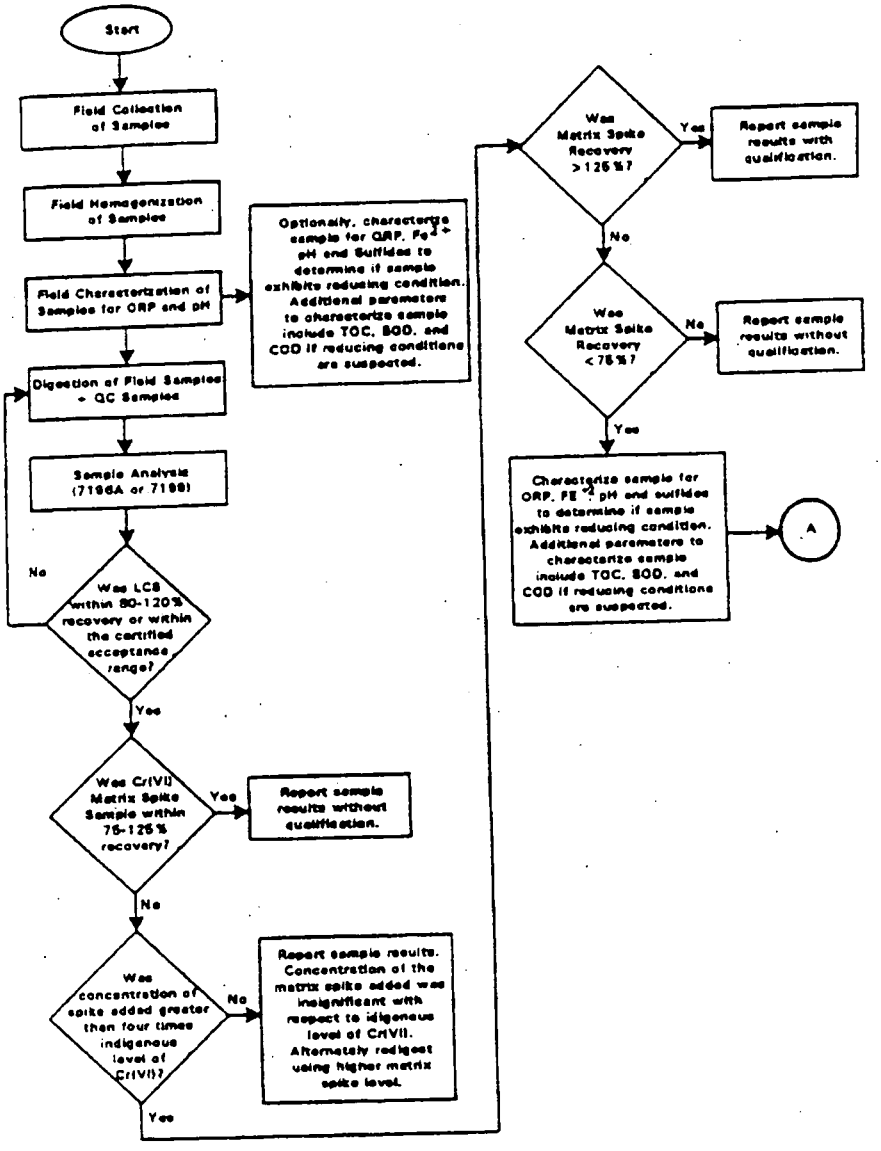
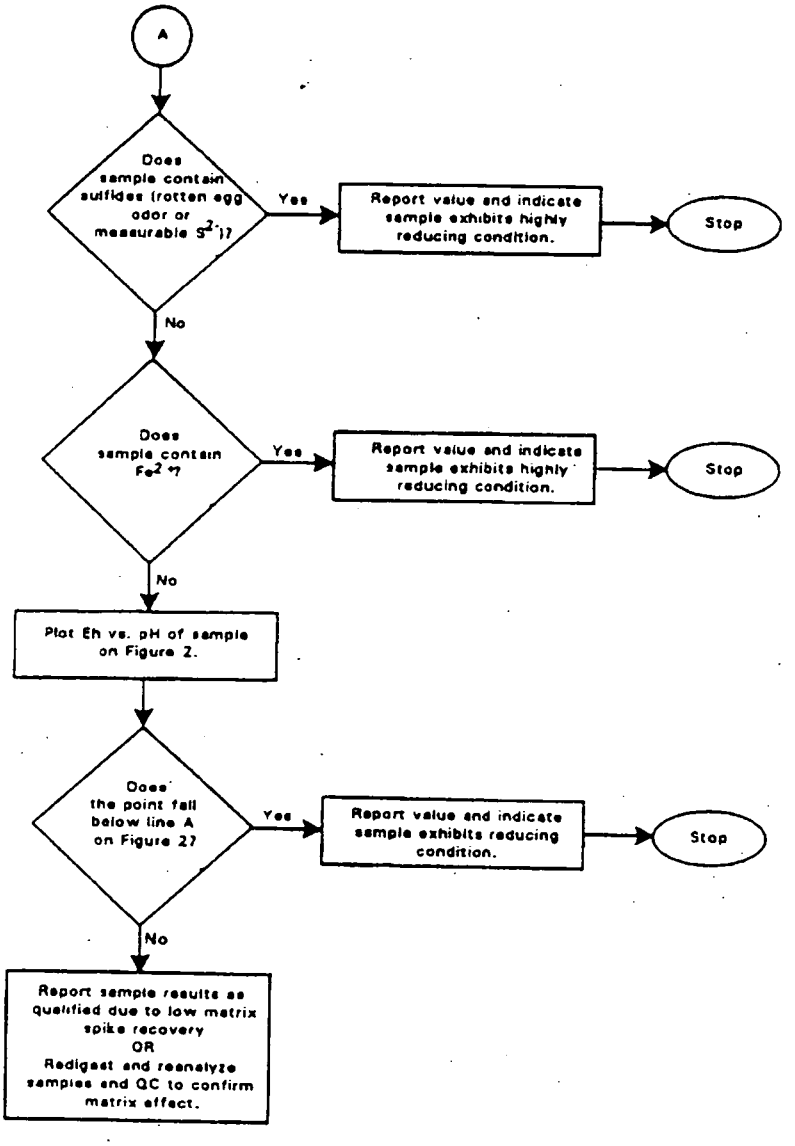
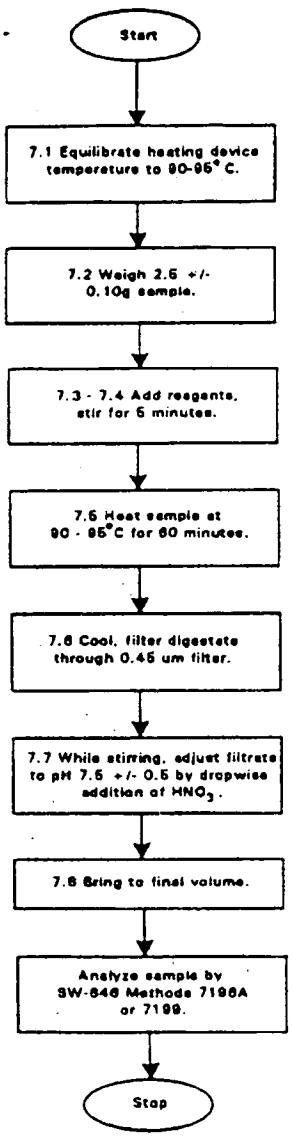


FIGURE 1
QUALITY CONTROL FLOW CHART (Continued)



METHOD 3060
ALKALINE DIGESTION FOR HEXAVALENT CHROMIUM



LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

Method #: LTL-7015

Title: **Acid Digestion of Sediments, Sludge, and Soils by SW846 Method 3050B**

Revision history:

Number	Date
0.0	1/18/99
1.0	3/20/00

Revised by: Stan Dittrick — Date: 3/20/00
Stan Dittrick, Chemist

Reviewed by: Paul Swift — Date: 03/21/2000
Paul Swift, Inorganic Division Manager

Reviewed by: Harry Romberg — Date: 3-20-2000
Harry Romberg, QA Officer

Written by: Kathy Kreps — Date: 03-22-2000
Kathy Kreps, Laboratory Director

Controlled Document
No. 20 Assigned to: Tetra

Table of Contents

1. INTRODUCTION AND SCOPE 3

2. SUMMARY OF METHOD 4

3. SAFETY PROCEDURES 4

4. EQUIPMENT 4

5. REAGENTS 5

6. ACID DIGESTION PROCEDURE 5

7. QUALITY CONTROL 6

8. REFERENCES 7

APPENDIX A, LTL-7015 DIGESTION LOGS 8

APPENDIX B, FLOWCHART FOR LTL-7015 11

APPENDIX C, QUALITY CONTROL SOLUTIONS 13

APPENDIX D, PROCEDURE CHECKLIST 22

1. Introduction and Scope

1.1. This method is an acid digestion procedure used to prepare sediments, sludges, and soil samples for analysis by flame or furnace atomic absorption spectroscopy (FLAA and GFAA, respectively) or by inductively coupled argon plasma spectroscopy (ICP) or inductively coupled argon plasma spectroscopy - mass spectrometry (ICP-MS). Samples prepared by this method may be analyzed by ICP for all the listed metals, or by FLAA or GFAA as indicated below (see also step 2.1):

FLAA/ICP-AES		GFAA/ICP-MS
Aluminum	Magnesium	Arsenic
Antimony	Manganese	Beryllium
*Arsenic	Molybdenum	Cadmium
Barium	Nickel	Chromium
Beryllium	Potassium	Cobalt
Cadmium	*Selenium	Iron
Calcium	Silver	Lead
Chromium	Sodium	Molybdenum
Cobalt	Thallium	Selenium
Copper	*Tin (FLAA only)	*Tin (ICP-MS only)
Iron	Vanadium	Thallium
Lead	Zinc	

* Although Arsenic and Selenium (analyzed by FLAA/ICP) and Tin (analyzed by FLAA or ICP-MS) are not listed in the original SW846 document as analytes that may be analyzed from samples prepared by Method 3050B, Laucks has demonstrated adequate recovery for these analytes from this digestate.

2. Summary of Method

2.1 A representative 1. to 1.5g sample is digested in nitric acid and hydrogen peroxide. The digestate is then either reduced to a low volume or refluxed with hydrochloric acid. Hydrochloric acid is used for flame AA and ICP analyses only. Dilute hydrochloric acid is used as the final reflux acid for (1) the ICP analysis of As and Se, (2) the flame AA or ICP analysis of Ag, Al, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Mo, Na, Ni, Pb, Sb, Sn, Tl, V, and Zn. The hydrochloric acid step is omitted for the furnace AA analysis of As, Be, Cd, Cr, Co, Fe, Pb, Mo, Se, and Tl. The diluted samples have an approximate acid concentration of 5% (v/v).

3. Safety Procedures

3.1 These procedures involve the use of strong and/or hot acid solutions. The analyst must wear eye protection, a lab coat, and gloves to protect against burns.

3.2. These procedures involve hot plates which may present the danger of burns from heated surfaces and electrical hazards. The analyst must take appropriate caution to avoid injury from these sources.

3.3. Samples and spiking solutions may contain high levels of toxic metals and other unknown constituents. The analyst must take every precaution to avoid contact with these potentially hazardous materials and should wash hands thoroughly before taking any breaks, eating, or going home for the day.

3.4. Fertilizer samples are chemically complex matrices and should be handled with extreme caution. Many components found in fertilizer are highly reactive to acid and all acid additions should be made with special care, in a hood, standing back from the sample. Also, these samples have been known to "pop" or explode during heating, so care should be taken not to stand close to or have any body part over the samples while they are being heated.

4. Equipment

- 250-mL Erlenmeyer flasks or equivalent, acid-washed
- Analytical balance capable of accurately weighing to the nearest 0.01 g
- Reflux caps fitted for Erlenmeyer flasks, acid-washed
- Eppendorf or other micropipets
- Hot plate or equivalent heating source, adjustable and capable of maintaining a temperature of 90-95° C
- 100-mL graduated cylinder, acid-washed

- Filter paper, Whatman No. 41 or equivalent
- Glass funnels, acid-washed
- 100-mL volumetric flasks, acid-washed
- Sample digestate bottles, acid-washed
- Thermometer

Note: All glassware used for this digestion must be prepared by the method stated in Inorganics Glass Cleaning Procedures, SOP #: LTL-7003.

5. Reagents

5.1 Reagent Water. Reagent water will be interference-free deionized water. All references to water in the method refers to reagent water unless otherwise specified.

5.2 Nitric acid (concentrated), HNO_3, ACS Reagent grade or better

5.3 Hydrochloric acid (concentrated), HCl, ACS Reagent grade or better

5.4 Hydrogen peroxide (30%), H_2O_2, ACS Reagent grade or better

The method blank prepared along with the samples is used as a contamination check. Since the holding time for all analytes associated with this method have a holding time of 6 months, repreparation would not be an issue if contamination was traced to a specific reagent.

6. Acid Digestion Procedure

See Appendix A for digestion logs.
See Appendix B for flowchart.
See Appendix C for Quality Control solutions
See Appendix D for Procedure Checklist

6.1 Mix the sample thoroughly to achieve homogeneity. For each digestion procedure, weigh 1.00-1.50 g of sample to the nearest 0.01 g directly into an Erlenmeyer flask. For samples with low percent solids, a larger sample size may be used to attain the equivalent of 1.00-1.50 g dry basis as long as digestion is completed. Add appropriate spiking mix aliquots to QC samples at this point.

6.2 Place the reflux caps on the flasks, add 5 mL of concentrated HNO_3, 5 mL H_2O and mix the slurry. Heat the samples to 95° C and reflux without boiling for 15 minutes. Allow the samples to cool. Record the temperature in the digest log. Add 5 mL of concentrated HNO_3, heat 30 min and allow the samples to cool. If brown fumes are generated continue to add

acid, heat and cool until fumes are no longer emitted. Reflux the samples for two hours or allow them to evaporate to 5 mL with reflux caps. Allow the samples to cool.

6.3 Add 2 mL water, 3 mL 30% H_2O_2, heat for 10 minutes and cool the samples. Add 1 mL 30% H_2O_2, reflux for 10 minutes and allow the samples to cool. Repeat the addition of 1 mL H_2O_2 and heat until effervescence is minimal or a maximum of 10 mL H_2O_2 is added. Make sure that sample losses do not occur due to excessive effervescence. Reduce the sample to 5 mL.

6.4 If the sample is being prepared for GFAA or ICP-MS analysis then skip this step otherwise add 10 mL of concentrated HCl. Heat the sample for 15 minutes and allow it to cool.

6.5 Dilute the sample to 100 mL with water in a graduated cylinder and place in an acid-washed digestate bottle. If only one graduated cylinder is to be used to measure all samples, then bring the blank to volume after the rest of the samples. This will help to determine if their is any sample carry over contamination.

Note: If a sample is allowed to go dry at any stage of the digestion procedure, the sample must be discarded and redigested.

6.6 If silicates or other insoluble material that could clog the nebulizer or the graphite furnace autosampler are present in the samples, allow the samples to settle overnight prior to analysis. If there is concern that the insoluble material will not settle out of solution filter the sample with Whatman No. 41 or equivalent filter paper.

6.7 In the digestion log record the start date/time, finish date/time, and the date/time digestates are relinquished. Transfer the samples to the metals analysis department. Have the analyst accepting the digestates initial and date the digestion log

7. Quality Control

7.1 In each batch or SDG consisting of no more than 20 samples, a preparation blank is created. This consists of an empty, acid-washed Erlenmeyer flask to which the appropriate reagents are added and digested in exactly the same manner as a sample The blank should be labeled on the digestate bottle in the following way: B, date, instrument, S or W (for soil or water) and the sequence number of the digestion. Example: B111794ICPS01.

7.2 Spiked samples should be employed to determine accuracy. In each batch or SDG consisting of no more than 20 samples, either matrix spike / sample duplicate (MS/Dup) or matrix spike / matrix spike duplicate (MS/MSD) samples will be prepared. For SW846-

specified quality control measures, MS/Dup samples will be prepared in the following way. Dispense one sample in triplicate. Two will be digested exactly as any other sample. The third must be spiked with the appropriate spike solutions determined by the analyst, and then treated as any other sample. All glassware and digestate bottles must be marked appropriately. If MS/MSD samples are required instead of the SW846-specified MS/Dup, both the second and third aliquots must be spiked and all glassware and digestate bottles marked appropriately. See Appendix C for instructions on the preparation and use of QC solutions. Spiking levels presented are to be used only as guidance. The actual analytes and concentrations may vary.

7.3 Determine the analytes required for analysis by consulting sample work order information. Each spiking solution is given its own unique number according to the page and line of the standards logbook which it is entered into. This number and the volume dispensed must be clearly recorded on the digestion log. A sample page from the standards logbook is included in the Appendix C.

7.4 In each batch or SDG consisting of no more than 20 samples, either a Blank Spike (BLK SPK) or a Laboratory Control Sample (ICP or GF LCSS) must be digested in exactly the same manner as a sample. A Blank Spike consists of an empty, acid-washed Erlenmeyer flask to which the appropriate spikes (determined as outlined in Section 7.3) and reagents are added. The Certified Value and Advisory range for each analyte in a particular LCSS can be accessed from the Quality Control Database.

8. References

8.1 USEPA, Test Methods for Evaluating Solid Waste Physical/Chemical Methods, USEPA SW846, most recent version, Method 3050B.

Appendix A, LTL-7015 Digestion Logs

LAUCKS TESTING LABORATORIES, INC.		ICP DIGESTION LOG	Place a √ in the box next to the method performed.			
	Digest start : date/time	Digest finish : date/time	SW-846 3005A		ILM04.0-water	
SDG #:			SW-846 3010A		ILM04.0-soil	
Analyst:			SW-846 3050A		SW-846 3050B	

	Sample ID	Sample Size (g:mL)	Final Volume (mL)	Color		Clarity		Text	Art	Comments	Bottle #
				Bef	Aft	Bef	Aft				
01											
02											
03											
04											
05											
06											
07											
08											
09											
10											
11											
12											
13											
14											
15											
16											
17											
18											
19											
20											
21											
22											
23											
24											

Chain of Custody	Digest Relinquished Date / Time : Analyst	Digest Accepted Analyst	Digest Disposed Date	Hot Plate Temperature, °C
Reagents Lot Number	HNO_3	HCl	H_2O_2	

Color 1=Red, 2=Blue, 3=Yellow, 4=Green, 5=Orange, 6=Violet, 7=White, 8=Brown, 9=Grey, 10=Black, 11=Colorless
Clarity 1=Clear, 2=Cloudy, 3=Opaque
P:\metals\bnchshee\icpdig.doc
Texture F=Fine (powdery), M=Medium (sandy), C=Coarse (rocky) Artifacts: If "Yes", give description in the comments field.

P:\metals\bnchshee\icpdig.doc

LAUCKS TESTING LABORATORIES, INC.		GRAPHITE FURNACE - DIGESTION LOG	Place a √ in the box next to the method performed.			
	Digest start : date/time	Digest finish : date/time	SW-846 3020A		ILM04.0-water	
SDG #:			SW-846-7060A		ILM04.0-soil	
Analyst:			SW-846 3050A		SW-846 3050B	

	Sample ID	Sample Size (g:mL)	Final Volume (mL)	Color		Clarity		Text	Art	Comments	Bottle #
				Bef	Aft	Bef	Aft				
01											
02											
03											
04											
05											
06											
07											
08											
09											
10											
11											
12											
13											
14											
15											
16											
17											
18											
19											
20											
21											
22											
23											
24											

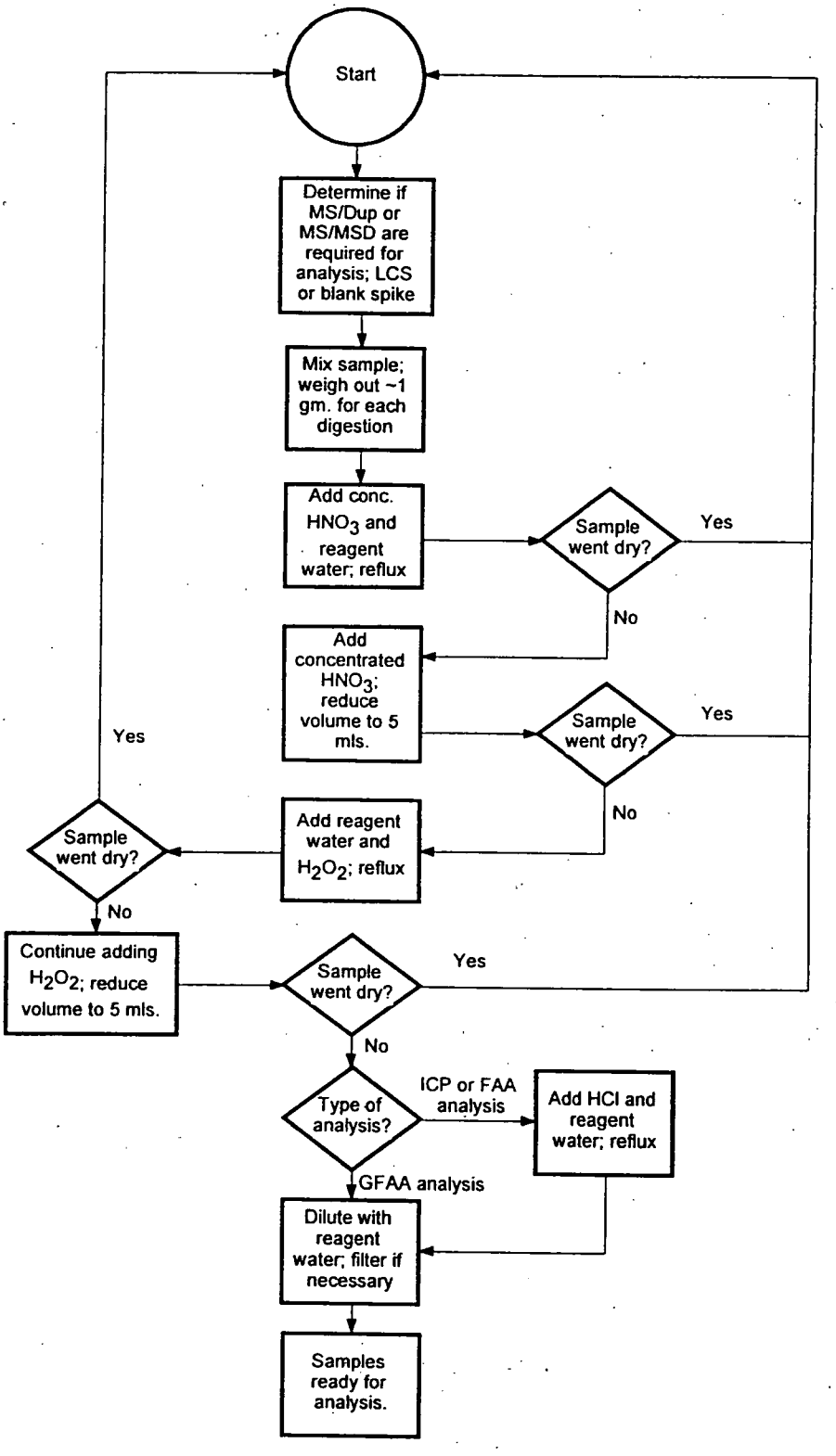
Chain of Custody	Digest Relinquished Date / Time : Analyst	Digest Accepted Analyst	Digest Disposed Date	Hot Plate Temperature, °C

Reagents Lot Number	HNO_3	HCl	H_2O_2

Color 1=Red, 2=Blue, 3=Yellow, 4=Green, 5=Orange, 6=Violet, 7=White, 8=Brown, 9=Grey, 10=Black, 11=Colorless
Clarity 1=Clear, 2=Cloudy, 3=Opaque
Texture F=Fine (powdery), M=Medium (sandy), C=Coarse (rocky) Artifacts: If "Yes", give description in the comments field.

P:\metals\benchsee\gfdig.doc

Appendix B, Flowchart for LTL-7015


Start
Determine if MS/Dup or MS/MSD are required for analysis; LCS or blank spike
Mix sample; weigh out ~1 gm. for each digestion
Add conc. HNO_3 and reagent water; reflux
Sample went dry?
Yes
No
Add concentrated HNO_3; reduce volume to 5 mls.
Sample went dry?
Yes
No
Add reagent water and H_2O_2; reflux
Sample went dry?
Yes
No
Continue adding H_2O_2; reduce volume to 5 mls.
Sample went dry?
Yes
No
Type of analysis?
ICP or FAA analysis
Add HCl and reagent water; reflux
GFAA analysis
Dilute with reagent water; filter if necessary
Samples ready for analysis.

Appendix C, Quality Control Solutions

ICP Spikes

- The CLP spike solution for ICP digest was made by diluting 10 mLs of CLPP-SPK-1™, 10 mLs of CLPP-SPK-2™, and 10 mLs of CLPP-SPK-3™ to 100 mLs with Type II water.

- Add 1000 µLs of CLP spike solution to water samples and 2000 µLs of CLP spike solution to soil samples prior to digestion.

ICP Analytes

Analyte	Concentration in Digest (ppb)	Analyte	Concentration in Digest (ppb)
Ag	50	Fe	1000
Al	2000	Mn	500
As	2000	Ni	500
Ba	2000	Pb	500
Be	50	Se	2000
Cd	50	Sb	500
Co	500	Tl	2000
Cr	200	V	500
Cu	250	Zn	500

Table based on a final volume of 100 mL for water digests, and 200 mL for soil digests.

GRAPHITE FURNACE SPIKES

- Add the analytes listed in the table below to a 100 mL volumetric flask which contains ~50 mL of Type II water and 5 mL of HNO_3.

Analyte	Volume	Stock Concentration
As	400 µL	1000 ppm
Pb	200 µL	1000 ppm
Se	100 µL	1000 ppm
Tl	500 µL	1000 ppm

- Dilute to 100 mL with Type II water. This spike solution now contains:

4.0 ppm	Arsenic
2.0 ppm	Lead
1.0 ppm	Selenium
5.0 ppm	Thallium

- Add 1000 µL of Spike Solution to water samples and 2000 µL to soil samples prior to digestion.

ICP
CLP-LOW SPIKES

- Add the analytes from the two tables below into separate 100 mL volumetric flasks which contain ~50 mL of Type II water and 5 mL of HNO_3.

ICP-1

Analyte	Volume	Stock Concentration	Spike Concentration
Sb	500 µL	1000 ppm	5 ppm
Al	5000 µL	1000 ppm	50 ppm
Ba	2000 µL	1000 ppm	20 ppm
Be	100 µL	1000 ppm	1 ppm
Cr	500 µL	1000 ppm	5 ppm
Co	1000 µL	1000 ppm	10 ppm
Cu	500 µL	1000 ppm	5 ppm
Fe	2500 µL	1000 ppm	25 ppm
Mn	500 µL	1000 ppm	5 ppm
Ni	1000 µL	1000 ppm	10 ppm
V	1000 µL	1000 ppm	10 ppm
Zn	1000 µL	1000 ppm	10 ppm

ICP-2

Analyte	Volume	Stock Concentration	Spike Concentration
Ag	500 µL	1000 ppm	5 ppm

- Add 1000 µL of ICP-1 and 1000 µL of ICP-2 spike solutions to water samples prior to digestion. For soil samples, add 2000 µL of each spike solution prior to digestion. The final digest will contain:

Analyte	Concentration in Digest	Analyte	Concentration in Digest
Sb	50 ppb	Al	500 ppb
Ba	200 ppb	Be	10 ppb

Cr	50 ppb	Co	100 ppb
Cu	50 ppb	Fe	250 ppb
Mn	50 ppb	Ni	100 ppb
V	100 ppb	Zn	100 ppb
Ag	50 ppb		

Table based on final volume of 100 mL for water digests, and 200 mL for soil digests.

GRAPHITE FURNACE
CLP-LOW SPIKES

- Add the analytes listed in the table below to a 100 mL volumetric flask which contains ~50 mL of Type II water.

Analyte	Volume	Stock Concentration	Spike Concentration
As	100 µL	1000 ppm	1000 ppb
Se	500 µL	1000 ppm	5000 ppb
Tl	500 µL	1000 ppm	5000 ppb
Pb	250 µL	1000 ppm	2500 ppb
Cd	100 µL	1000 ppm	1000 ppb

- Add 1000 µL of Spike Solution to water samples and 2000 µL to soil samples prior to digestion. The final digest will contain*:

10 ppb Arsenic
50 ppb Selenium
50 ppb Thallium
25 ppb Lead
10 ppb Cadmium

*Based on 100 mL final volume for water digests and 200 mL for soil digests.

ICP LCSW

The LCSW for the ICP digest was made by diluting 10 mLs of CLP-SPIKE and 50 mLs of LTL-6 solutions, and 2 mLs of conc. HNO_3 to 1000 mLs with type II water. A 100 mL aliquot of this solution was then digested.

ICP Analytes

Analyte	Concentration in Digest (ppb)	Analyte	Concentration in Digest (ppb)
Al	2000	Pb	500
Ba	2000	Mg	50000
Be	50	Mn	500
Cd	50	Ni	500
Ca	50000	K	50000
Cr	200	Ag	50
Co	500	Na	50000
Cu	250	V	500
Fe	1000	Zn	500
Sb	500		

Other Analytes

Analyte	Concentration in Digest (ppb)	Analyte	Concentration in Digest (ppb)
As	2000	Tl	2000
Se	2000		

Tables based on a final volume of 100 mL.

Graphite Furnace LCSW

- Add the analytes listed in the table below to a 100 mL volumetric flask which contains ~50 mL of Type II water and 2 mL of HNO_3. Dilute to volume with Type II water.

Analyte	Volume	Stock Concentration
As	500 µL	1000 ppm
Pb	500 µL	1000 ppm
Se	250 µL	1000 ppm
Tl	250 µL	1000 ppm
Cd	100 µL	1000 ppm

- Dilute to 100 mL with Type II water. This LCSW stock solution now contains:

5.0 ppm	Arsenic
5.0 ppm	Lead
2.5 ppm	Selenium
2.5 ppm	Thallium
1.0 ppm	Cadmium

The LCSW for the GFAA digest was made by diluting 10 mLs of LCSW stock solution and 20 mLs of conc. HNO_3 to 1000 mLs with type II water. A 100 mL aliquot of this solution was then digested.

Graphite Furnace Analytes

Analyte	Concentration in Digest (ppb)	Analyte	Concentration in Digest (ppb)
As	50	Tl	25
Pb	50	Cd	10
Se	25		

Tables based on a final volume of 100 mL.

PDCA
SPIKES and LCSW

The LCSW for the PDCA extraction was make by adding 10 mL SPEX ICV-1™ solution to 100 mL of Type II water prior to extraction. The spike was made by adding 1000 µL of CLP SPK solution to a 100 mL aliquot of the sample prior to extraction.

ICP Analytes

Analyte	Concentration in Digest	Analyte	Concentration in Digest
Cd	50 ppb	Mn	500 ppb
Co	500 ppb	Ni	500 ppb
Cu	250 ppb	Ag	50 ppb
Fe	1000 ppb	V	500 ppb
Zn	500 ppb		

Graphite Furnace Analytes

Analyte	Concentration in Digest	Analyte	Concentration in Digest
Pb	500 ppb	Tl	2000 ppb
Se	2000 ppb		

Tables based on a final volume of 100 mL.

APPENDIX D, PROCEDURE CHECKLIST

SW846-3050B

ICP/FAA

- [ ] 1.0-2.0g Sample
- [ ] Add Spikes
- [ ] 5 mL H_2O
- [ ] 5 mL HNO_3
- [ ] Heat 15 min
- [ ] Cool
- [ ] 5 mL HNO_3
- [ ] Heat 30 min
- [ ] Repeat (5 mL HNO_3) until no brown fumes exist
- [ ] Reduce to 5 mL or heat 2 hours
- [ ] Cool
- [ ] 2 mL H_2O
- [ ] 3 mL H_2O_2
- [ ] Heat until effervescence subsides
- [ ] Cool
- [ ] Repeat with 1 mL aliquots H_2O_2 until effervesence is minimal or 7 times
- [ ] Reduce to 5 mL or heat 2 hours
- [ ] Cool
- [ ] 10 mL HCl
- [ ] Heat 15 min
- [ ] Cool
- [ ] Final Volume 100 mL

GFAA/ICP-MS

- [ ] 1.0-2.0g Sample
- [ ] Add Spikes
- [ ] 5 mL H_2O
- [ ] 5 mL HNO_3
- [ ] Heat 15 min
- [ ] Cool
- [ ] 5 mL HNO_3
- [ ] Heat 30 min
- [ ] Repeat (5 mL HNO_3) until no brown fumes exist
- [ ] Reduce to 5 mL or heat 2 hours
- [ ] Cool
- [ ] 2 mL H_2O
- [ ] 3 mL H_2O_2
- [ ] Heat until effervescence subsides
- [ ] Cool
- [ ] Repeat with 1 mL aliquots H_2O_2 until effervesence is minimal or 7 times
- [ ] Reduce to 5 mL or heat 2 hours
- [ ] Cool
- [ ] Final Volume 100 mL

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-7105

Title: Method Protocols for the Analysis of Metals by ICP Using SW 846 6010B

Revision history:

Number	Date
0	1/29/98
1	03/24/00
2	12/21/01

Revised by: _____ Date: 1/8/02
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No. 20 Assigned to: TETR

Table of Contents

1. INTRODUCTION AND SCOPE 4

1.1 SCOPE 4
1.2 SAMPLE COLLECTION, SAMPLE STORAGE, HOLDING TIMES 4
1.3 DEFINITION OF TERMS 4

2. EQUIPMENT LIST AND STANDARDS 6

2.1 INSTRUMENTATION 6
2.2 COMPUTER AND SOFTWARE 6
2.3 STANDARDS 6

3. SAFETY PRECAUTIONS AND WASTE DISPOSAL 7

3.1 SAFETY 7
3.2 WASTE DISPOSAL 7

4. CALIBRATION AND QUALITY CONTROL 7

4.1 METHOD DETECTION LIMIT STUDY 7
4.2 LINEAR RANGE STUDY 7
4.3 INITIAL CALIBRATION AND PRELIMINARY CALIBRATION CHECK STANDARDS 8
4.4 INITIAL CALIBRATION STANDARD ICV: 8
4.5 INITIAL CALIBRATION BLANK 9
4.6 CONTINUING CALIBRATION VERIFICATION (CCV) AND BLANK (CCB) 9
4.7 INTERFERENCE CHECK SOLUTIONS A (ICSA) AND AB (ICSAB) 10
4.8 METHOD BLANKS 10
4.9 LABORATORY CONTROL SAMPLE 11
4.10 MATRIX SPIKE 11
4.11 MATRIX SPIKE DUPLICATE/SAMPLE DUPLICATE 13
4.12 SERIAL DILUTION 14

5. OPERATION PROCEDURES 14

5.1 INSTRUMENTAL CONDITIONS 14
5.2 CALIBRATION 14
5.3 ANALYSIS SEQUENCE 14
5.4 ANALYTICAL OPERATION 14

6. REPORTS 15

6.1 DATA PACKET ORGANIZATION 15
6.2 FLAGGING 15
6.3 CONTROL CHART(S) 15
6.4 REFERENCES: 15

APPENDIX I 16

QC SUMMARY TABLE 16

APPENDIX II 19

FLOW CHART 19

APPENDIX III 21

SAMPLE HANDLING AND PRESERVATION 21

APPENDIX IV **22**
ANALYTICAL SEQUENCE 22

1. Introduction and Scope

1.1 Scope

1.1.1 This method is to be used as a supplement to the instrumental SOP, LTL-7106, in order to follow the method requirements of SW 846 6010B for ICP analysis. Operating parameters are to be followed in the individual instrument SOP.

1.1.2 The analyst should become familiar with SW 846 protocols prior to using this SOP.

1.1.3 This method is restricted to use by, or under the supervision of analysts experienced in the technique described. Each analyst performing this method must have demonstrated the ability to perform the described analysis.

1.2 Sample Collection, Sample Storage, Holding Times

1.2.1 Samples are to be collected in either glass or plastic containers. Water samples are to be preserved to a pH < 2 using nitric acid. A one-liter sample bottle is sufficient volume for analysis. Soil samples do not require preservation but need to be stored at 4° C. At least 200 grams of sample should be collected. The holding time for ICP metals is 6 months. See Appendix III for Sample Handling and Preservation Table.

1.3 Definition of Terms

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

ICV - Initial Calibration verification - This is a standard run immediately following the initial calibration. The ICV is made from an independent source. Agreement within 10% and a relative standard deviation less than 5% RSD from replicate (minimum of two) integrations is required.

ICB - Initial calibration blank - An instrument blank is made up in the same way as calibration standards, without target analytes.

CCV - Continuing calibration verification. - This is a standard analyzed after every 10 samples during the analysis sequence to determine whether the instrument or system has remained in calibration. Agreement within 10% and less than 5% RSD from replicate (minimum of two) integrations, is required.

CCB - Continuing Calibration Blank - This is a blank that is used to determine if there is carry-over between sample injections. A CCB succeeds every CCV, every 10 samples.

CRDL - Contract Required Detection Limit - The minimum level of detection at ±20% analyte recovery. This limit represents the analyte concentrations below which data must be evaluated and qualified as estimated concentrations. The commercial CRDL solution may be used as a check standard, but must be fortified with As, Pb, and Se to a final concentration before analysis of 10ug/L.

DIW - Deionized water - Lab reagent water. This water should be free of virtually all analytes.

IEC - Interelement Correction Factors - IECs are experimentally-determined constants used by the calibration modeling software in estimating the extent of interference caused by IEC elements on the other analyzed elements. This list is to include the nine interferents listed in SW846 Method 6010B - Al, Ca, Cr, Cu, Fe, Mg, Mn, Tl, and V.

ICSA - Interference Check Solution A - This solution is prepared to contain known concentrations of interfering elements that will provide an adequate test of the correction factors.

ICSAB - Interference Check Solution AB - This solution is prepared by spiking the ICSA with known quantities of analyte. Adequate recovery of the analytes within this interfering matrix indicates proper application of the correction factors.

IDL - Instrument detection limit - The lowest concentration of a target analyte that is detectable. The IDL is three times the average standard deviation of seven replicates at 2 to 5 times the estimated IDL over three non-consecutive days. Used as a starting point for selecting MDL study spiking levels. IDL should be determined quarterly.

Method Blank Sample / Preparation Blanks Sample- A sample containing only reagent and representative matrix (DIW or blank solid material, as appropriate). This sample is to be carried through the entire preparation procedure and analyzed as a regular sample. The level of analytes in this sample must not exceed one-half the concentration requested as reporting limits for the project, or twice the MDL, whichever is lower. Corrective action will be either qualification of data associated with that blank sample or repreparation/reanalysis of the associated samples.

MS - Matrix spike. The matrix spike sample is to be prepared such that the amounts of analyte added are near the action levels of concern for the project, but above 20x the MDLs. Preparation of the matrix spike sample is described in the appropriate SOP.

MDL - Method detection limit - The lowest concentration which will yield a positive result that is greater than zero at a known level of confidence. The MDL is preparation specific and empirically determined by Laucks.

LCS - Laboratory Control Sample - This is a material of approximately the same matrix as the samples, containing a known and usually certified amount of target analyte and which is prepared and analyzed in the same manner as a typical sample. This sample is used to demonstrate that the analytical system is in control. It may be considered to be a blank spike for most inorganic analyses and is preferred over artificially spiking blank materials.

Post digestion spike - The sample selected for QC analysis will be post-spiked and analyzed in the event that the matrix spike sample exceeds recovery criteria. The final spike level should be identical to the predigestion spike, near the action levels of concern for the project but above 20x the MDL.

QC period - Quality control period - An analysis sequence initiated by the analysis of one or more standards, followed by samples, and terminated with a standard and blank analysis.

RSD or %RSD - Relative standard deviation or percent relative standard deviation - The ratio of the standard deviation of a set of values to the mean of the set of values. A measure of the similarity of the values one to another.

Sequence - A set of sample digests and standard solutions introduced into an instrument in a chronologically continuous group.

2. Equipment List and Standards

2.1 Instrumentation

2.1.1 Thermo Jarrell Ash ICAP 61E Trace Analyzer or equivalent.

2.2 Computer and software

2.2.1 Pentium 75 computer (or faster), running Thermospec software version 6.2 or later.

2.3 Standards

2.3.1 SW 846 Method 6010B requires the use of one standard and a blank. See the instrument SOP for standards and their preparation.

3. Safety precautions and Waste Disposal

3.1 Safety

3.1.1 See the instrument SOP (LTL-7106) for detailed safety precautions

3.1.2 All standards, samples and sample solutions should be handled as if they are hazardous substances.

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

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

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

3.1.6 Caution should be used when handling acidic digestates.

3.2 Waste Disposal

3.2.1 Waste segregation and disposal from the point of collection is further covered in the Laucks SOP on waste segregation and disposal, LTL-2001. All acidified samples and standards should be neutralized prior to sewer disposal.

4. Calibration and Quality Control

4.1 Method Detection Limit Study

4.1.1 Prior to the analysis of any samples, it is necessary to establish method detection limits. This procedure is fully described in the Laucks SOP on performing MDL studies. Briefly, it involves the analysis of 7 replicate samples spiked at a concentration approximately 3 to 5 times the estimated method detection limit. A Student's T-test is then applied to these measured values to calculate the MDL.

4.2 Linear range study

4.2.1 The upper limit of the linear dynamic range must be established by running a multi-point calibration using 4 standards and then running an upper range standard. The upper range limit should be an observed signal with no more than a 10% variation from the true value of the upper range standard. All samples with elements exceeding the level of the upper range standard must be diluted.

4.3 Initial Calibration and Preliminary Calibration Check Standards

4.3.1 Analyze standard solutions consisting of a blank and three standards. The calibration curve must be verified by running an Initial Calibration Standard (ICV) and obtaining agreement within 10% of the expected concentration and a 5% RSD for replicate integrations.

4.3.2 One check standard at the CRDL is to be analyzed, and the recovery criteria shall be ±20%.

4.3.3 One check standard at or below the linear range for each analyte may be analyzed in order to confirm the linearity of the calibration curve between the single-point high standard and the estimated linear range maxima. Recovery of 90-110% allows the use of the calibration curve in this region to quantify sample results.

4.3.4 Criteria and Corrective Action:

4.3.4.1 Calibration - The standard curve is validated by evaluating the ICV and the subsequent CCVs. If the corresponding control limits for the ICV and CCV are exceeded, then the sample analysis must be discontinued, the cause determined and the instrument recalibrated. All samples following the last acceptable ICV, CCV must be reanalyzed.

4.3.4.2 Linear range standard - If this standard is not included in the analysis then any data acquired with concentration values above the high standard but within the linear range of the instrument must be qualified as estimated quantities. Preferably, the samples would be diluted and reanalyzed

4.3.4.3 CRDL - If the stated criterion cannot be met then the reporting limit will be adjusted such that this analysis will be in control. This may involve adjusting the CRDL concentration for individual analytes which failed to meet acceptance followed by reanalysis.

4.4 Initial Calibration Standard ICV:

4.4.1 Immediately after the calibration curve, an ICV is analyzed.

4.4.1.1 The calculated concentration of the standard must be within 10% of the expected value and the RSD must be less than 5% for replicate integrations.

4.4.2 Corrective Action:

4.4.2.1 If the ICV criteria are not met, no samples can be analyzed. Perform system maintenance and re-check the ICV. If the criteria still cannot be met, the system must be recalibrated.

4.5 Initial Calibration Blank

4.5.1 After the analysis of the high standard, an instrument blank (ICB) is analyzed. The levels of target analytes in the ICB should not exceed ½ of the reporting limit.

4.5.2 Corrective Action:

4.5.2.1 The source of contamination must be identified and corrected before proceeding with the analysis. If the blank is less than 1/10 the concentration of the action level of interest, and no sample is within 10% of the action limit, analyses need not be rerun and recalibration need not be performed before continuation of the run.

4.6 Continuing Calibration Verification (CCV) and Blank (CCB)

4.6.1 A continuing calibration verification standard is analyzed after every 10 samples. Immediately following the CCV, a blank solution is analyzed. In addition, this standard and blank must be the last samples analyzed in the run. The CCV must also be prepared from an independent source.

4.6.1.1 The CCV must fall within ± 10% of the true value and the RSD must be less than 5% for replicate integrations.

4.6.1.2 The levels of target analytes in the CCB should not exceed one half of the reporting limit.

4.6.2 Corrective action:

4.6.2.1 If CCV limits are exceeded, check calculations or perform instrument maintenance. Recalibrate and reanalyze. No sample results may be reported that are not bracketed by a successful calibration and CCV/CCB which are in control or by preceding and following CCV/CCBs which are within limits.

4.6.2.2 If the CCB analyte levels exceed one half of the reporting limit, terminate the analysis, correct the problem, recalibrate, and reanalyze the previous 10 samples. If the blank is less than 1/10 the concentration of the action level of interest, and no sample is within ten percent of the action limit, analyses need not be rerun and recalibration need not be preformed before continuation of the run.

4.7 Interference Check Solutions A (ICSA) and AB (ICSAB)

(ICSA):

4.7.1 At the beginning and at the end of each run, an interference check solution A is analyzed. This solution contains interfering elements only. All other elements are not present in the solution. All elements not present should show a recovery of no greater than ± the CRDL.

4.7.2 Corrective Action:

4.7.2.1 If the analytes do not recover within the specified control limits, then the system is out of control. The problem needs to be identified and corrected prior to beginning another run.

(ICSAB):

4.7.3 At the beginning and end of each analytical sequence an ICSAB must be analyzed. Analytes must recover between 80-120%.

4.7.4 Corrective Action:

4.7.4.1 If the analytes do not recover within the specified control limits, then the system is out of control. The problem needs to be identified and corrected prior to beginning another analysis.

4.8 Method Blanks

4.8.1 Method blanks are used to verify contamination free reagents and apparatus. They are prepared with every set of samples prepared at the same time or at least one blank every 20 samples, which ever is more frequent. Any analyte response above the MDL is reported. For a method blank to be acceptable for use with the accompanying samples, the concentration of the blank of any analyte of concern should not be higher than the highest of either:

(1) One-half of the reporting limit, which is approximately twice the value of the MDL, or
(2) Five percent of the regulatory limit for that analyte, or
(3) Five percent of the measured concentration in the sample.

4.8.2 Corrective Action:

4.8.2.1 Corrective action may necessitate re-preparation and re-analysis of the sample set. For example if an analyte was detected in the blank but not in any of the associated samples then sample group may not require re-analysis. In any case,

if re-preparation and re-analysis is not being undertaken, the analyst must first discuss the issue with the Quality Control Officer. It is the laboratory's responsibility to ensure that method interference caused by contaminants in acids, solvents, reagents, glassware, and other sample processing hardware leading to discrete artifacts and/or elevated baselines in the analytical run be minimized. In the extreme case of chronic contamination, blanks may have to be analyzed from each stage of the sample processing to determine the contamination source so it can be eliminated. In all cases where blank contamination exceeds the control limit, a narrative comment must be made which documents the corrective actions taken.

4.9 Laboratory Control Sample

4.9.1 The LCS is made from an independent source of the same matrix (soil or water) and is carried through the entire digestion procedure. The LCSW is to be spiked at a level near the action levels of concern for the project but above 20x the MDL. The SDG Routing Form will indicate appropriate spike level. A LCS is performed with each digestion batch. At a minimum, LCSW (water) control limits, are 80% to 120%. Control limits for the LCSW will be empirically determined and must be within the method specified control limit noted above.

4.9.2 The manufacturer supplies LCSS (soil) control limits. LCSS control limits are not derived by the laboratory due to the small number of data points available from each lot of certified material.

4.9.3 Corrective Action

4.9.3.1 If the LCS is not within the required control limits but the matrix spike is in control, corrective action may not be required unless otherwise specified. If both LCS and matrix spikes exceed limits, particularly if the recoveries are low <u>or</u> there are reportable quantities of the affected target analytes, then a redigestion will occur for the affected analytes. Other extenuating circumstances may exist that allow for exceptions without redigestion and reanalysis but they must first be approved by QA.

4.10 Matrix Spike

4.10.1 Unless specific samples are specified for use as matrix Spike Samples, a sample is chosen at random from the samples to be analyzed, and an aliquot of spiking solution is added to this sample prior to preparation. Spike samples will be identified on the SDG Routing Forms as required. The analyst should attempt to avoid selecting samples that are identified by the client as blanks.

4.10.2 The matrix spike sample is to be prepared such that the amounts of analyte added are near the action levels of concern for the project, but above 20x the MDLs. Preparation of the matrix spike sample is described in LTL-7106.

4.10.3 As the purpose of the matrix spike is to test the system under "typical" conditions, the analyst may also avoid selecting the most difficult sample of the batch for spiking. It is not always required that a matrix spike analysis be performed with each preparation/analysis batch, however, the minimum frequency for MS analysis is 1 each per 20 samples per matrix. This will be best accomplished by running one with every batch for many analyses. This matrix spike sample is used to evaluate the matrix effect of the sample upon recovery of the analytes. The recovery of spike analytes is calculated as follows:

$$recovery,\% = \frac{(SS - S)}{SA} * 100$$

where:

SS = concentration in spiked sample
S = native concentration in unspiked sample
SA = spike added, the amount of spiking material actually added calculated on the sample basis.

4.10.4 For ICP, control limits for spike recoveries will be 75-125% unless otherwise specified in the project specific QAPP. In-house control limits are based on historical performance.

4.10.5 The recovery criteria are detailed in the QC Database and will change from time to time.

4.10.6 Corrective Action:

4.10.6.1 Samples with spike recoveries outside control limits will be reviewed for possible corrective action. Corrective action will first involve recalculation, followed by possible re-preparation, and/or reanalysis. This process should also look at the recovery of matrix spiking compounds from the SRM and/or blank spike analysis.

4.10.6.2 In some cases a Post Digestion Spike is required when matrix interference is suspected. The sample selected for QC analysis will be post-spiked and analyzed in the event that the matrix spike sample exceeds recovery criteria. The final spike level should be identical to the predigestion spike, near the action levels of concern for the project but above 20x the MDL. The same 75-125% recovery criteria exist for this spike as for the matrix spike or a matrix effect is expected. In all cases a narrative explanation of the condition is

required to detail the corrective actions taken. Data reported in validatable packages will be flagged with an "N" indicating the out-of-control event.

4.11 Matrix Spike Duplicate/Sample Duplicate

4.11.1 Method QC consists of MS/MSD. A duplicate maybe performed instead of a MSD. Other types of QC can performed at the client's request.

4.11.2 Criteria

4.11.2.1 At least one matrix spike duplicate sample per 20 samples per matrix is required when matrix spikes are being performed. RPD values are calculated in a manner similar to MS/MSD RPDs:

$$RPD = \frac{|SS - SSD|}{(SS + SSD)/2} * 100$$

where:
SS = concentration in spiked sample
SSD = concentration in matrix spiked duplicate sample

4.11.2.2 For sample concentrations greater than ten times the IDL, control limits for RPD of duplicates will be ±20% unless otherwise specified in the project specific QAPP. For sample concentrations less than ten times the IDL, control limits for the difference will be ± the reporting limit. In-house control limits are based on historical performance. The RPD control limits are detailed in the current QC Database and will change from time to time.

4.11.3 Corrective Action:

4.11.3.1 If a trend in out of control RPD values is observed, the methods used must be examined to determine the source of variance. Once this source is identified, the method must be changed so that samples can be analyzed with a predictable reproducibility. Generally, if recoveries are in control and no analyte of interest was detected in any of the samples, no immediate action will be taken on that sample set. If integrity of reported sample values is in doubt, re-analysis may be called for. Corrective actions should be discussed with the Quality Control Officer. In a validatable package, data associated with an out-of-control sample duplicate will be flagged with an "*".

4.12 Serial Dilution

4.12.1 A five-fold dilution is performed on the QC sample in each analytical batch. The difference between the initial value and the serial dilution should agree within 10%. In a validatable package, if the difference is greater than 10% on analytes that exhibit a level 50 times greater than the MDL, then results for those analytes will be flagged with an "E".

4.12.2 Corrective Action:

4.12.2.1 No corrective action is necessary other than appropriately flagging the data.

5. Operation procedures

5.1 Instrumental Conditions

5.1.1 See instrument SOP for operating procedures, LTL-7106.

5.2 Calibration

5.2.1 Calibrate the instrument with a blank and one standard.

5.2.2 Immediately following the calibration, analyze the ICV followed by an ICB. Concentration values obtained for the ICV should not deviate from the actual values by more than 10% and less than 5% RSD for replicate integrations.

5.2.3 Analyze both the CRDL standard and the linear range standard. The CRDL recoveries should not exceed ±20% and the linear range standard should not deviate from the actual values by more than 10% and less than 5% RSD for replicate integrations.

5.2.4 Flush the system with the calibration blank solution for at least 1 minute before the analysis of each sample.

5.2.5 Analyze a CCV and CCB every 10 samples. The CCV must agree within 10% of its expected value and less than 5% RSD for replicate integrations. The results of the calibration blanks are to be below one half of the reporting limit or terminate the analysis, correct the problem, recalibrate, and reanalyze the previous 10 samples.

5.3 Analysis Sequence

5.3.1 See Appendix IV for Analytical Sequence

5.4 Analytical Operation

5.4.1 See instrument SOP for operating procedures, LTL-7106.

6. Reports

6.1 Data Packet Organization

6.1.1 See the metals validation SOP, LTL-7001, for a check list detailing data packet organization

6.1.2 If requested, all analysis performed under SW 846 guidelines can be reported via CLP SOW ILMO4.1 forms. These forms provide all relevant QC information.

6.1.3 Data packages will be produced via MARRS. Analyte levels that are less than the IDL or MDL will be reported as the SDL followed by a "U". Analyte levels that fall between the IDL or MDL and the reporting limit will be flagged with a "B". Analyte levels greater than or equal to the reporting limit PQL will be reported without a flag.

6.2 Flagging

CODE	Definition	
U	The analyte of interest was not detected, to the limit of detection indicated.	❁◆
B	The analyte of interest was detected between the MDL and the reporting limit.	❁
N	The spike recovery exceeded the control limits.	❁
*	The duplicates exceeded the RPD control limit or their difference exceeded the reporting limit.	❁
E	The Serial Dilution did not agree within 10%.	❁
S	The analyte concentration was determined by MSA.	❁

◆ Used in all reports.
❁ Used in data validatable packages.

6.3 Control Chart(s)

6.3.1 The recovery values for ICP analytes Al, B, Cd, Cr, and Ag in the LCS are plotted on control charts. Other analytes may be added at the discretion of QA without immediate revision of this SOP.

6.4 References:

Test Methods for Evaluating Solid Waste, SW-846, Method 6010B, Revision 2, December 1996

Appendix I

QC Summary Table

Laucks Testing Laboratories

Method 6010B QA Requirements and Corrective Actions

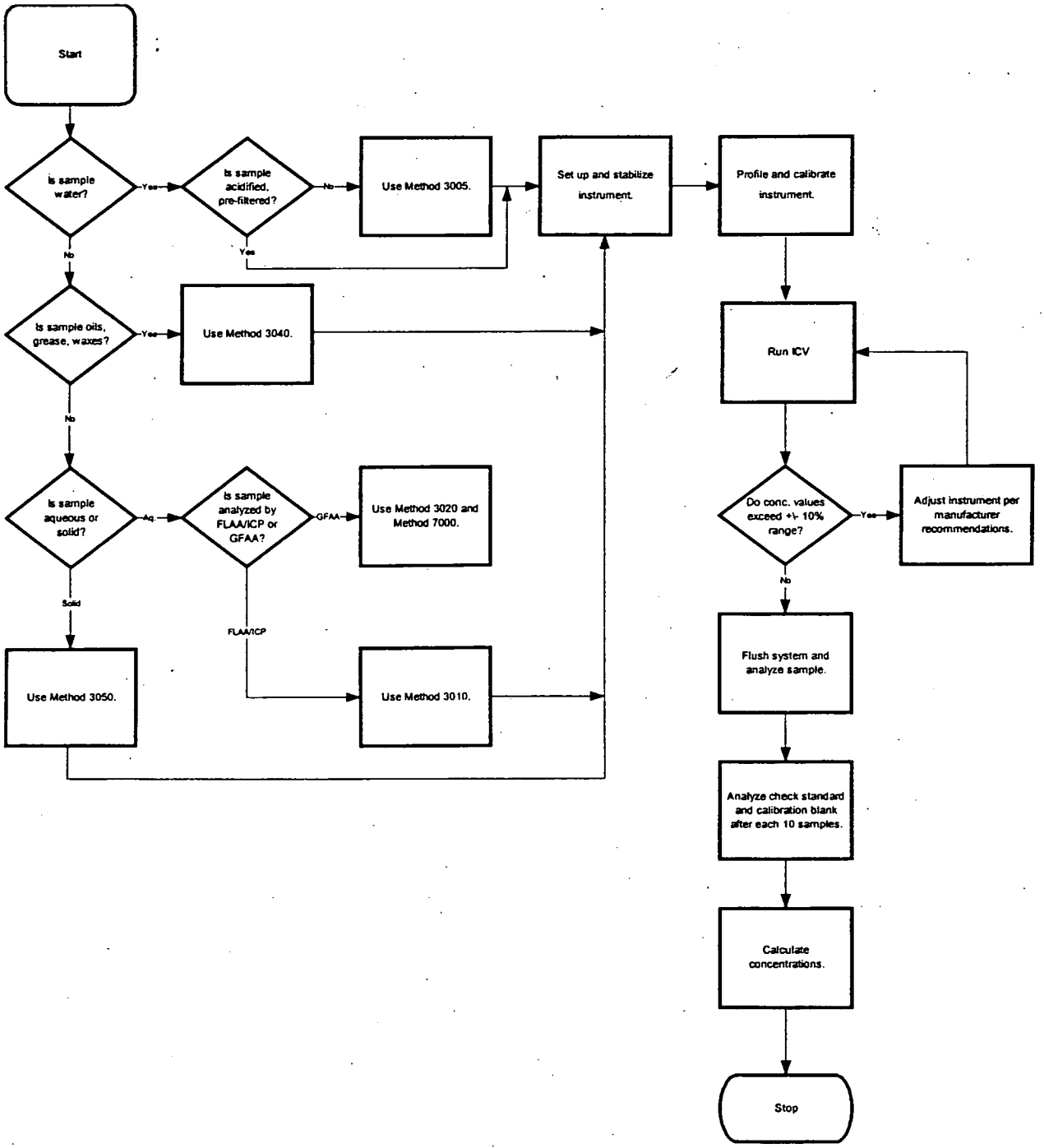
QA Element	Method Criterion	Laucks Criterion	Frequency	Corrective Action	Documentation
Initial Calibration	One standard and a Blank	One standard and a Blank	Beginning of each run.		In raw data.
Initial Calibration Verification	±10% of nominal value. Made from an independent source.	±10% of nominal value and RSD < 5% for replicate integrations. Made from an independent source.	Beginning of each run immediately following ICAL.	Recalibration required.	Form 2A or in raw data.
Continuing Calibration Verification	±10% and RSD < 5% for replicate integrations.	±10% and RSD < 5% for replicate integrations.	Every 10 samples. Mid-range	Recalibrate and rerun affected samples.	Form 2A or in raw data.
Instrument Blank	±3 sigma of average.	< 1/2 reporting limit	Every 10 samples.	Recalibrate and rerun affected samples.	Form 3 or in raw data.
Method Blank	<MDL or <5% of the concentration of any sample or <5% of the regulatory limit (whichever is highest).	< one-half the reporting limit or <5% of the regulatory limit or <5% of the sample conc. (whichever is highest).	One/batch	Flag data per QCO or redigest samples	Form 3, in raw data, or database report.
Laboratory Control Sample		LCSW: 80%-120% LCSS: Manufacturer Specs.	One/batch	Redigest samples	Form 7, in raw data, or database report
Matrix Spike Recovery	75-125% or within documented historical limits	75-125% or current QC database criteria.	5% or per batch	Consult QCO	Form 5, in raw data, or database report

Laucks Testing Laboratories

Method 6010B QA Requirements and Corrective Actions					
QA Element	**Method Criterion**	**Laucks Criterion**	**Frequency**	**Corrective Action**	**Documentation**
Post Digestion Spike Recovery	75-125% used for new or unusual sample matrices	75-125%. Spike level either 2x RL or 2x native sample level, whichever is greater	used when MS is out of control	none	Form 5B, in raw data, or database report
Duplicate % Difference	± 20% if sample conc. >10x the IDL	± 20% or current QC database criteria. ±RL if sample is <10x the IDL	5% or per batch	Consult QCO	Form 6, in raw data, or database report
Serial Dilution	± 10% difference used for new or unusual matrices	± 10% difference if sample conc. > 50x the MDL	One/batch	Flag data with an "E".	Form 9 or in the raw data.
ICSA,AB	±20% true value of analytes, or ± the CRDL.	±20% true value of analytes, or ± the CRDL.	Beginning and end of run	Recalibrate and rerun affected samples	Form 4, or in raw data.
Standard Reference Material (SRM) Recovery	See QC control catalog	Control limits set by vendor	5% or per batch	Redigest samples	Form 7, in raw data, or database report

Appendix II

Flow Chart


Start
Is sample water?
Yes
Is sample acidified, pre-filtered?
No
Use Method 3005.
Yes
Set up and stabilize instrument.
Profile and calibrate instrument.
No
Is sample oils, grease, waxes?
Yes
Use Method 3040.
Run ICV
No
Is sample aqueous or solid?
Aq
Is sample analyzed by FLAA/ICP or GFAA?
GFAA
Use Method 3020 and Method 7000.
Do conc. values exceed +/- 10% range?
Yes
Adjust instrument per manufacturer recommendations.
No
Solid
FLAA/ICP
Use Method 3050.
Use Method 3010.
Flush system and analyze sample.
Analyze check standard and calibration blank after each 10 samples.
Calculate concentrations.
Stop

Appendix III

Sample Handling and Preservation

Metals (except hexavalent chromium and mercury):

Measurement	Digestion Volume Required.	Collection Volume	Preservation/Holding Time
Total recoverable	100 mL	600 mL	HNO_3 to pH <2 6 months
Dissolved	100 mL	600 mL	Filter on site; HNO_3 to pH <2 6 months
Suspended	100 mL	600 mL	Filter on site 6 months
Total	100 mL	600 mL	HNO_3 to pH <2 6 months

Solid samples should be at least 200 g and usually require no preservation other than storing at 4°C until analyzed. Either plastic or glass containers may be used for sample collection.

Appendix IV

Analytical Sequence

Standard 0
Standard 1
Standard 2
ICV
ICB
CRDL Check
CCV
CCB
ICSAI
ICSABI
CCV
CCB
Sample #1
Sample #1D
Sample #1S
Sample #1L
Sample #1P
Sample #2
Sample #3
Sample #4
Sample #5
Sample #6
CCV
CCB

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-7202

Title: Metals Analysis Using Inductively Coupled Plasma - Mass Spectrometry (ICP/MS), SW 846 Method 6020

Revision history:

Number	Date
1.0	04/26/95
2.0	02/09/98
3.0	4/22/98
4.0	7/22/99

Written by: _____ Date: 7-22-99
Ming-Hwa Hwang, Metals Supervisor

Approved by: _____ Date: 7-22-99
Harry Romberg, QA Officer

Approved by: _____ Date: 7-22-99
Kathy Kreps, Laboratory Director

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Table of Contents

1. INTRODUCTION AND SCOPE 4

1.1 METHOD DESCRIPTION 4
1.2 SAMPLE COLLECTION, SAMPLE STORAGE, HOLDING TIMES 4
1.3 DEFINITION OF TERMS 4

2. EQUIPMENT LIST AND STANDARDS 7

2.1 INSTRUMENTATION: 7
2.2 STANDARDS 7
2.3 INTERNAL STANDARDS 7

3. SAFETY PRECAUTIONS AND WASTE DISPOSAL 7

3.1 SAFETY PRECAUTIONS 7
3.2 WASTE DISPOSAL 8

4. QUALITY CONTROL 8

4.1 METHOD DETECTION LIMIT STUDY 8
4.2 LINEAR RANGE STUDY 8
4.3 INTERNAL STANDARDS 8
4.4 INITIAL CALIBRATION 9
4.5 INITIAL CALIBRATION VERIFICATION 9
4.6 INITIAL CALIBRATION BLANK 10
4.7 CONTINUING CALIBRATION VERIFICATION (CCV) AND BLANK (CCB) 10
4.8 INTERFERENCE CHECK SOLUTIONS A (ICSA) AND AB (ICSAB) 10
4.9 METHOD BLANKS 11
4.10 LABORATORY CONTROL SAMPLE 12
4.11 MATRIX SPIKE 12
4.12 POST-DIGESTION SPIKE 13
4.13 MATRIX SPIKE DUPLICATE/SAMPLE DUPLICATE 13
4.14 SERIAL DILUTION 14

5. PROCEDURE 15

5.1 INSTRUMENTAL CONDITIONS 15
5.2 ANALYTICAL OPERATION 15

6. REPORTS 16

6.1 DATA PACKET ORGANIZATION 16
6.2 REFERENCES: 16

APPENDIX I 17

STANDARD SOLUTIONS 17

APPENDIX II 20

ICSA AND ICSAB SOLUTIONS 20

APPENDIX III 22

QC SUMMARY TABLE 22

Controlled Document
Assigned to: _____
No. _____

APPENDIX IV **25**

FLOW CHART 25

APPENDIX V **27**

ROUTINE REPORTING LIMITS 27

1. Introduction and Scope

1.1 Method Description

Inductively coupled plasma-mass spectrometry (ICP-MS) is a technique which is applicable to µg/L concentrations of a large number of elements in water and wastes after appropriate sample preparation steps are taken. When dissolved constituents are required, samples must be filtered and acid preserved prior to analysis. No further digestion is required prior to analysis for dissolved elements. Acid digestion prior to filtration and analysis is required for groundwater, aqueous samples, industrial waste, soil, sludge, sediment, and other solid waste for which total (acid-leachable) elements are required.

See Appendix V for reporting limits.
See Appendix I for analytical masses and standard concentrations.

This method is restricted to use by, or under the supervision of analysts experienced in the technique described. Each analyst performing this method must have demonstrated their ability to perform the described analysis.

1.2 Sample Collection, Sample Storage, Holding Times

Water samples should to be collected in plastic or Teflon containers and preserved to a pH < 2. A one liter sample bottle is sufficient volume for analysis. Soil samples do not require preservation but need to be stored at 4° C and may be collected in glass if plastic containers are not available. At least 200 grams of sample should be collected. The holding time for metals is 6 months. If mercury is being analyzed by this technique, which is not currently approved or done without specific client arrangement, the holding time is 28 days.

1.3 Definition of Terms

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

Batch Identifier - A number given to each analysis group which uniquely identifies that batch. This number is preceded by an "A", mmddyy, ICPMS, matrix (W for water, S for soil), sequence number (i.e. A022595ICPMSW01).

CCB - Continuing Calibration Blank - This is the same acronym used in the CLP program. This is a blank which is analyzed immediately after the CCV (almost always after every 10 samples and at the end of the analytical run) during the analysis sequence to determine whether the instrument or system has maintained a stable baseline.

CCV - Continuing calibration verification. - This is the same acronym used in the CLP program. This is a standard analyzed at some prescribed frequency (almost always after every 10 samples and at the end of the analytical run) during the analysis sequence to determine whether the instrument or system has remained in calibration.

CLP - Contract Laboratory Program - The USEPA program that contracts with laboratories to provide laboratory services. The term has come to mean a much broader set of methods and deliverables. In context of this SOP, CLP means procedures or operations which are detailed in the CLP contract and which are extended to a broader working definition.

DIW - Deionized water - Lab reagent water. This water should be free of virtually all analytes.

ICB - Initial calibration blank - This term is borrowed from CLP. An instrument blank is made up in the same matrix as calibration standards, without target analytes.

ICV - Initial calibration verification - This term is borrowed from the CLP protocol. It is a standard which is analyzed at the start of each analytical run that is compared to the initial multi-point calibration to determine whether the instrument calibration is accurate.

IDL - Instrument detection limit. IDL's can be estimated by analyzing seven replicates of a standard analyte solution over three nonconsecutive days. The analyte concentration should be 3-5 times the estimated IDL. Multiplying the average standard deviation by three will yield the IDL for that analyte. Each measurement must be performed as though it were a separate analytical sample. IDL's must be determined quarterly

MDL - Method detection limit - The lowest concentration of an analyte which will yield a positive result that is greater than zero at a known level of confidence. MDLs are empirically determined and are performed annually.

LCS - Laboratory Control Sample. This is a material of approximately the same matrix as the samples, containing a known and usually certified amount of target analyte and which is prepared and analyzed in the same manner as a typical sample. This

sample is used to demonstrate that the analytical system is in control. It may be considered to be a blank spike for most inorganic analyses and is preferred over artificially spiking blank materials.

Serial Dilution - If the analyte concentration is within the linear range of the instrument and is sufficiently high (minimally, a factor of 100 above the IDL/MDL), an analysis of a fivefold dilution must agree within ±10% of the original determination. If not, an interference effect must be suspected. One serial dilution must be analyzed for each twenty samples or less of each matrix in a batch. A serial dilution is denoted in the raw data by an "L".

ICSA - Interference Check Solution A. The ICSA is a solution that contains the interfering analytes. This solution is analyzed to indicate if a high level of interfering compounds will have an affect on the analytes of interest.

ICSAB - Interference Check Solution AB. The ICSAB is a solution that contains the interfering analytes and the analytes of interest. This solution is analyzed to indicate if a high level of interfering compounds will have an affect on the recovery of the analytes of interest.

Internal Standards - Internal standards are added to all blanks, standards, and samples. They monitor the affect of a sample's matrix on the quantification of the analytes of interest. The internal standards used are Sc45, In115, and Bi209.

Post-Digestion Spike - An analyte spike added to a portion of a prepared sample should be recovered to within 75% to 125% of the known value or within the laboratory derived acceptance criteria.

Standard-Addition - The standard addition technique involves adding known amounts of standard to an aliquot of the sample. This technique compensates for a sample constituent that enhances or depresses the analyte signal thus producing a different slope than that of the calibration standards.

QC Period - Quality control period - An analysis sequence initiated by the analysis of one or more standards, followed by samples, and terminated with a standard and blank analysis. A QC period can be open-ended chronologically, but calibration verification must be documented using the procedures in this SOP

RSD or %RSD - Relative standard deviation or percent relative standard deviation - The ratio of the standard deviation of a set of values to the mean of the set of values. A measure of the similarity of the values one to another.

2. Equipment List and Standards

2.1 Instrumentation:

Perkin-Elmer ELAN 5000

2.2 Standards

SW 846 requires the use of one standard and a blank. Standards are made in a 1% HNO_3. See Appendix I for standard concentrations.

2.3 Internal Standards

Sc45, In115 and Bi209 are used as internal standards.

3. Safety precautions and Waste Disposal

3.1 Safety Precautions

All standards, samples and sample solutions should be handled as if they are hazardous substances.

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

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

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

Radio Frequency - The ELAN 5000 uses a high energy RF. Although the generator is well shielded, care should be taken when operating the instrument. Pace makers can be adversely affected by exposure to high energy RF.

Plasma Radiation - Do not view the torch without the proper eye wear. Severe eye damage can occur if the plasma is viewed directly.

3.2 Waste Disposal

Waste segregation and disposal from the point of collection is further covered in the Laucks SOP on waste segregation and disposal.

4. Quality Control

4.1 Method Detection Limit Study

Prior to the analysis of any samples, it is necessary to establish method detection limits. This procedure is fully described in the Laucks SOP on performing MDL studies. Briefly, it involves the analysis of 7 replicate samples spiked at a concentration approximately 3 to 5 times the estimated method detection limit. A Student's T-test is then applied to these measured values to calculate the MDL.

4.2 Linear range study

Linear ranges for each analyte are determined by analyzing a high concentration "sample". The analytically determined concentration must be within 5% of the true value. The true value is the upper limit of the ICP/MS linear range. Linear ranges must be verified quarterly.

4.3 Internal Standards

A 50 uL aliquot of a 20 ppm of Sc45, 10 ppm of In115 and 10 ppm of Bi209 is added to a 10 mL of all standards and samples prior to analysis.

Criteria

The intensities of all internal standards for instrument check standards must be between 80 and 120 percent of the intensities of the internal standards in the initial calibration standard.

Corrective action

If the criteria are not met, terminate the analysis, correct the problem, recalibrate, verify the new calibration, and re-analyze the affected samples.

Criteria

The intensities of all internal standards in the samples must be between 30 and 120 percent of the intensities of the internal standards in the initial calibration standard.

Corrective action

When the intensity of any internal standard in the sample fails to fall between the required levels, the sample must be diluted fivefold (1+4) and re-analyzed with the addition of appropriate amounts of internal standards. This procedure must be repeated until all internal standards fall within the prescribed windows.

4.4 Initial Calibration

Analyze standard solutions using a minimum of a calibration blank and one standard. The calibration curve must be verified by running an Initial Calibration Standard (ICV) and obtaining agreement within 10% of the expected concentration.

Criteria and Corrective Action:

Since a linear regression is not possible when using a two point calibration on the ELAN 5000, the standard curve is validated by evaluating the ICV and the subsequent CCVs. If the corresponding control limits for the ICV and CCV are exceeded, then the sample analysis must be discontinued, the cause determined and the instrument recalibrated. All samples following the last acceptable ICV, CCV must be reanalyzed..

4.5 Initial Calibration Verification

Immediately after the calibration curve, analyze a standard from a source other than that from which the calibration material was obtained.

Criteria

The calculated concentration of the ICV should be within 90%-110% of the true value.

Corrective action

If the ICV criteria are not met, the analysis is terminated. Perform system maintenance and re-calibrate the instrument.

4.6 Initial Calibration Blank

After the analysis of the ICV standard an instrument blank (ICB) is analyzed. The levels of target analytes in the ICB should not exceed the contract required detection limit .

Corrective action

If the initial ICB contains target analyte levels above the contract required detection limit, the system is out of control. The source of contamination must be identified and corrected before proceeding with the analysis.

4.7 Continuing Calibration Verification (CCV) and Blank (CCB)

A continuing calibration verification standard is analyzed after every 10 samples. Immediately following the CCV, a blank solution is analyzed. In addition, this standard and blank must be the last samples analyzed in the run.

Criteria

The CCV must fall within ± 10% of the true value.

The levels of target analytes in the CCB should not exceed the contract required detection limit.

Corrective action

If CCV limits are exceeded, check calculations or perform instrument maintenance. Recalibrate and reanalyze. No sample results may be reported that are not bracketed by a successful calibration and a CCV which is in control or by preceding and following CCVs which are within limits.

If the initial CCB contains target analyte levels above the contract required detection limit, the system is out of control. The source of contamination must be identified and corrected and the affected samples re-analyzed. As with the CCVs, no sample results may be reported that are not bracketed by a successful initial and continuing calibration blank which are in control or by preceding and following CCBs which are within limits.

4.8 Interference Check Solutions A (ICSA) and AB (ICSAB)

Due to the high sensitivity of the ICP-MS technique and instrument developments that have occurred since the method was written, the high dissolved solids content of the specified ICS

solutions are not recommended by the manufacturer for modern instruments. The ICSA and ICSAB solutions are prepared at different concentration levels from method 6020 to avoid clogging of the sampler cone orifice and damage to the instrument. Therefore, Al, Ca, Fe, Mg, Na, P, K, S, C, and Cl in the ICSA and the ICSAB are at 1/10 of the specified levels. See Appendix II for ICSA and ICSAB solutions concentrations.

(ICSA):

At the beginning and at the end of each run, an interference check solution A is analyzed. This solution contains interfering elements only. All other elements are not present in the solution. All elements not present should show a recovery of zero, or ± the contract required detection limit.

Corrective Action:
If the analytes do not recover within the specified control limits, then the system is out of control. The problem needs to be identified and corrected prior to beginning another run.

(ICSAB):

At the beginning and end of each analytical sequence an ICSAB must be analyzed. Analytes must recover between 80-120%.

Corrective Action:

If the analytes do not recover within the specified control limits, then the system is out of control. The problem needs to be identified and corrected prior to beginning another analysis.

4.9 Method Blanks

Method blanks are used to verify contamination free reagents and apparatus. They are prepared with every set of samples prepared at the same time or at least one blank every 20 samples, which ever is more frequent. Any analyte response above the CRDL is reported. For a method blank to be acceptable for use with the accompanying samples, the concentration of the blank of any analyte of concern should not be higher than the highest of either:

(1) The reporting limit, or
(2) Five percent of the regulatory limit for that analyte, or
(3) Five percent of the measured concentration in the sample.

Corrective Action:

Corrective action may necessitate re-preparation and re-analysis of the sample set. For example if an analyte were found in the blank but not in any of the associated samples then sample group may not require re-analysis. In any case, if re-preparation and re-analysis is not being undertaken, the analyst must first discuss the issue with the Quality Control Officer. It is the laboratory's responsibility to ensure that method interference caused by contaminants in acids, solvents, reagents, glassware, and other sample processing hardware leading to discrete artifacts and/or elevated baselines in the analytical run be minimized. In the extreme case of chronic contamination, blanks may have to be analyzed from each stage of the sample processing to determine the contamination source so it can be eliminated. In all cases where blank contamination exceeds the control limit, a narrative comment must be made which documents the corrective actions taken.

4.10 Laboratory Control Sample

The LCS is made from an independent source of the same matrix (soil or water) and is carried through the entire digestion procedure. An LCS is performed with each digestion batch. At a minimum, LCSW(water) control limits are 80% to 120%.

LCSS(soil) control limits are supplied by the manufacturer. LCSS control limits are not derived by the laboratory due to the small number of data points available from each lot of certified material.

Corrective Action

If the LCS is not within the required control limits, a redigestion will occur for the affected analytes.

4.11 Matrix Spike

A sample is chosen at random from the samples to be analyzed, and an aliquot of spiking solution is added to this sample prior to preparation. The analyst should attempt to avoid selecting samples which are identified by the client as blanks. As the purpose of the matrix spike is to test the system under "typical" conditions, the analyst may also avoid selecting the most difficult sample of the batch for spiking. The minimum frequency for MS analysis is 1 each per 20 samples per matrix. This will be best accomplished by running one with every batch for many analyses. This matrix spike sample is used to evaluate the matrix effect of the sample upon recovery of the analytes. The recovery of spike analytes is calculated as follows:

$$recovery,\% = \frac{(SS - S)}{SA} * 100$$

where:

SS = concentration in spiked sample
S = native concentration in unspiked sample
SA = spike added, the amount of spiking material actually added calculated on the sample basis.

For ICP/MS, control limits for spike recoveries will be 75-125% unless the sample result is greater than 4 times the spike concentration or unless otherwise specified in the project specific QAPP. In-house control limits are based on historical performance.
The recovery criteria are detailed in the QC Database QC_DB and will change from time to time.

Corrective Action:

Samples with spike recoveries outside control limits will be reviewed for possible corrective action. Corrective action will first involve recalculation, followed by possible re-preparation, and/or reanalysis. This process should also look at the recovery of matrix spiking compounds from the SRM and/or blank spike analysis. In all cases a narrative explanation of the condition is required to detail the corrective actions taken. Data reported in validatable packages will be flagged with an "N" indicating the out-of-control event.

4.12 Post-Digestion Spike

A post digestion spike is also performed to a portion of a prepared sample. The minimum frequency for MS analysis is 1 each per 20 samples per matrix, control limits for post-spike recoveries will be 75-125%

Corrective Action:

Samples with post-spike recoveries outside control limits will be diluted and re-analyzed to compensate for matrix effects. The results must agree to within 10% of the original measured concentrations. A standard-addition technique may also be used to compensate for matrix effects.

4.13 Matrix Spike Duplicate/Sample Duplicate

Method QC consists of MS/MSD. A duplicate maybe be performed instead of a MSD. Other types of QC can performed at the client's request.

Criteria

At least one matrix spike duplicate sample per 20 samples per matrix is required when matrix spikes are being performed. RPD values are calculated in a manner similar to MS/MSD RPDs:

$$RPD = \frac{|SS - SSD|}{(SS + SSD)/2} * 100$$

where:
SS = concentration in spiked sample
SSD = concentration in matrix spiked duplicate sample

For sample concentrations greater than 5 times the CRDL, control limits for RPD of duplicates will be ±20% unless otherwise specified in the project specific QAPP. For sample concentrations less than 5 times the CRDL, control limits for the difference will be ± the CRDL. In-house control limits are based on historical performance. The RPD control limits are detailed in the current QC Database QC_DB and will change from time to time.

Corrective Action:

If a trend in out of control RPD values is observed, the methods used must be examined to determine the source of variance. Once this source is identified, the method must be changed so that samples can be analyzed with a predictable reproducibility. Generally, if recoveries are in control and no analyte of interest was detected in any of the samples, no immediate action will be taken on that sample set. If integrity of reported sample values is in doubt, re-analysis may be called for. Corrective actions should be discussed with the Quality Control Officer. In a validatable package, data associated with an out-of-control RPD will be flagged with an "*".

4.14 Serial Dilution

A five-fold dilution is performed on the QC sample in each analytical batch. The difference between the initial value and the serial dilution should agree within 10%. If the difference is greater than 10% on analytes that exhibit a level 50 times greater than the IDL, then results for those analytes will be flagged with an "E".

Corrective Action:

No corrective is necessary other than appropriately flagging the data.

5. Procedure

5.1 Instrumental Conditions

- Refer to manufacture's instruction for specific operating procedures. Allow at least 30 minutes for the instrument to stabilize before initiating any analysis.

- Conduct mass calibration and resolution checks in the mass regions of interest. The mass calibration and resolution parameters are required criteria which must be met prior to any sample being analyzed. The monitored masses of Mg, Rh, and Pb must meet the following criteria:

Element	RSD for replicate (minimum of four) integrations.	Mass, amu	Resolution @ 10 % peak height, amu
Mg	< 5%	23.90-24.10	<0.9
Rh	< 5%	102.80-103.00	<0.9
Pb	< 5%	207.90-208.10	<0.9

5.2 Analytical Operation

- Calibrate the instrument, using a calibration blank and a standard. Refer to Appendix I for the applied levels of concentration.

- All masses which could affect data quality are monitored to determine potential effects from matrix components on the analytes of interest.

- After the calibration has been established, an ICV solution is analyzed to verify the validity of the curve. Measurements for the analytes of interest must be at ±10% of the true value. A re-calibration and re-analysis is required for any analyte which falls outside the control limit.

- Analyze the interference check samples (ICSA and ICSAB) prior to and after the analysis of samples

- Analyze a CCV and a CCB once every 10 analytical samples.

- Dilute samples that exceed the established linear range of the instrument.

6. Reports

6.1 Data Packet Organization

- See the SOP metals validation for a check list detailing data packet organization
- If requested, all analysis performed under SW 846 guidelines the data can be reported via CLP SOW 3/90 forms.
- Data packages will be produced via Enviroforms. Analyte levels that are less than the MDL will be reported as the SDL followed by a "U". Analyte levels that fall between the MDL and the reporting limit will be flagged with a "B". Analyte levels greater than or equal to the reporting limit PQL will be reported without a flag.

CODE	Definition	
U	The analyte of interest was not detected, to the limit of detection indicated.	❁◆
B	The analyte of interest was detected between the MDL and the reporting limit.	❁
N	The spike recovery exceeded the control limits.	❁
*	The duplicates exceeded the RPD control limit or their difference exceeded the reporting limit.	❁
E	The Serial Dilution did not agree within 10%.	❁
S	The analyte concentration was determined by MSA.	❁

◆ Used in all reports.
❁ Used in data validatable packages.

6.2 References:

Test Methods for Evaluating Solid Waste, SW-846, Method 6020, Revision 0, September 1994

Appendix I

Standard Solutions

Elements	Mass	STD, µg/L
Be	9	50
Na	23	500
Mg	26	500
Al	27	400
K	39	500
Ca	44	500
V	51	100
Cr	52	100
Mn	55	150
Fe	54,57	200
Co	59	100
Ni	60	400
Cu	63,65	50
Zn	66	200
As	75	100
Se	82	200
Ag	107	100
Cd	111	50
Sb	121	60
Ba	137	400
Tl	205	100
Pb	208	50
Li	7	200
B	11	200
Mo	98	200
Sn	118,120	200
U	238	250

The STD is made by diluting 10.0 mL of the ICP/MS stock standard to a 100 mL final volume.

The ICP/MS stock standard which consists of:

100 µL	ICAL-1
1000 µL	ICAL-2
200 µL	ICAL-3
1000 µL	ICAL-4
100 µL	ICAL-5
250 µL	1000 ppm U
200 µL	1000 ppm B
200 µL	1000 ppm Mo
200 µL	1000 ppm Li
200 µL	1000 ppm Sn
40 µL	1000 ppm Be
150 µL	1000 ppm Se

Note: 50 µL of 20 ppm Sc45, 10 ppm In115 and 10 ppm Bi209 is added to a 10 mL aliquot of standard.

Appendix II

ICSA and ICSAB Solutions

Element	ICSA (PPM)	ICSAB (PPM)
Aluminum	4	4
Arsenic		0.020
Cadmium		0.020
Calcium	12	12
Chromium		0.040
Cobalt		0.040
Copper		0.040
Iron	10	10
Magnesium	4	4
Manganese		0.040
Molybdenum		0.16
Nickel		0.040
Potassium	4	4
Selenium		0.020
Silver		0.040
Sodium	10	10
Vanadium		0.040
Zinc		0.020

Appendix III

QC Summary Table

Laucks Testing Laboratories

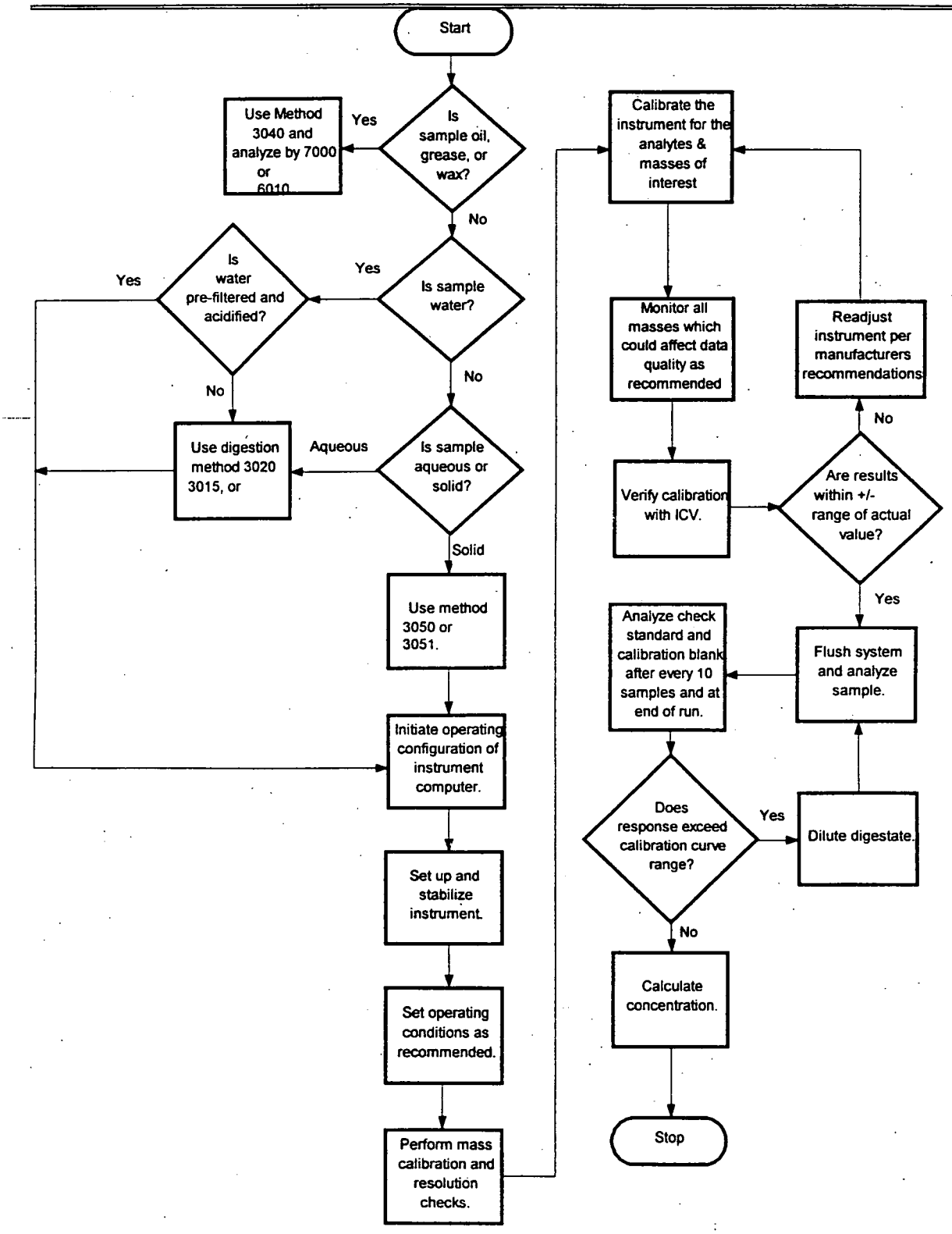
Method SW 846 6020 QA Requirements and Corrective Actions

QA Element	Method Criterion	Laucks Criterion	Frequency	Corrective Action	Documentation
Mass Calibration Check	must be performed in mass regions of interest and be within ±0.1 amu of the actual value.	Mg 23.90-24.10 Rh 102.80-103.00 Pb 207.90-208.10	Beginning of each analysis.	Perform new mass calibration.	Instrument Logbook
Resolution Check	< 0.9 amu full width at 10% peak height	< 0.9 amu full width at 10% peak height	Beginning of each analysis.	Adjust resolution.	Instrument Logbook
Initial Calibration	Blank and at least one standard	Blank and one standard	Beginning of each analysis	NA	In the raw data and/or on FORM 14.
Initial Calibration Verification	±10% of true value. Made from an independent source.	±10% of true value. Made from an independent source..	Immediately following calibration.	Recalibrate and reverify.	Form 2, in the raw data
Initial Calibration Blank	Values must be < 3x the IDL for each element.	Values must be < CRDL	Immediately following ICV.	Recalibrate, reverify, and rerun the ICB.	Form3, in the raw data
Continuing Calibration Verification	±10% true value.	±10% true value. Analyte levels are at the mid-range of the calibration.	Every 10 samples and end of run.	Recalibrate and rerun affected samples.	Form 2, in the raw data
Continuing Calibration Blank	Values must be < 3 times the IDL for each element.	Values must be < CRDL	Immediately following CCV.	Recalibrate and rerun affected samples.	Form 3, in the raw data
Method Blank	< CRDL or <5% of regulatory limit or any sample	< CRDL or <5% of regulatory limit or any sample	One/batch	Redigest samples	Form 3, in the raw data.

QA Element	Method Criterion	Laucks Criterion	Frequency	Corrective Action	Documentation
Internal Standards	1.Samples: 30 % - 120 % of the initial calibration standard 2. Instrument Check Standards: 80 -120 of the initial calibration standard.	1. Samples 30 % - 120 % of the initial calibration blank. 2. Instrument Check Standards: 80 -120 of the initial calibration blank.	1. All samples 2. All instrument check standards	1. dilute and reanalyze. 2. recalibrate, re-analyze the affected samples.	In the raw data.
Serial Dilution	within ±10% of the original value if the analyte conc. is > 100 x the IDL.	within ±10% of the original value if the analyte conc. is > 100 x the IDL.	One/batch	Flag data with an "E".	Form 9,.in the raw data
Duplicate, % Difference	± 20% for analyte values greater than 100 times the IDL.	± 20% or current QC database criteria.	5% or per batch	reanalyze digestates, if still fail, consult QCO	Form 6, in raw data, or database report
Matrix Spike Recovery		75-125% or current QC database criteria.	5% or per batch	Consult QCO	Form 5A, in raw data, or database report
Post-Digestion Spike	75%-125%	75%-125%	5% or per batch	dilute and re-analyze or MSA	Form 5B, in the raw data
Laboratory Control Sample	One/batch, no acceptance criteria	LCSW: 80%-120% LCSS: Manufacturer Specs.	One/batch	Redigest samples.	Form 7, in raw data, or database report
ICSA and ICSAB	±20% true value of analytes, or ± the CRDL.	±20% true value of analytes, or ± the CRDL.	Beginning and end of run	reanalyze affected samples	Form 4, or in raw data.

Appendix IV

Flow Chart


Start
Is sample oil, grease, or wax?
Yes
Use Method 3040 and analyze by 7000 or 6010
No
Is sample water?
Yes
Is water pre-filtered and acidified?
Yes
No
Use digestion method 3020 3015, or
No
Is sample aqueous or solid?
Aqueous
Solid
Use method 3050 or 3051.
Initiate operating configuration of instrument computer.
Set up and stabilize instrument.
Set operating conditions as recommended.
Perform mass calibration and resolution checks.
Calibrate the instrument for the analytes & masses of interest
Monitor all masses which could affect data quality as recommended
Verify calibration with ICV.
Are results within +/- range of actual value?
No
Readjust instrument per manufacturers recommendations
Yes
Flush system and analyze sample.
Analyze check standard and calibration blank after every 10 samples and at end of run.
Does response exceed calibration curve range?
Yes
Dilute digestate.
No
Calculate concentration.
Stop

Appendix V

Routine Reporting Limits

SW 846 6020 Reporting Limits

Element	Reporting Limit, µg/L	Element	Reporting Limit, µg/L
Ag	3.0	Mn	1.0
As	1.0	Mo	10.0
B	10.0	Ni	10.0
Ba	2.0	Pb	1.0
Be	1.0	Sb	1.0
Cd	1.0	Se	3.0
Co	3.0	Sn	10.0
Cr	5.0	U	100
Cu	2.0	V	2.0
Li	10.0	Zn	10.0

Reporting limits are approximately 2-10 times the instrumental MDL. The MDL is based on samples prepared using SW 846 3015. Values actually reported may be less than the routine reporting limits but above our method detection limit.

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-7401

Title: **The Colorimetric Determination of Hexavalent Chromium by SW-846 7196A**

Revision history:

Number	Date
2	04/04/01
1	03/06/98
0	02/01/96

Controlled Document
No. 20 **Assigned to:** Tetra

Written by: Tracy Schaff, Inorganics Analyst — Date: 4/4/01

Reviewed by: Joan Phillips, Inorganics Supervisor — Date: 4/5/01

Reviewed by: Harry Romberg, QC Officer — Date: 4-5-01

Approved by: Kathy Kreps, Laboratory Director — Date: 4-5-01

Table of Contents

1. **INTRODUCTION AND SCOPE** 3
1.1. METHOD DESCRIPTION 3
1.2. SAMPLE COLLECTION, SAMPLE STORAGE, HOLDING TIMES 3
1.3. INTERFERENCES 3
2. **EQUIPMENT LIST AND STANDARDS** 6
2.1. INSTRUMENT 6
2.2. STANDARDS AND REAGENTS 6
3. **SAFETY PRECAUTIONS AND WASTE DISPOSAL** 6
3.1. SAFETY PRECAUTIONS 6
3.2. WASTE DISPOSAL 7
4. **CALIBRATION AND QUALITY CONTROL** 7
4.1. METHOD DETECTION LIMIT STUDY 7
4.2. INITIAL MULTI-POINT CALIBRATION 7
4.3. INITIAL CALIBRATION VERIFICATION 8
4.4. INITIAL CALIBRATION BLANK 8
4.5. METHOD DETECTION LIMIT STANDARD 9
4.6. CONTINUING CALIBRATION VERIFICATION (CCV) AND BLANK (CCB) 9
4.7. METHOD BLANKS 9
4.8. METHOD BLANK SPIKES 10
4.9. MATRIX SPIKE 11
4.10. MATRIX SPIKE DUPLICATE 12
4.11. SAMPLE DUPLICATE 12
5. **OPERATION PROCEDURES** 13
5.1. SAMPLE ANALYSIS SEQUENCE 13
5.2 INSTRUMENTAL CONDITIONS 14
5.3 ANALYTICAL OPERATION 14
6. **REPORTS** 15
6.1. DATA PACKET ORGANIZATION 15
6.2. QUALITY CONTROL REPORTS 15
6.3. SAMPLE RESULT REPORTS 15
7 **REFERENCES** 15
APPENDIX I 16
QC SUMMARY TABLE 17

1. Introduction and Scope

1.1. Method Description

1.1.1. Method 7196 is used to determine the concentration of dissolved hexavalent chromium in TCLP extracts, ground waters, domestic and industrial wastes. Highly turbid or colored samples will present the possibility of positive interference, and will need to be corrected spectrophotometrically. This method is used to measure Cr^{+6} in samples from 5-100 µg/L at a wavelength of 540 nm. Higher concentrations will require either dilution or the use of a shorter cell path.

1.1.2. This method is restricted to use by, or under the supervision of analysts experienced in the technique described. Each analyst performing this method must have demonstrated the ability to perform the described analysis.

1.2. Sample Collection, Sample Storage, Holding Times

1.2.1. Water samples should be collected in a 500 ml unpreserved bottle. Glass and plastic are acceptable. Soil samples should be collected in â 4 or 8 oz glass soil container. Samples should be stored at 4°C ± 2°C until extraction or analysis. Water samples should be analyzed within 24 hrs of collection. Soil samples may be extracted using a number of different methods. The holding time will be dependent upon the method chosen.

1.3. Interferences

1.3.1 The reaction with diphenylcarbazide is nearly specific for chromium. Hexavalent molybedium and mercury salts will react to form color with reagent but intensities are much lower than for chromium at the specific pH. Concentrations as high as 200mg Mo or Hg/L can be tolerated. Vanadium interferes strongly, but concentrations up to 10 times that of chromium will not cause trouble. Potentail interferences from permanganate is eliminated by prior reduction with azide. Iron in concentrations greater than 1 mg/L may produce a yellow color but the ferric ion (Fe^{3+}) color is not strong and no difficulty is encountered normally if the absorbance is measured photometrically at the appropriate wavelength. Reducing conditions will cause Hexavalent Chromium to be present in lower quantities and will result in significantly reduced spike recoveries.

1.4 Definition of Terms

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

1.4.2. **Batch Identifier** - A number given to each preparation or analysis group which uniquely identifies that batch. This number is an analysis number which is similar to the blank ID, only preceded by an "A" rather than a "B" for inorganic batches. The preparation batch IDs are discussed in other documentation. The second analysis for a water sample performed on Jan 2, 1996 would have the identifier A010296CR6W02.

1.4.3. **Blank spike** - A background free matrix (DIW for water, clean sand for soils/sediments) to which known amounts of target analytes are added each time samples are prepared. Blank spikes are required on all NFESC work. Note that an LCS or SRM (see below) will substitute as a blank spike for most inorganic analyses. In the context of this SOP, a blank spike is the same as a QC check standard. See also QC check standard.

1.4.4. **CCB** - Continuing Calibration Blank - This is the same acronym used in the CLP program. This is a blank which is analyzed immediately after the CCV (almost always after every 10 samples and at the end of the analytical run) during the analysis sequence to determine whether the instrument or system has maintained a stable baseline.

1.4.5. **CCV** - Continuing Calibration Verification - This is the same acronym used in the CLP program. This is a standard analyzed at some prescribed frequency (almost always after every 10 samples and at the end of the analytical run) during the analysis sequence to determine whether the instrument or system has remained in calibration.

1.4.6. **CLP** - Contract Laboratory Program - The USEPA program that contracts with laboratories to provide laboratory services. The term has come to mean a much broader set of methods and deliverables. In context of this SOP, CLP means procedures or operations which are detailed in the CLP contract and which are extended to a broader working definition.

1.4.7. **Corr Coef, CC** - Correlation coefficient - A measure of the "goodness of fit" of a set of data to a regression model. The closer the value is to 1, the higher the degree of confidence in the correlation

1.4.8. **DIW** - Deionized Water - Lab reagent water. This water should be free of virtually all analytes.

1.4.9. Initial Calibration Blank - This term is borrowed from CLP. An instrument blank is made up in the same way as calibration standards, without target analytes.

1.4.10. **ICV** - Initial Calibration Verification - This term is borrowed from the CLP protocol. It is a standard which is analyzed at the start of each analytical run that is compared to the initial multi-point calibration to determine whether the instrument calibration is accurate. For most inorganic methods, this verification standard is from a source different from that used to make the calibration standards.

1.4.11. **IDL** - Instrument Detection Limit. The lowest concentration of a target analyte that will yield a signal:noise ratio of least 3x. Used as a starting point for selecting MDL study spiking levels.

1.4.12. **MDL** - Method Detection Limit - The lowest concentration a sample which will yield a positive result that is greater zero at a known level of confidence. MDLs are empirically determined by Laucks.

1.4.13. **MDL standard** - Method detection limit standard - A standard prepared so that the concentrations of the target analytes are no greater than 4x the empirically determined MDLs. This standard is used to verify that the instrument or system is capable of detecting the target analytes on an ongoing basis.

1.4.14. **QC check standard** - Quality control check standard. Referred to in this SOP as a blank spike. A QC check standard is used to determine whether the analytical system is in control if MS/MSD recoveries are out of control. See also blank spike.

1.4.15. **SRM or LCS** - Standard Reference Material or Laboratory Control Sample. This is a material of approximately the same matrix as the samples, containing a known and usually certified amount of target analyte and which is prepared and analyzed in the same manner as a typical sample. This sample is used to demonstrate that the analytical system is in control. It may be considered to be a blank spike for most inorganic analyses and is preferred over artificially spiking blank materials.

1.4.16. **RSD or %RSD** - Relative Standard Deviation or percent relative standard deviation - The ratio of the standard deviation of a set of values to the mean of the set of values. A measure of the similarity of the values one to another.

2. Equipment List and Standards

2.1. Instrument

2.1.1. This analysis uses a Varian Cary 50 Bio spectrophotometer or equivalent instrumentation. Instrumental conditions are 540 nm, slit 1, integration 2, and a 1 cm path length cell.

2.2. Standards and Reagents (All should be stored in the refrigerator at 4°C +/- 2°C)

2.2.1. Potassium dichromate standard solution 1, 100 µg/ml
Dissolve 0.2829 g of Primary Standard Reagent $K_2Cr_2O_7$ in DIW and dilute to 1000 ml.

This solution should be prepared annually.

2.2.2. Potassium dichromate standard solution 2, 0.10 µg/ml
Dilute 250 µl of Standard 1 to 250 mls with DIW.

This solution should be prepared fresh daily.

2.2.3. Diphenylcarbazide Solution: Dissolve 250 mg of 1,5-diphenylcarbazide in 50 mls of Reagent grade Acetone. Store this solution in an amber bottle. Discard when it becomes discolored.

2.2.4. Sulfuric Acid Solution, 10% (v/v): Dilute 10 mls of metals grade H_2SO_4 to 100 mls with DIW

3. Safety precautions and Waste Disposal

3.1. Safety Precautions

3.1.1. All standards, samples and sample solutions should be handled as if they are hazardous substances.

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

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

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

3.1.5. Hexavalent chromium is highly toxic. Care should be taken to avoid ingestion.

3.2. Waste Disposal

3.2.1. All highly concentrated Chromium waste from this analysis should disposed of in with the COD waste, currently in a container under the sink where Chromium analysis is performed. Low concentration samples can be disposed down the sink with water.

3.2.2. Waste segregation and disposal from the point of collection is further covered in the Laucks SOP on Waste Segregation and Disposal.

4. Calibration and Quality Control

4.1. Method Detection Limit Study

4.1.1. Prior to the analysis of any samples, it is necessary to establish method detection limits. This procedure is fully described in the Laucks on MDL studies. Briefly, it involves the analysis of 7 replicate samples spiked at a concentration near the anticipated method detection limit. A Student's T-test is then applied to these measured values to calculate the MDL. Currently, the MDL for water analysis is 1.0 ug/ L, while soil analysis is 1.0 mg/kg.

4.2. Initial Multi-Point Calibration

4.2.1. A calibration curve is prepared by measuring 5, and 25 mls of solution 2 and 50, 250, and 500 µls of solution 1 into 50 ml nessler tubes. The concentration of Cr^{+6} in the flasks is 0.01, 0.05, 0.10, 0.50, and 1.0 mg/L, respectively. 5 µg/L is the reporting limit. Any samples above 1.0 mg/L should be diluted and reanalyzed.

The instrument is calibrated from the lowest to the highest concentration.

Due to the inherent instability of Cr^{+6}, solution 2 should be prepared fresh daily.

4.2.2. Criteria

4.2.3. Initial calibration data is evaluated using the correlation coefficient of a linear regression analysis. The correlation coefficient must be 0.995 or greater for a 5-point calibration. All CCVs and sample extract concentrations must be computed using the regression equation.

4.2.4. Corrective action

4.2.5. If the criteria are not met, the instrument must be recalibrated.

4.3. Initial Calibration Verification

4.3.1. Immediately after the calibration curve, analyze a standard from a source other than that from which the calibration material was obtained.

4.3.2. Criteria

4.3.3. The calculated concentration of the ICV must be within the limits supplied by the manufacturer. If limits for the ICV have not been supplied by the vendor, default limits of 90-110% will be used. Cr^{+6} is an inherently unstable analyte. Thus, the ICV solutions and their corresponding limits will change frequently.

4.3.4. Corrective action

4.3.5. If the ICV criteria are not met, no samples can be analyzed. Perform system maintenance and re-check the ICV. If the criteria still cannot be met, the system must be recalibrated.

4.4. Initial Calibration Blank

4.4.1. After the analysis of the ICV standard an instrument blank (ICB) is analyzed. The levels of target analytes in the ICB should not exceed ½ the reporting limit.

4.4.2. Corrective action

4.4.3. If the initial ICB contains target analyte levels above the reporting limit, the system is out of control. The source of contamination must be identified and corrected before proceeding with the analysis.

4.5. Method Detection Limit Standard

4.5.1. After the calibration is performed, but before the analysis of any sample extracts, an MDL standard is to be analyzed. The MDL standard is used to provide on-going verification of the ability of the system to detect analytes at a concentration near the method detection limit. This standard is usually at the reporting limit. It must be detected for the system to be considered in control.

4.5.2. Corrective Action

4.5.3. If target analytes are not detected, the analysis must be terminated until the problem has been solved. Alternatively, if the affected samples are well above the detection limit (ie bracketed by appropriate standards), they may be reported. No undetected values should be reported if the MDL standard for that analyte(s) is undetected.

4.6. Continuing Calibration Verification (CCV) and Blank (CCB)

4.6.1. A mid-range calibration standard is analyzed after every 10 samples. Immediately following the CCV, a blank solution is analyzed. In addition, this standard and blank must be the last samples analyzed in the run.

4.6.2. The CCV must fall within ± 10% of the true value.

4.6.3. The levels of target analytes in the CCB should not exceed ½ the reporting limit.

4.6.4. Corrective action

4.6.5. If CCV limits are exceeded, check calculations or perform instrument maintenance. Recalibrate and reanalyze. No sample results may be reported that are not bracketed by a successful calibration and a CCV which is in control or by preceding and following CCVs which are within limits.

4.6.6. If the initial CCB contains target analyte levels above the reporting limit, the system is out of control. The source of contamination must be identified and corrected and the affected samples re-analyzed. As with the CCVs, no sample results may be reported that are not bracketed by a successful initial and continuing calibration blank which are in control or by preceding and following CCBs which are within limits.

4.7. Method Blanks

4.7.1. Method blanks are used to verify contamination free reagents and apparatus. They are prepared with every set of samples prepared at the same time or at least one blank for every 20 samples, whichever is more frequent. Any analyte response above the reporting limit is reported. Method blank control limits are that contamination should not exceed ½ the reporting limit.

4.7.2. Corrective action

4.7.3. Corrective action may necessitate re-preparation and re-analysis of the sample set. For example if an analyte were found in the blank but not in any of the associated samples then sample group may not require re-analysis. In addition, if sample levels exceed 20 times the blank, the level of contamination may be considered insignificant. In any case, if re-preparation and re-analysis is not being undertaken, the analyst must first discuss the issue with the Quality Control Officer. It is the laboratory's responsibility to ensure that method interferences caused by contaminants in acids, solvents, reagents, glassware, and other sample processing hardware leading to discrete artifacts and/or elevated baselines in the analytical run be minimized. In the extreme case of chronic contamination, blanks may have to be analyzed from each stage of the sample processing to determine the contamination source so it can be eliminated. In all cases where blank contamination exceeds the control limit, a narrative comment must be made which documents the corrective actions taken.

4.8. Method Blank Spikes

4.8.1. A method blank spike follows the same protocol as with the matrix spike analysis except that the spiking solution is added to a method blank solution instead of an actual sample. A method blank with added analytes is a method blank spike. A method blank spike is the same as a QC check standard. A blank spike or a standard reference material (SRM) should be analyzed . The SRM is the preferred material. The blank spike should only be analyzed when an SRM is not available. We currently use a material from APG.

4.8.2. Corrective action

4.8.3. If the MS/MSD recoveries are out of control, the blank spike recoveries are examined to assess whether the method was in control during sample preparation and analysis. Re-prepare and reanalyze any samples for which both the matrix spike recoveries are low and out of control and for which the associated blank spike demonstrates out of control and low recoveries.

4.9. Matrix Spike

4.9.1. The method requires a spike be run on every sample matrix to verify that neither a reducing condition nor a chemical interference is affecting color development. The amount of spike added should double the concentration found in the sample, and should be at least 30 ug/L A sample is chosen at random from the samples to be analyzed, and an aliquot of spiking solution is added to this sample prior to preparation. The analyst should attempt to avoid selecting samples which are identified by the client as blanks. As the purpose of the matrix spike is to test the system under "typical" conditions, the analyst may also avoid selecting the most difficult sample of the batch for spiking. The minimum frequency for MS analysis is 1 each per 20 samples per matrix. This will be best accomplished by running one with every batch for many analyses. This matrix spike sample is used to evaluate the matrix effect of the sample upon recovery of the analytes. The recovery of spike analytes is calculated as follows:

$$\% \text{ recovery} = \frac{(SS - S) * 100}{SA}$$

where:
SS = concentration in spiked sample
S = native concentration in unspiked sample
SA = spiked added, the amount of spiking material actually added to the sample calculated on the sample basis

4.9.2. The recovery criteria are detailed in the Quality Control Database and will change from time to time. Currently, for water analysis the allowed recoveries are 50-148%, while that of soil analysis is 50-150%.

4.9.3. Corrective action

4.9.4. Samples with spike recoveries outside control limits will be reviewed for possible corrective action. Corrective action will first involve recalculation, followed by possible re-preparation, and/or reanalysis of a diluted aliquot of the sample. This process should also look at the recovery of at the recovery of matrix spiking compounds from the SRM and/or blank spike analysis. In all cases a narrative explanation of the condition is required to detail the corrective actions taken.

4.10. Matrix Spike Duplicate

4.10.1. The compound recovery criteria are identical to those for the matrix spike sample. In addition, the matrix spike duplicate is used measure method precision. This is done by computing the relative percent difference (RPD) between the matrix spike and matrix spike duplicate recovery values. The control limits for the RPD are detailed in the quality control database and are currently 30%. The calculation is as follows:

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

where:
S1 = measured concentration for MS sample
S2 = measured concentration for MSD sample

4.11. Sample Duplicate

4.11.1. Criteria

4.11.2. Sample duplicates are required only when the client requests, when CLP practices are employed, or when the method specifically calls for duplicates. At least one duplicate sample per 20 samples per matrix is required when matrix spikes are being performed. RPD values are calculated in a manner similar to MS/MSD RPDs:

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

where:
S1 = measured concentration in the initial analysis
S2 = measured concentration in the duplicate analysis

4.11.3. The RPD control limits are detailed in the Quality Control Database and will change from time to time. Currently, the RPD allowed for both soil and water analysis is 30.

4.11.4 Corrective action

4.11.4. If a trend in out of control RPD values is observed, the methods used must be examined to determine the source of variance. Once this source is identified, the method must be changed so that samples can be analyzed with a predictable reproducibility. Generally, if recoveries are in control and no analyte of interest was detected in any of the samples, no immediate action will be taken on that sample set. If integrity of reported sample values is in doubt, re-analysis may be called for. Corrective actions should be discussed with the Quality Control Officer.

5. Operation procedures

5.1. Sample Analysis Sequence

S0.01
S0.05
S0.10
S0.50
S1.0
ICV
ICB
MDL
PBW
Sample 1
Sample 1D (or S, depending upon client needs)
Sample 1S (or MSD, depending upon client needs)
Sample 2
Sample 3
Etc
after 10 samples
CCV 1
CCB 1
Sample 11
Etc
CCV 2
CCB 2

5.2 Instrumental Conditions

5.2.1 The samples are measured with a Varian Cary 50 Bio UV-VIS spectrophotometer.

The slit is set at 1 nm
The integration is set for 1 sec
The wavelength is 540 nm.
Use 1 cm cells.
The zero standard is used to auto-zero the instrument.

5.3 Analytical Operation

5.3.1 If Cr^{+6} in soil is requested, the sample may be extracted using the SW-846 3060A method. These are detailed in the applicable Laucks SOPs.

5.3.2. Starting with a water sample or aqueous extract:

1. Transfer 50 mls to a 50 ml nessler tube.
2. Add 1.0 ml of the diphenylcarbazide solution.
3. Add 1.0 ml of 10% H_2SO_4 and mix.
4. Let the solution stand for 40+ mins for maximum color development
5. Measure the absorbance at 540 nm using the zero standard as a reference.

5.3.3. If the sample appears to be turbid, it will have to be corrected for. This is done by preparing a second aliquot. This is treated like the sample, except that the diphenylcarbazide is not added. This solution is read and the adsorbance is subtracted from the reading of the sample.

5.3.4. In some instances, the adsorbance reading will be due largely to turbidity. If the reading is greater than the high standard, the sample needs to be diluted and reanalyzed. If the client needs lower reporting limits than this can achieve:

1. Filter an aliquot of the sample, and spike an aliquot of sample and then filter. If the recovery is 85-115%, the method is in control.

2. Analyze the sample by SW 846 7197. This method is not affected by sample turbidity.

5.3.5 Compound Quantification

Cr^{+6}, mg/L = Measured concentration at instrument * dilution

If a soil sample was analyzed, the mg/L value is divided by the %Total Solids/100.

6. Reports

6.1. Data Packet Organization

6.1.1. Each data package will contain a bench sheet showing all volumes, weights, dilutions, dates and analyst's initials, a copy of the instrumental output, and a copy of the Quality Control report.

6.2. Quality Control Reports

6.2.1. All results for quality control tests are entered into the lab data base using the quality control database. A summary of all data entered must be included in the data packet. The routine minimum is a method blank report, and an MS/MSD or MS/duplicate report. Many analyses will also require an SRM, blank spike or other report.

6.3. Sample Result Reports

6.3.1. Data Qualifying Flags

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

CODE	Definition
U	: The analyte of interest was not detected, to the limit of detection indicated.

7. References

USEPA Test Methods for Evaluating Solid Wastes, Vol. IA, Latest Revision, Method 7196A.

Appendix I

QC Summary Table

Laucks Testing Laboratories
Method 7196A QA Requirements and Corrective Actions

QA Element	Method Criterion	Laucks Criterion	Frequency	Corrective Action	Documentation
Initial Calibration	Multi-point	Multi-point R>.995	One per run	Recalibrate	Printout of calibration
Initial Calibration Verification	Required One per 15 samples	Manufacturer supplied, or 90-110% if not specified	One per run	Recalibrate or Reanalyze	Printout of result
Initial Calibration Blank	One per batch	< ½ Reporting limit	One per run	Reanalyze	Printout of result
Continuing Calibration Verification	Not required	90-110 % recovery	One after every 10 samples, and at the end of the run.	Recalibrate	Printout of result
Method Blank	One per batch	< ½ Reporting limit	One per run	Reanalyze	Printout of result
Continuing Calibration Blank	Not required	< ½ Reporting limit	One after every 10 samples, and at the end of the run.	Recalibrate	Printout of result
MDL standard recovery	Not required	Detectable	One per run	Recalibrate	Printout of result
Matrix Spike Recovery	85-115 % One per matrix	See QC database	One MS/MSD or one MS/DUP per 20	Dilute and Reanalyze or Out of Control Document filed	Printout of result
*MS/MSD RPD	Not Required	See QC database	One MS/MSD or one MS/DUP per 20	Out of Control Document filed	Printout of result
*Duplicate % Difference	Not Required	See QC database	One MS/MSD or one MS/DUP per 20	Dilute and Reanalyze or Out of Control Document filed	Printout of result

* Either an MSD or a Duplicate/MS will be analyzed

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-7501

Title: **Operation of the PE FIMS 400 for the Analysis of Hg for EPA Methods SW-846 7470/7471, 245.1/245.5, and CLP ILM04.1**

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Table of Contents

1. INTRODUCTION AND SCOPE 3
1.1 METHOD DESCRIPTION: 3
1.2 SAMPLE COLLECTION, SAMPLE STORAGE, HOLDING TIMES 3
1.3 INTERFERENCES 3
1.4 DEFINITION OF TERMS 4
2. EQUIPMENT, STANDARDS AND REAGENTS 6
2.1 EQUIPMENT 6
2.2 STANDARDS 6
2.3 REAGENTS: 7
3. SAFETY PRECAUTIONS AND WASTE DISPOSAL 8
3.1 SAFETY PRECAUTIONS 8
4. CALIBRATION AND QUALITY CONTROL 9
4.1 METHOD DETECTION LIMIT STUDY (MDL). 9
4.2 INITIAL MULTI-POINT CALIBRATION: 9
4.3 CALIBRATION CRITERIA 9
4.4 CONTINUING CALIBRATION: 10
4.5 MATRIX SPIKE 10
4.6 MATRIX SPIKE DUPLICATE 11
4.7 SAMPLE DUPLICATE 12
5. OPERATION PROCEDURES 12
5.1 DIGESTION SET-UP - BOD BOTTLES - SOIL OR WATER: 12
5.2 DIGESTION - WATER SAMPLES 13
5.3 DIGESTION - SOIL SAMPLES 14
5.4 ANALYSIS ON FIMS 400 15
5.5 SAMPLE INFORMATION SHEET. 15
6. REPORTS 18
6.1 DATA PACKET ORGANIZATION 18
6.2 DATA QUALIFYING FLAGS 18
6.2 CONTROL CHARTS 18
7. REFERENCES 18
APPENDIX I - QC SUMMARY TABLE 19
APPENDIX - II - SAMPLE DIGESTION LOG 21
APPENDIX III - EXAMPLE REPORT FORM 23

1. Introduction and Scope

1.1 Method Description:

1.1.1 This SOP covers the cold vapor analysis of Hg using the Perkin-Elmer Flow Injection Mercury System, Model 400 (FIMS 400). Sample handling and preparation are as required by SW-846 methods 7470/7471 and 245.1/245.5. This SOP is also valid for the preparation and analysis of samples under CLP protocol when used in conjunction with the SOW ILM04.1. Where the requirements of these procedures differ, the more stringent requirements are used. The requirement of analyzing soil samples in triplicate under 7471 is not used except upon request.

1.1.2 After preparation and digestion to convert Hg to its ionic form, 0.5 mL of sample is introduced into a stream of carrier solution (3% HCl), mixed with a $SnCl_2$ suspension to reduce the Hg ion to its metallic form, which is then carried into an absorption tube through which passes light at a wavelength of 253.7 nm. The absorption is then measured.

1.1.3 This method is restricted to use by, or under the supervision of analysts experienced in the technique described. Each analyst performing this method must have demonstrated the ability to perform the described analysis.

1.2 Sample Collection, Sample Storage, Holding Times

1.2.1 Samples are stored either in glass or plastic. The holding time is 28 days from collection. Soil samples are stored at 4° ±2° C. Water samples are stored at room temperature. Water samples are preserved by the addition of HNO_3 to a pH <2.

1.3 Interferences

1.3.1 This section covers interferences which may potentially occur with the FIMS associated with sample or standard preparation or gas or liquid flow through the system.

1.3.2 Foaming

1.3.2.1 If excess foaming results in liquid being transported into the quartz cell, the reagent stream should be stopped immediately, and the tubing and cell assemblies should be disassembled and cleaned.

1.3.3 Precipitates

1.3.3.1 All solutions used with the FIMS (samples, carrier solutions, and reducing agents) must be free of solid particles since they are transported through the narrow tubing and valve openings. In addition, the formation of precipitates during the course of a reaction must be avoided. When such a case occurs, an appropriate digestion or separation method should be used to remove the offending component.

1.3.4 Contamination

1.3.4.1 The detection limits obtainable using the FIAS-System are very often a function of the degree of contamination in the samples and the reagents rather than of instrument capabilities. Contamination is the main source of error when working at the nanogram levels typical for FIMS analyses. Careful operation of the instrument, selection of proper sample preparation techniques, and good laboratory cleanliness practices are the most important prerequisites for minimizing contamination.

1.4 Definition of Terms

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

1.4.2 **LFB** - Laboratory Fortified Blank - A sample of DIW to which a known amount of Hg is added.

1.4.3 **CRA** – a sample of DIW to which a known amount of Hg is added. This is a low concentration to verify the curve is valid near the reporting limit of 0.2 µg/L.

1.4.4 **CCB** - Continuing Calibration Blank - This is the same acronym used in the CLP program. This is a blank which is analyzed immediately after the CCV (almost always after every 10 samples and at the end of the analytical run) during the analysis sequence to determine whether the instrument or system has maintained a stable baseline.

1.4.5 **CCV** - Continuing calibration verification. - This is the same acronym used in the CLP program. This is a standard analyzed at some prescribed frequency (almost always after every 10 samples and at the end of the analytical run) during the analysis sequence to determine whether the instrument or system has remained in calibration.

1.4.6 **CLP** - Contract Laboratory Program - The USEPA program that contracts with laboratories to provide laboratory services. The term has come to mean a much broader set of methods and deliverables. In context of this SOP, CLP means procedures or operations which are detailed in the CLP contract and which are extended to a broader working definition.

1.4.7 **Corr Coef, CC** - Correlation coefficient - A measure of the "goodness of fit" of a set of data to a linear regression model. The closer the value is to 1, the higher the degree of confidence in the correlation

1.4.8 **DIW** - Deionized water - Lab reagent water. This water should be free of virtually all analytes.

1.4.9 **ICB** - Initial calibration blank - This term is borrowed from CLP. An instrument blank is made up in the same way as calibration standards, without target analytes.

1.4.10 **ICV** - Initial calibration verification - is a standard which is analyzed at the start of each analytical run that is compared to the initial multi-point calibration to determine whether the instrument calibration is accurate. This verification standard is from a source different from that used to make the calibration standards

1.4.11 **IDL** - Instrument detection limit. The lowest concentration of a target analyte that will yield a signal:noise ratio of least 3x. Determined quarterly using blanks to which a known quantity of standard has been added. The IDL is determined by analyzing 7 replicates spiked at 2-5 times the expected IDL on three non-consecutive days. The sum of the standard deviations of the three runs is the IDL.

1.4.12 **MDL** - Method detection limit - The lowest concentration a sample which will yield a positive result that is greater than zero at a known level of confidence. MDLs are empirically determined by Laucks annually.

1.4.13 **MDL** standard - Method detection limit standard - A standard prepared so that the concentration of Hg is 1-5 times the anticipated MDL

1.4.14 **LCS** - Laboratory Control Sample - A sample containing a known quantity of Hg used to verify digestion and analysis. LCSW indicates a water sample. It is used interchangeably with ICV. LCSS indicates a soil sample.

1.4.15 **RSD or %RSD** - Relative standard deviation or percent relative standard deviation - The ratio of the standard deviation of a set of values to the mean of the set of values. A measure of the similarity of the values one to another.

1.4.16 **PBW** - A blank carried through the sample preparation and digest procedures.

2. Equipment, Standards and Reagents

2.1 Equipment

2.1.1 Equivalent equipment may be used where appropriate.

FIMS 400 automatic Hg analyzer
Perkin Elmer AS-90 autosampler
Epson LQ300 dot-matrix printer
Hot water bath
BOD bottles
Analytical balance
Micropipettes: 20, 50, 100, 200, 500 and 1000 µl
50 mL and 15 mL plastic centrifuge tubes

2.2 Standards

2.2.1 All standards must be prepared in 0.15% HNO_3.

2.2.2 **1000 ppm Hg stock solution**: Vendor supplied. Currently purchased from Inorganic Ventures.

2.2.3 **Working Hg solution**: Dilute 100 µl of 1000 ppm Hg stock solution to 100 mL in DIW to which 0.15 mL HNO_3 has been added. The identification number of the standard solution used and the dilution must be entered in the standards logbook. This solution must be prepared daily. The identification number of the working standard must also be recorded on the bench sheet (digestion log-Appendix II).

2.2.4 **0.2 µg/L standard** - Place 100 mL DIW in a BOD bottle. Add 20 µl working Hg solution.

2.2.5 **0.5 µg/L standard** - Place 100 mL DIW in a BOD bottle. Add 50 µl working Hg solution.

2.2.6 **1.0 µg/L standard** - Place 100 mL DIW in a BOD bottle. Add 100µl working Hg solution.

2.2.7 **2.0 µg/L standard** - Place 100 mL DIW in a BOD bottle. Add 200 µl working Hg solution.

2.2.8 **5.0 µg/L standard** - Place 100 mL DIW in a BOD bottle. Add 500 µl working Hg solution.

2.2.9 **10.0 µg/L standard** - Place 100 mL DIW in a BOD bottle. Add 1000 µl working Hg solution.

2.2.10 **100 ppm Hg stock solution**: Vendor supplied. Currently purchased from Spex Certiprep.

2.2.11 **ICV Standard** - Dilute 1000µl of 100 ppm Hg stock solution to 100 mL in DIW to which 0.15 mL HNO_3 has been added. The identification number of the standard solution used and the dilution must be entered in the standards logbook and the digestion log (Appendix II). This solution must be prepared monthly from an independent stock solution, ie a different vendor than the stock solution used to prepare the calibration and quality control samples.

2.2.12 **ICV solution** - Add 100 mls DIW to a BOD bottle. Add sufficient ICV solution to produce a concentration of 1.0 to 5.0 µg/L. Currently a concentration of 4.0 µg/L is in use.

2.2.13 **CCV/CCB/Blank Spike (Soils only)** - In setting up the run, use the 5.0 µg/L standard for the CCV and Blank Spike; for the CCB use the calibration blank.

2.2.14 **MS/MSD or MS/Duplicate** - Add 500 µl of the working Hg solution to the samples being spiked. When performing Hg under CLP protocols use 100 µl of the working Hg solution and perform MS/Duplicate.

2.2.15 **LCSS** - Laboratory control soil sample. Concentrations and limits are certified by the vendor.

2.3 **Reagents**:

2.3.1 DIW - deionized water free of impurities.

2.3.2 H_2SO_4 (sulfuric acid) - concentrated, reagent grade

2.3.3 HNO_3 (nitric acid) - concentrated, reagent grade

2.3.4 HCl (hydrochloric acid) - concentrated, reagent grade

2.3.5 Stannous chloride - Add 27.5 g. $SnCl_2 \cdot 2H_2O$ (stannous chloride dihydrate) to 2.5 L DIW to which has been added 75 mL concentrated HCl.

2.3.6 $KMnO_4$ (potassium permanganate) - Dissolve 100 g. $KMnO_4$, marked "suitable for Hg determination", in 2 L DIW. This is a saturated solution. Sufficient undissolved $KMnO_4$ should be allowed remaining in the bottom of the bottle to assure a saturated solution.

2.3.7 $K_2S_2O_8$ (potassium persulfate) - Dissolve 50 g. $K_2S_2O_8$, marked "suitable for Hg determination", in 1 L DIW. This is a saturated solution. Sufficient undissolved $K_2S_2O_8$ should remain in the bottom of the bottle to assure a saturated solution.

2.3.8 $NaCl/NH_2OH{\cdot}HCl$ (sodium chloride/hydroxylamine hydrochloride) - Dissolve 240 g NaCl (table salt is generally used) and 240 g $NaCl/NH_2OH{\cdot}HCl$ (reagent grade marked "suitable for Hg determination") in 2 L DIW. Contamination has been experienced with this solution. For this reason a 50% solution should be analyzed by the FIMS 400 whenever a new bottle is made up. If the absorbance (peak height) exceeds .004 the solution of $NaCl/NH_2OH{\cdot}HCl$ should be disposed and remade.

2.3.9 Carrier solution - The carrier is a 3% HCl solution.

3. Safety Precautions and Waste Disposal

3.1 Safety Precautions

3.1.1 All standards, samples and sample solutions should be handled as if they are hazardous substances.

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

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

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

3.1.5 Because of the toxic nature of Hg vapor, inhalation or skin contact should be avoided.

3.1.6 The usual precautions should be taken in handling acids.

3.1.7 $SnCl_2$ is a skin and eye irritant; avoid contact.

3.1.8 The addition of hydroxylamine after digestion may release gas including chlorine. Avoid inhalation; use hood if necessary.

3.1.9 Waste disposal – See SOP LTL-2001 for general information on waste disposal. In addition, sample digestates are acidic in nature and must be neutralized prior to disposal.

4. Calibration and Quality Control

4.1 Method Detection Limit Study (MDL).

4.1.1 The estimated MDL for waters is currently 0.05 µg/L. The estimated MDL for soils is currently 0.04 mg/kg.

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

4.2 Initial Multi-Point Calibration:

4.2.1 Concentrations:

Standard Name	Concentration
S0	0.0 µg/L
S0.2	0.2 µg/L
S0.5	0.5 µg/L
S1.0	1.0 µg/L
S2.0	2.0 µg/L
S5.0	5.0 µg/L
S10.	10.0 µg/L

Standards are prepared fresh daily from the working standard in 0.15% HNO_3. See section 2.2.2 through 2.2.8 for the preparation details.

4.2.2 The calibration blank, the ICV, the LCS, the Blank Spike, the ICB and all standards are digested along with the samples being analyzed.

4.3 Calibration Criteria

4.3.1 Calibration curve – "best fit" of points in calibration curve. Linear correlation coefficient (R) must be equal to or greater than 0.995.

4.3.2 ICV - This is run immediately following a successful curve. This is to verify from a source of mercury independent from the calibration source that the curve is valid. For CLP and EPA 245.1/245.5 analyses the recovery limits are 80-120 %, for SW-846 7470/7471 the limits are 90-110%. If recovery is outside of the control limits an immediate reanalysis is permitted. If still out of control the curve must be reanalyzed followed by another ICV otherwise continue with an ICB.

4.3.3 ICB – This is run immediately following a successful ICV. This is to verify that there is no contamination and that the curve is valid. Limitations presently in use are ±0.1 µg/L (1/2 of the reporting limit). If recovery is outside the limit an immediate reanalysis is permitted. If still out of control, a corrective action must follow and a reanalysis of the curve.

4.3.4 A PBW and an LFB are run immediately following the successful calibration. The limitations on the PBW are the same as the limitation on the ICB. The limitations on the LFB are as set forth in the laboratory QC database (80-120%).

4.3.5 If the above criteria are not met, recalibration must be performed.

4.4 Continuing Calibration:

4.4.1 A CCV followed immediately by a CCB must be run every 10 samples and after the last sample. The ICB, PBW and Blank Spike count as samples for this purpose.

4.4.2 Criteria - For CCV's the recovery limitations are 80% to 120%. For blanks the limitations are ±0.1 µg/L.

4.4.3 Corrective action - If a CCV or CCB is out of control, an immediate reanalysis is permitted. However if the reanalysis is out of control a reanalysis of all samples since the previous successful CCV and/or CCB must be performed.

4.5 Matrix Spike

4.5.1 A sample is chosen at random from the samples to be analyzed, and 500 µl of the working standard is added (100µl for CLP). The spike level is then 5.0 µg/L(1.0 µg/L for CLP). The analyst should attempt to avoid selecting samples which are identified by the client as blanks. As the purpose of the matrix spike is to test the system under "typical" conditions, the analyst may also avoid selecting the most difficult sample of the batch for spiking. It is not always required that a matrix spike analysis be performed with each preparation/analysis batch, however, the minimum frequency for MS analysis is 1 each per 20 samples per matrix. This will be best accomplished by running one with every batch for many analyses. This matrix spike sample is used to evaluate the matrix effect

of the sample upon recovery of the analytes. The recovery of spike analytes is calculated as follows:

$$\text{recovery,\%} = \frac{(SS - S)}{SA} * 100$$

where:
SS = concentration in spiked sample
S = native concentration in unspiked sample
SA = spiked added, the amount of spiking material actually added to the sample calculated on the sample basis

4.5.2 The recovery criteria are listed in the quality control database and will change from time to time. Current acceptable recovery limits are 85-116% for SW-7470. All other methods are 75-125%.

4.5.3 Corrective action

4.5.3.1 Samples with spike recoveries outside control limits will be reviewed for possible corrective action. Corrective action will first involve recalculation, followed by possible re-preparation, and/or reanalysis. This process should also look at the recovery of Hg from the LCS and/or blank spike analysis. In all cases a narrative explanation of the condition is required to detail the corrective actions taken.

4.6 Matrix Spike Duplicate

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

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

where:
S1 = measured concentration for MS sample
S2 = measured concentration for MSD sample

4.6.2 RPD control limits are listed in the quality control database and will change from time to time. The currently acceptable RPD limit is 20%.

4.7 Sample Duplicate

4.7.1 Sample duplicates are required when CLP practices are employed, or when the method specifically calls for duplicates. At least one duplicate sample per 20 samples per matrix is required when matrix spikes are being performed.

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

where:
S1 = measured concentration in the initial analysis
S2 = measured concentration in the duplicate analyses

4.7.2 The RPD control limits listed in the quality control database and will change from time to time.

4.7.3 Corrective action

4.7.3.1 If a trend in out of control RPD values is observed, the methods used must be examined to determine the source of variance. Once this source is identified, the method must be changed so that samples can be analyzed with a predictable reproducibility. Generally, if recoveries are in control and no analyte of interest was detected in any of the samples, no immediate action will be taken on that sample set. If integrity of reported sample values is in doubt, re-analysis may be called for. Corrective actions should be discussed with the Quality Assurance Officer.

5. Operation Procedures

5.1 Digestion Set-up - BOD bottles - Soil or Water:

5.1.1 Digestion log (Appendix II): The digestion log should list the sample numbers, the date, the name of the analyst, the time placed in the hot water bath, the time when the temperature reached 95 ± 5° C, and the time removed from the hot water bath. One should be prepared for each run. An example is attached as Appendix II. The numbers of each BOD bottle must be placed in the leftmost column of this form to identify which sample is in each BOD bottle.

5.1.2 Preparation of standards:

5.1.2.1 Prepare a working standard daily by adding 100 µl of the 1000 mg/L stock standard to 100 mL of 0.15% HNO_3 in DIW, giving a concentration of 1.0 mg/L of Hg.

5.1.2.2 Place 100 mL DIW in each of 9 BOD bottles. Mark these BOD bottles with the concentration of each standard in µg/L, plus one marked "Blank", one marked "ICV", and one more marked ICB.

5.1.2.3 Add from the working standard the following amounts to the following bottles:

0 µg/L	0 µl
0.2 µg/L	20µl
0.5 µg/L	50 µl
1.0 µg/L	100 µl
2.0 µg/L	200 µl
5.0 µg/L	500 µl
10.0 µg/L	1000 µl

5.1.2.4 To the ICV BOD bottle add that amount of the ICV standard which will produce a concentration from 1.0 µg/L to 5.0 µg/L. Currently a contration of 4.0 µg/L is in use.

5.1.2.5 The standards, the ICV and the ICB must be digested along with the PBW, Blank Spike and samples.

5.2 Digestion - Water Samples

5.2.1 These procedures are for methods SW-846 7470 and EPA 245.1 unless noted otherwise.

5.2.2 Place 100 mL DIW in the PBW BOD bottle.

5.2.3 Place 100 mL DIW + 500 µl intermediate standard in the Blank Spike BOD bottle.

5.2.4 Place 100 mL sample in the BOD bottle designated for that sample, being sure to place 100 mL sample in the designated duplicate, MS and MSD BOD bottles for the sample involved. TCLP samples and spikes should be diluted 20 mL/100 mL (dilution factor of 5) in DIW. The TCLP spikes should be diluted 2 mL/100 mL (dilution factor of 50) in DIW.

5.2.5 Add the required quantity (See Sec. 4.5.1) of the working standard to the MS and MSD BOD bottles.

5.2.6 Add the following reagents to each BOD bottle (for all methods):

5 mL concentrated H_2SO_4
2.5 mL concentrated HNO_3
15 mL $KMnO_4$ solution
8 mL $K_2S_2O_8$ solution

5.2.7 The $KMnO_4$ color must persist for 15 minutes. If it does not, more $KMnO_4$ must be added.

5.2.8 Place all the BOD bottles, being careful to keep them in order, in the hot water bath in a hood, and record the time on the digest log. Monitor the temperature of the hot water bath and record the time when the temperature reaches 90°C. Continue to heat the water bath, keeping the bath at 95°C ± 5°C for two hours.

5.2.9 Remove the BOD bottles from the hot water bath and allow to cool.

5.2.10 Add 7 mL $NaCl/NH_2OH{\cdot}HCl$ solution and mix until the mixture clears. If some undissolved permanganate remains, it will usually disappear on standing after a few minutes.

5.3 Digestion - Soil samples

5.3.1 These procedures are (for Methods SW-846 7471 and EPA 245.5 unless noted otherwise):

5.3.2 Place 50 mL DIW in the calibration BOD bottles. Add appropriate working standard to each.

5.3.3 Place approximately 0.5 g, or for certain CLP and SW-846 analyses, approximately 0.2 g of sample in each of the sample BOD bottles to which that sample applies. Record the exact weight on the digestion log.

5.3.4 Add 3.75 mL HCl , 1.25 mL HNO_3 to all the BOD bottles and 10 ml DIW to each sample BOD bottle.

5.3.5 Place all sample BOD bottles in a hot water bath in a hood that is at 95°C ± 5°C. Maintain the heat at this level for 10 minutes.

5.3.6 Cool for 20-30 minutes and add 40 mL DIW and 15 mL $KMnO_4$ to all sample BOD bottles (also add 8 mL $K_2S_2O_8$ for CLP). Mix.

5.3.7 The $KMnO_4$ color must persist for 15 minutes. If it does not, more $KMnO_4$ must be added.

5.3.8 Place all BOD bottles in a hot water bath that is at 95°C ± 5°C. Record the time on the digestion log. Maintain the hot water bath at this temperature for 30 minutes. Remove the BOD bottles and allow to cool.

5.3.9 Add 7 mL $HCl/NH_4NH_2OH{\cdot}HCl$ solution to all BOD bottles and mix to remove color.

5.3.10 Add 50 mL DIW to all BOD bottles. Allow to cool.

5.4 Analysis on FIMS 400

5.4.1 This is a programmed analysis. This is a 6 point curve with a blank correction. The development of the calibration curve, including the ICV and ICB, is automatic. If the calibration is unsuccessful, recalibration is attempted. The 5.0 µg/L standard is used for CCV's; the calibration blank for CCB's, both of which are programmed to be run every 10 samples. If a CCV or a CCB fails to meet quality control specifications, the system stops and all samples since the last preceding valid CCV or CCB are reanalyzed. A CCV and a CCB are also run after the last sample in the run.

5.5 Sample Information sheet.

5.5.1 Click the basic functions button on the screen which appears after activating the program.

5.5.2 Click the "Sample Information" button on the toolbar.

5.5.3 A blank sample information sheet appears. Previous sample information sheets may be obtained by activating the File - Open menu, designating "Sample Information" from the dialog which appears. The sample information sheet should be filled in to tell the computer to which auto-sampler locations to go.

5.5.4 Sample ID column - Some typing can be saved by double clicking on the column title. On the resulting form the job order may be typed in and the sample numbers will then be incremented by 1 for the autosampler locations indicated. The sample ID's will then be filled in. Sample: "0102094-0001..0002. .".

5.5.5 Sample weight column - For soil samples the weight of the samples should be filled in. At the top, the volume units should be filled in "mL", the weight units "g".

5.5.6 Sample units column - Typing may be saved by double clicking on the column title and indicating the units on the dialog box which appears, and indicating the AS locations

involved. In the case of water samples this will ordinarily by "µg/L ". For soil samples the button "weight:weight" should be clicked and the desired units specified in the dialog box selected. If the dialog box has been properly filled in, the machine is programmed to compute the proper units (e.g. mg/kg). It should be kept in mind that this is on an as received basis, not a dry basis.

5.5.7 User dilution column - Where a dilution is involved (e.g. TCLP analyses) the dilution factor should be entered. As in the sample weight and sample units columns, above, typing may be saved by double clicking the column title.

5.5.8 The autosampler tray being used at present is "Tray B".

Sampler Location	Size	Contents
1	50 mL tube	Calibration Blank
2	50 mL tube	0.2 µg/L standard
3	50 mL tube	0.5 µg/L standard
4	50 mL tube	1.0 µg/L standard
5	50 mL tube	2.0 µg/L standard
6	50 mL tube	5.0 µg/L standard
7	50 mL tube	10.0 µg/L standard
8	50 mL tube	ICV
9	15 mL tube	ICB
10	15 mL tube	LCSS/LCSW
11	15 mL tube	CRA
12	15 mL tubes	PBW
13-109	15 mL tubes	Samples

5.5.9 All tubes should be marked with their contents before being filled. Then the tubes are filled from the BOD bottles and placed in the autosampler locations designated either by the method or by the sample information sheet.

5.5.10 At the beginning of each day of analysis, the following steps are taken to prepare the instrument:

5.5.10.1 The flows of the carrier solution and the reductant solution (the stannous chloride solution) are measured by the methodology suggested in the Perkin-Elmer handbook "Setting Up and Performing Analyses", 2.4.2-2.4.4.

5.5.10.2 The rinse container (autosampler location 0) is rinsed and filled with 3% HCl solution. This is then analyzed using the location button in the analysis window of the automated analysis window, and the curve produced displayed through the curve button in the toolbar. The curve should be flat and less than 0.0004 absorbance units.

5.5.11 Analysis - The analysis is ordinarily started by clicking the "Analyze All" button in the Automated Analysis window. If only calibration is desired, the "Calibrate" button may be used. If samples are to be analyzed by an already existing calibration curve use the "Analyze Samples" button.

5.5.11.1 Once the analysis has been started, the autosampler places the sampling probe in the sample/calibration cup. The peristaltic pump begins pulling sample through the sample loop. After the line has been purged, the injection valve cycles and injects the sample into the carrier flow. This flow is then combined with the $SnCl_2$ and mixed. Next it goes to a liquid/vapor separator. The liquid goes to waste and the vapor is transported into the absorption cell of the spectrophotometer. All samples and standards are measured in duplicate.

5.5.11.2 Run log - A run log will be printed as the analysis progresses. The print program is designed to transmit the data to be printed to the printer one page at a time. Thus several autosampler locations will be analyzed before anything appears on the printer. If anything else is printed (e.g. the sample information sheet) while the automated analysis window is open, the computer must be rebooted, or a run log will not be printed.

5.5.11.3 Additional samples may be analyzed after the run designated on the sample information sheet by using the "Select Location" button on the "Analyze" page of the Automated Analysis window. The autosampler locations should be filled in the resulting dialog. The instrument will then analyze the samples indicated. This may be used to reanalyze the samples with questionable results, or to analyze diluted samples.

5.5.11.4 Dilution - Where analysis results are above the high standard (10.0 µg/L) an aliquot of the sample diluted with the calibration blank may be analyzed as set forth in 5.5.11.3 above.

6. Reports

6.1 Data Packet Organization

6.1.1 A report is prepared using the reformat procedure on the main menu. The reformatted design is entitled "Summary". It is transferred to a floppy disk, which is then imported to a spreadsheet using Microsoft Excel 2000. This must then be formatted into the form included in Appendix III.

6.1.2 The final data packet is to include the following:

1) The report (See Appendix III).
2) The quality control database Report Form
2) The digestion log (Appendix II).
4) The run log, if required.

6.1.3 All results for quality control tests are entered into the quality control database. A summary report of all data entered must be included in the data packet. The routine minimum is a method blank report, an MS/MSD or MS/duplicate report and a SRM/LCSW.

6.2 Data Qualifying Flags

Flag	Definition
U	The analyte of interest was not detected, to the limit of detection indicated.

6.2.1 Control charts

7. References

USEPA, Test Methods for Evaluating Solid Waste Physical/Chemical Methods, USEPA SW846, most recent version, Method 7470/7471

USEPA, Determination of Mercury in Water by cold vapor atomic absorption spectrometry, USEPA 245.1/245.5, most recent version, Method 245.1/245.5

CLP Inorganics Statement of Work, ILMO4.1

Appendix I - QC Summary Table

Laucks Testing Laboratories
Method 245.1/245.57471/7470, & CLP ILM04.1 QA Requirements and Corrective Actions

QA Element	Method Criterion	Laucks Criterion	Frequency	Corrective Action	Documentation
Initial Calibration	R > 0.995	R > 0.995 6-point curve + blank	once per batch	recalibrate	run log
Initial Calibration Verification	SW846 90-110% EPA 245 and CLP 80-120%	Per the method	Once per batch	recalibrate	run log, report
Initial Calibration Blank	Below MDL	Below ½ R.L.	one per batch	recalibrate	run log, report
Method Blank (PBW)	Below R.L.	Below ½ R.L.	One per batch	Consult QC officer. Flag data or reanalyze.	Quality control database
Continuing Calibration Verification	80-120 %	80-120 %	every 10 samples and after the last sample	rerun samples since last CCV	run log
Continuing Calibration Blank	Below MDL	Below ½ R.L.	every 10 samples and after the last sample	rerun samples since last CCB	run log
Matrix Spike Recovery	See method	See quality control database	Every 20 samples	Consult QC officer. Flag data or reanalyze.	Quality control database
MS/MSD RPD	See method	See quality control database	Every 20 samples	Consult QC officer. Flag data or reanalyze.	Quality control database

QA Element	Method Criterion	Laucks Criterion	Frequency	Corrective Action	Documentation
Duplicate % Difference	See method	See quality control database	Every 20 samples	Consult QC officer. Flag data or reanalyze.	Quality control database
Standard Reference Material (LCS) Recovery	Soil Vendor's limits Water See ICV	Vendor's limits	Once per 20	Recalibrate	Run log

Appendix - II - Sample Digestion Log

insert log here

Appendix III - Example Report Form

Hg in Water by SW-7470
Digested 11/13/2000
Analyzed 11/14/2000

Sample	Time	Value		% Rcvry	RPD	Flag
Calib Blank	11:49:54		µg/L			
S0.2	11:52:45		µg/L			
S0.5	11:55:38		µg/L			
S1.0	11:58:33		µg/L			
S2.0	12:01:30		µg/L			
S5.0	12:04:26		µg/L			
S10.0	12:07:22		µg/L			
ICV1 ME-13-4-1=4	12:10:26	3.951	µg/L	99%		
ICB	12:13:21	0.021	µg/L			
LCSW1	12:16:10	3.964	µg/L	99%		
CRA	12:19:03	0.199	µg/L			
PBW	12:21:57	0.017	µg/L			
0011185-01	12:24:51	0.023	µg/L			
011185-02	12:27:45	0.021	µg/L			
011185-03	12:30:42	0.024	µg/L			
011185-04	12:33:37	0.015	µg/L			
011185-04D	12:36:31	0.018	µg/L			
011185-04MS Spk=5	12:39:21	5.076	µg/L	102%		
CCV1=5	12:42:14	5.143	µg/L	103%		
CCB1	12:45:08	0.026	µg/L			
011185-05	12:47:58	0.017	µg/L			
011185-06	12:50:49	0.019	µg/L			
011185-07	12:53:40	0.021	µg/L			
011185-08	12:56:32	0.016	µg/L			
011185-09	12:59:24	0.017	µg/L			
011185-10	13:02:17	0.013	µg/L			
011185-11	13:05:12	0.009	µg/L			
0011143-02	13:08:05	0.016	µg/L			
011143-02MS Spk=5	13:10:59	5.160	µg/L	103%		
CCV2=5	13:13:54	5.143	µg/L	103%		
CCB2	13:16:46	0.028	µg/L			
011143-02MSD	13:19:39	5.150	µg/L	103%	0%	
0011149-02	13:22:34	0.018	µg/L			
0011167-02	13:25:31	0.016	µg/L			
011167-03	13:28:27	0.017	µg/L			
0011168-02	13:31:21	0.029	µg/L			
011168-03	13:34:10	0.017	µg/L			
0011196-02	13:37:01	0.021	µg/L			
011196-03	13:39:51	0.023	µg/L			
0011198-02	13:42:42	0.021	µg/L			
CCV3=5	13:45:34	5.163	µg/L	103%		
CCB3	13:48:28	0.026	µg/L			

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP : LTL-9104

Title: **Preparation and Analysis of Total and Amenable Cyanide according to EPA Methods 335.1, 335.3, and SW 846 method 9012A.**

Revision history:

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Table of Contents

1. INTRODUCTION AND SCOPE 3
1.1 METHOD DESCRIPTION 3
1.2 METHOD DEVIATIONS AND COMMENTS 3
1.3 METHOD SUMMARY 3
1.4 SAMPLE HANDLING, SAMPLE STORAGE, HOLDING TIMES 4
1.5 DEFINITION OF TERMS 4

2. EQUIPMENT, REAGENTS, AND STANDARDS 6
2.1 EQUIPMENT 6
2.2 REAGENTS 6
2.3 STANDARDS 7

3. SAFETY PRECAUTIONS 7

4. CALIBRATION AND QUALITY CONTROL 8
4.1 METHOD DETECTION LIMIT STUDY 8
4.2 INITIAL MULTI-POINT CALIBRATION 8
4.3 INITIAL CALIBRATION VERIFICATION 9
4.4 INITIAL CALIBRATION BLANK 9
4.5 CONTINUING CALIBRATION VERIFICATION (CCV) AND BLANK (CCB) 9
4.6 PREPARATION BLANKS 9
4.7 MATRIX SPIKE 10
4.8 MATRIX SPIKE DUPLICATE 11
4.9 BLANK SPIKE 11
4.10 SAMPLE DUPLICATE 11

5. OPERATION PROCEDURES 12
5.1 PRE-DISTILLATION SAMPLE SCREEN FOR WATERS 12
5.2 PRETREATMENT FOR CYANIDES AMENABLE TO CHLORINATION 12
5.3 MANUAL DISTILLATION 13
5.4 INTERFERENCES 14

6. SAMPLE ANALYSIS USING A TECHNICON AUTO ANALYZER 15
6.1 INSTRUMENT START-UP AND OPERATION 15
6.2 ANALYSIS PROCEDURE 15
6.3 COMPOUND QUANTIFICATION 16

7. REPORTS 17
7.1 DATA QUALIFYING FLAGS 17

APPENDIX I 18
METHOD LTL 9104 QA REQUIREMENTS AND CORRECTIVE ACTIONS 18

APPENDIX II 19
EXCEL SPREADSHEET USED FOR CYANIDE CALCULATIONS. 19

APPENDIX III 20
AUTOANALYZER FLOW DIAGRAM 20

1. Introduction and Scope

1.1 Method Description

Cyanide in samples is determined by a preparation with manual distillation followed by an automated spectrophotometric analysis on an auto analyzer with a UV digester and in-line distillation. The routine reporting limit is 10 µg/L in waters, and 0.50 mg/kg DB in soils and sediments. Pretreatment for the determination of cyanides amenable to chlorination in waters is also included in this SOP.

This SOP is applicable to the sample preparation (for cyanide analysis) of drinking waters, surface and saline waters, domestic and industrial wastes according to EPA methods 335.1, 335.3 and SW 846 method 9012A with the main difference being the use of the in-line still and UV digestion in EPA 335.3. Also, MIDI distillation with a 50 mL water sample instead of the normal distillation of a 500 mL sample is used in this SOP. Other differences from 9012A are listed on the QA chart at the end of this SOP. Soils and sediments are MIDI distilled as a 1 g sample.

This method is restricted to use by, or under the supervision of analysts experienced in the technique described and in SW 846 methodology. Each analyst performing this method must have demonstrated the ability to perform the described analysis.

1.2 Method Deviations and Comments

The following items represent deviations of Method 9012A, as published, which are followed as standard operating procedures in the performance of this method at Laucks:

- The MIDI distillation procedure is used, exactly as written in the CLP statement of work. This distillation uses a 50 mL aliquot of sample and a final distillation volume of 50 mls. This procedure allows the lab to use less sample, and dispose of smaller volumes of waste material. All molar ratios in this distillation are identical to the method as written.

- The calibration curve specified in 9012A is reduced by 2 to compensate for the lack of sample concentration in the distillation step.

1.3 Method Summary

Sample Preparation: The MIDI distillation procedure applies to relatively solid samples such as soils, sediments and sludges, or if requested to water samples. Cyanide determined by this method is defined as cyanide ion and complex cyanides converted to hydrocyanic acid by

reaction in a reflux system with mineral acid in the presence of magnesium ion. The cyanide as hydrocyanic acid (HCN) is released from cyanide complexes by means of a MIDI reflux-distillation operation and absorbed in a scrubber containing sodium hydroxide solution. The cyanide in the distillate is then determined in a spectrophotometric analysis.

Water samples analyzed under EPA 335.3 are not externally distilled and are directly analyzed unless otherwise requested.

Spectrophotometric Analysis: On a Technicon auto analyzer, the samples are acidified and passed through a UV digester and then distilled in-line. The cyanide released is converted to cyanogen chloride, CNCl, by reaction with chloramine-T at pH less than 8 without hydrolysis to the cyanate. After the reaction is complete, color is formed on the addition of pyridine-barbituric acid reagent. The absorbance is read at 570 nm. To obtain colors of comparable intensity, it is essential to have the same salt content in both the samples and the standards.

1.4 Sample Handling, Sample Storage, Holding Times

Samples are preserved with 8 pellets of sodium hydroxide per liter of sample (pH > 12) at the time of collection. Samples must be stored at 4°C ± 2°C and must be analyzed within the specified holding time of 14 days from date of collection. The samples can be submitted in either plastic or glass containers. The pH is checked at the time of analysis and the client should be notified if the pH is less than 12.

Samples are to be checked for chlorine interference at the time of collection and the appropriate action should be taken (see section 5.4 on interferences).

1.5 Definition of Terms

This section defines terms and acronyms as they are used in this SOP. Other terms are not defined here since it is assumed that the user of this SOP already understands their more general meaning.

Batch Identifier - A number given to each preparation or analysis group which uniquely identifies that batch. This number is generally the blank ID for preparation batches only preceded by an "A" rather than a "B" for inorganic batches. The preparation batch IDs are discussed in other documentation. The batch identifier for the second set of water samples analyzed on Feb 6, 1998 would be A020698AA1W02.

CCB - Continuing Calibration Blank - This is the same acronym used in the CLP program. This is a blank which is analyzed immediately after the CCV (almost always after every 10 samples and at the end of the analytical run) during the analysis sequence to determine whether the instrument or system has maintained a stable baseline.

CCV - Continuing Calibration Verification. - This is the same acronym used in the CLP program. This is a standard analyzed at some prescribed frequency (almost always after every 10 samples and at the end of the analytical run) during the analysis sequence to determine whether the instrument or system has remained in calibration.

CLP - Contract Laboratory Program - The USEPA program that contracts with laboratories to provide laboratory services. The term has come to mean a much broader set of methods and deliverables. In context of this SOP, CLP means procedures or operations which are detailed in the CLP contract and which are extended to a broader working definition.

Corr Coef, CC, r - Correlation coefficient - A measure of the "goodness of fit" of a set of data to a regression model. The closer the value is to 1, the higher the degree of confidence in the correlation.

DIW - Deionized water - Lab reagent water. This water should be free of virtually all analytes.

ICB - Initial calibration blank - This term is borrowed from CLP. An instrument blank is made up in the same way as calibration standards, without target analytes.

ICV - Initial calibration verification - This term is borrowed from the CLP protocol. It is a standard which is analyzed at the start of each analytical run and compared to the initial calibration to determine whether the instrument calibration is accurate.

MDL - Method detection limit - The lowest concentration a sample which will yield a positive result that is greater zero at a known level of confidence. MDLs are empirically determined by Laucks.

SRM or LCS - Standard Reference Material or Laboratory Control Sample. This is a material of approximately the same matrix as the samples, containing a known and usually certified amount of target analyte and which is prepared and analyzed in the same manner as a typical sample. This sample is used to demonstrate that the analytical system is in control.

QC period - Quality control period - An analysis sequence initiated by the analysis of one or more standards, followed by samples, and terminated with a standard and blank analysis. A QC period can be open-ended chronologically, but calibration verification must be documented using the procedures in this SOP

Sequence - A set of sample extracts and standard solutions injected into an instrument in a chronologically continuous group. See also QC period.

2. Equipment, Reagents, and Standards

2.1 Equipment

Required instrumentation includes the following:

1) Midi reflux distillation system with heating block capable of maintaining 125°C ± 5°C. We use the MIDI-VAP model MC-100 ("CYN-TEN").

2) Technicon Auto-Analyzer with in-line distillation unit and UV digester.

2.2 Reagents

1) 0.25 N NaOH: dissolve 10.0 g NaOH pellets in 1 L DIW. **CAUTION**: this reagent will get hot upon mixing!

2) $MgCl_2$ solution: dissolve 510 g $MgCl_2 \cdot 6H_2O$ and dilute to 1 L with DIW.

3) 1:1 H_2SO_4: carefully and slowly add with mixing 500 mL of conc. H_2SO_4 to 500 mL of DIW. **CAUTION**: this reagent will get **EXTREMELY** hot upon mixing! Mix in a Pyrex or equivalent container! **DO NOT ADD WATER TO ACID!**

4) Phosphate buffer: dissolve 13.6 g monobasic potassium phosphate (KH_2PO_4) and 0.28 g anhydrous dibasic sodium phosphate (Na_2HPO_4) and dilute to 1 L with DIW.

5) Chloramine -T solution: dissolve 1.0 g in 250 mL of DIW. Prepare this reagent fresh daily.

6) Color reagent: place 15 g of barbituric acid in the bottom of a container, and rinse down the sides of the container with DIW. Add 100 mL of DIW and swirl gently. Add 75 mL of pyridine and mix. Add 15 mL of conc. HCl, mix, and cool. Dilute to 1 L with DIW. This reagent should be stored at 4°C. This reagent should be made under a hood.

7) Distillation reagent: to 500 mL of DIW add 250 mL conc. H_3PO_4 and 50 mL 50% H_3PO_2 and then dilute to 1 L with DIW.

8) Sodium Hypochlorite solution: Commercial bleach such as Clorox (5.5%).

2.3 Standards

(a) Stock cyanide solution for standards and spiking, 100 mg/L CN. Place 0.2510 g of KCN and approx. 1.5 g NaOH in a 1.0 L volumetric flask and dissolve. Dilute up to the mark with DIW. Record this standard in a standards logbook. The stock standard is good for one year.

(b) Intermediate cyanide standard solution, 1.00 mg/L CN. Dilute 1.0 mL of stock standard solution (a) to 100 mL with 0.25 N NaOH. Prepare fresh solution at least weekly and record in a standards logbook.

(c) Blank solution for zero standard, blanks and diluting samples and standards: Dilute 200 µL 10 N NaOH to 100 mL with DIW. Prepare fresh solution at least weekly and as needed.

(d) Calibration standards:

Concentration (mg/L)	µLs of (b)	Final volume (mL)
0.200	1000	5.0
0.150	750	5.0
0.100	500.	5.0
0.050	250.	5.0
0.020	100	5.0
0.010	50.	5.0
0	0	5.0

All standards should be diluted up to final volume with (c) Blank solution. Standards are used up in analysis so prepare fresh standards for each analytical run.

3. Safety Precautions

Refer to the Laucks SOP on lab safety practices and the instrument manufacturer's manual for routine instrument precautions.

All standards, samples and sample solutions should be handled as if they are hazardous substances. Some forms of cyanide can be released as a gaseous form upon acidification. Treat all samples as if they definitely contain cyanide.

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

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

Sodium hydroxide and sulfuric phosphoric acid are extreme irritants if they come in contact with skin or eyes. When using these chemicals, the analyst should wear gloves and safety glasses.

The color reagent (containing pyridine) should be prepared under a hood.

4. Calibration and Quality Control

Quality Control criteria are further detailed in Appendix I.

4.1 Method Detection Limit Study

Prior to the analysis of any samples, it is necessary to establish method detection limits. This procedure is fully described in the Laucks SOP on determining detection limits. Briefly, it involves the analysis of 7 replicate samples spiked at a concentration 1-5 times the anticipated method detection limit. A Student's T-test is then applied to these measured values to calculate the MDL. MDLs are determined annually.

4.2 Initial Multi-Point Calibration

The calibration standards listed in section 2.3 (d) should be analyzed in order from lowest concentration to highest. This calibration MUST IMMEDIATELY PRECEDE sample analysis. Initial calibration data is evaluated using the correlation coefficient of a linear regression analysis. The correlation coefficient must be 0.995 or greater. All calibration verifications and sample extract concentrations must be computed using the regression equation. If the criteria are not met, the instrument must be recalibrated.

The highest standard used in the linear regression calculation defines the upper usable working range of the instrument. Any sample concentration value that exceeds the concentration of this high standard must be diluted with blank solution (standard c) and reanalyzed.

4.3 Initial Calibration Verification

Immediately after the calibration curve, analyze an appropriate ICV. The calculated concentration of the ICV must be within ±15% of the true value. If the ICV criteria are not met, no samples can be analyzed. Perform system maintenance and re-calibrate. An LCSW is analyzed for every 20 samples per analytical run and only the first one is also an ICV.

4.4 Initial Calibration Blank

After the analysis of the ICV standard an instrument blank (ICB) is analyzed. The ICB consists of a sample of blank solution, standard (c). The levels of cyanide in the ICB must not exceed the reporting limit. If the initial ICB contains CN levels above the reporting limit, the system is out of control. The source of contamination must be identified and corrected before proceeding with the analysis. The instrument must be recalibrated.

4.5 Continuing Calibration Verification (CCV) and Blank (CCB)

The mid-range calibration standard of 0.100 mg/L is analyzed after every 10 samples (starting after the ICB) as the CCV. Immediately following the CCV, a sample of blank solution (standard (c)) is analyzed as the CCB. In addition, the CCV/CCB must be the last samples analyzed in the run.

Cyanide concentration in the CCV must fall within ± 15% of the true value. The levels of cyanide in the CCB must not exceed the reporting limit. If CCV limits are exceeded, check calculations or perform instrument maintenance. If the CCB limit is exceeded, identify the source of the contamination. No sample results may be reported that are not bracketed by a successful ICV/ICB and/or successful CCV/CCBs, and only ten samples within each successful bracket can be reported.

4.6 Preparation Blanks

For manual distillation preparation, the preparation blank (PB) consists of a 50 mL sample of DIW and is used to verify contamination free reagents and apparatus. A PB is distilled every 20 samples per analytical run. The cyanide concentration of the PB must not exceed the reporting limit. If the reporting limit is exceeded in a manually distilled PB, the lowest reportable concentration of CN in the associated samples is twenty times the PB concentration. Otherwise, all samples associated with the PB with cyanide concentrations less than twenty times the PB concentration and above the reporting limit, must be re-distilled and reanalyzed with a new PB.

For autoanalyzed water samples, a PBW is analyzed for every 20 samples (or with each SDG) analyzed. This does not include distillates from manual distillation which are represented by their distilled PB. This consists of a sample of blank solution (standard (c). If the cyanide concentration is above the reporting limit, then the blank solution is reprepared and reanalyzed as well as any samples diluted with the solution since the last PBW was checked (including the calibration). If the new PBW is in control, then the samples are verified by the new PBW. If contamination persists, then source isolated and the instrument is recalibrated and all samples are verified by the PBW are reanalyzed.

4.7 Matrix Spike

For manual distillation preparation, a sample is chosen at random from the samples to be analyzed (or a sample that is specifically requested by the client if they send excess sample volume). 50 µL of 100 mg/L CN standard solution, standard (a), is added to the 50 mL of sample prior to distillation, for a spike value of 0.100 mg/L in the 50 mL of distillate. For 9012A a 50 mL aliquot is spiked with 20 µL for a spike concentration of 0.040 mg/L The analyst must attempt to avoid selecting samples which are identified by the client as blanks. As the purpose of the matrix spike is to test the system under "typical" conditions, the analyst may also avoid selecting the most difficult sample of the batch for spiking. The minimum frequency for MS analysis is 1 each per 20 samples per matrix. This matrix spike sample is used to evaluate the matrix effect of the sample upon recovery of the analytes.

For autoanalyzed water samples, the same criteria for sample selection and frequency applies. The MS is prepared by measuring out 10 mL of the selected sample, then adding 10 µL of the 100 mg/L CN standard (standard (a)) for a spike value of 0.100 mg/L. If the sample needs dilution, the diluted sample is spiked in the same manner for a spike value of 0.100 (multiplied by the dilution factor) mg/L.

The recovery of spike analytes is calculated as follows:

$$\% \text{ recovery} = \frac{(SS - S)}{SA} * 100$$

where:

SS = concentration in spiked sample
S = native concentration in unspiked sample
SA = spiked added; the amount of spiking material actually added to the sample calculated on the sample basis

If the spike recovery is not at or within the limits stated in QC-DB, an action such as re-analysis of the spike at a higher dilution, or selection of an alternate sample for spiking (if possible) is

taken. An exception to this rule is granted in situations where the blank spike is in control, or the sample concentration exceeds the spike level by a factor of four or more. In these cases, a narration is included with the data.

4.8 Matrix Spike Duplicate

For both manual distillation and autoanalysis; at least one duplicate sample per 20 samples per matrix is required. RPD values are calculated as follows:

$$RPD = \frac{|SR1 - SR2|}{(SR1 + SR2)/2} * 100$$

where:

SR1 = measured concentration in the initial spike analysis
SR2 = measured concentration in the duplicate spike analysis

The recovery and RPD must fall within the control limit stated in QC-DB.

4.9 Blank Spike

To verify the manual distillation procedure, a 50 mL sample of DIW is spiked with 50 µL of the 100 mg/L standard (standard (a)) for a spike value of 0.100 mg/L, and is distilled and analyzed with the other samples. The calculated recovery must be within the limits stated in QC-DB. The minimum frequency for the BS is one per 20 samples per analytical run.

4.10 Sample Duplicate

4.10.1.1 Sample duplicates are required. At least one duplicate sample per 20 samples per matrix is required when matrix spikes are being performed. RPD values are calculated in a manner similar to MS/MSD RPDs:

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

where:

S1 = measured concentration in the initial analysis
S2 = measured concentration in the duplicate analysis

4.10.1.2 The RPD control limits are detailed in the current Control Limits Catalog and in the Quality Control Database (QC_DB) and will change from time to time.

4.10.2 Corrective action

4.10.2.1 If a trend in out of control RPD values is observed, the methods used must be examined to determine the source of variance. Once this source is identified, the method must be changed so that samples can be analyzed with a predictable reproducibility. Generally, if recoveries are in control and no analyte of interest was detected in any of the samples, no immediate action will be taken on that sample set. If integrity of reported sample values is in doubt, re-analysis may be called for. Corrective actions should be discussed with the Quality Control Officer.

5. Operation procedures

5.1 Pre-distillation Sample Screen for Waters

Water samples are screened for sulfide and chlorine interferences prior to distillation as follows:

1) Pour a small amount of pH 4 buffer into a 1oz. plastic cup.

2) Check for the presence of sulfide with lead acetate test paper. Wet the paper with pH 4 buffer and then drop ~10 µL of sample onto it. A darkening of the paper indicates the presence of S^{2-}. If present then see Section 5.3 Interferences before proceeding with the distillation.

3) Check for the presence of chlorine with potassium iodide-starch test paper. Wet the paper with pH 4 buffer and then drop ~10 µL of sample onto it. A bluish discoloration indicates the presence of chlorine. If present then see Section 5.3 Interferences before proceeding with the distillation.

5.2 Pretreatment for Cyanides Amenable to Chlorination

Two sample aliquots are required for chlorine amenable cyanide determination. One is pretreated, the other is not pretreated. Both are prepped and analyzed and the difference between the two is the cyanide amenable to chlorination.

1.) To the sample aliquot designated for pretreatment add bleach solution dropwise until an excess of chlorine is maintained. Test for residual chlorine with KI-starch paper. Paper will turn blue.

2.) Maintain excess for one hour with continued agitation using a Teflon stir bar and a stir plate.

3.) After one hour, add ascorbic acid in small increments (~0.01 g) until KI-starch paper shows no residual chlorine and analyze.

5.3 Manual Distillation

Samples are distilled using the MIDI distillation method. The distillation procedure is as follows:

1) Remove fritted tubes from absorbers, rinse, and set aside.

2) Rinse out absorbers, and invert to drain.

3) Add 50.0 mL of a liquid sample (or 1.0 g of a solid sample) to a sample tube. For solid samples, dilute with DIW to the 50 mL mark on the tube.

4) Set up sample tubes for the appropriate QC (BS, PB, MS/MSD), but do not spike the BS or the "spiked" sample yet.

5) Add 3-4 boiling stones to each tube.

6) Insert reflux impingers into the sample tubes.

7) Fill the absorber tubes with 50.0 mL of 0.25 N NaOH, and insert the fritted tubes.

8) Place sample tubes in the back row of the heating block, and the absorbers into the front row.

9) Insert the cold-finger condensers into the top of the sample tubes.

10) Hook up all vacuum hoses.

11) Turn on water flow, and adjust flow meter to approx. the same number of samples that are being distilled (about 9-10 for 10 samples).

12) Plug in the vacuum pump.

13) Stabilize the flow rate at about 3 bubbles per second and wait for about 5 minutes.

14) Spike the BS and the MS/MSD with the appropriate spiking material.

15) Add 5 mL of 1:1 H_2SO_4 to each tube, and let mix for about 3 minutes.

16) Add 2 mL $MgCl_2$ to each tube, and rinse tubes with DIW.

17) Turn power on, and turn knob to mark.

18) When distillation is complete (10 minutes to heat to reflux, 60 mins of reflux, and 15 minutes of cooling) the heating block will automatically shut itself off. Replace tubes in tube rack, removing the fritted tubes before turning the vacuum pump off.

19) Pour off the liquid from the absorbers into a clean container, label, and store at 4°C if the distillates will not be analyzed immediately.

20) Rinse all glassware with DIW, and store fritted tubes in DIW.

5.4 Interferences

1.) Sulfide interference: Some sulfides convert CN^- to SCN^-. Add cadmium carbonate to sample until lead acetate test paper no longer darkens (treat 25 mL more than is necessary for the distillation). Yellow cadmium sulfide precipitates if the sample contains sulfide. Avoid a large excess of cadmium carbonate and a long contact time in order to minimize a loss by complexation or occlusion of cyanide on the precipitated material. Filter sample through a dry filter paper into a dry container then proceed with the distillation procedure. A narration is included in the final report. Run a standard or BS treated in the same manner with the treated samples.

2.) Chlorine interference: Chlorine decomposes most cyanides. Add ascorbic acid to the 50 mL sample ~0.02 g at a time until KI-starch test paper no longer turns bluish then add an additional 0.02 g of ascorbic acid. Proceed with the distillation procedure. A narration is included in the final report.

3.) NO_3 / NO_2 interference: Nitrates and/or nitrites can form oximes with organic compounds which can decompose under test conditions to form HCN. If high levels are suspected (e.g. fertilizers) then sulfamic acid is added to the sample to remove the interference.

4.) Surfactants. For manual distillation, the presence of surfactants may cause the sample to foam during refluxing. If this occurs, add an antifoam agent such as Baker Antifoam B Silicone Emulsion.

5.) Interferences are eliminated or reduced by the distillation procedure

6. Sample Analysis Using a Technicon Auto Analyzer

6.1 Instrument start-up and operation

The auto-analyzer should be set up for cyanide analysis according to the Laucks SOP on configuring the Technicon Autoanalyzer. Turn on power and allow heating bath to reach 150 degrees C while flushing reagent tubes with DIW. After the temperature is reached, place the reagent tubes into their proper reagents. Turn on chart drive motor and allow reagents to flow through completely. When baseline has settled, set baseline at about 90%T.

6.2 Analysis procedure

1) Prepare the 1.0 ppm working standard and the blank solution

2) From these solutions make the standard curve in 1 oz plastic cups, swirl and transfer to sampler cups, and load onto sampler.

3) Immediately after the calibration standards, load an EPA approved LCSW as the ICV. Immediately after the ICV, load the blank solution for the ICB.

4) Load standards, water samples, and distillates from manual distillation (distillates are analyzed the same as water samples) as in the following example and press the reset button on the autosampler to begin the analysis:

cup#	cup contains
1	STD 1 0 ppb
2	STD 2 10 ppb
3	STD 3 20 ppb
4	STD 4 50 ppb
5	STD 5 100 ppb
6	STD 6 150 ppb
7	STD 7 200 ppb
8	ICV/LCSW1
9	ICB
10	PBW1
11	Sample 1
12	Sample 1MS
13	Sample 1MSD
14	Next Sample
15	. . .
16	CCVx
17	CCBx

After sampling is finished and the last sample peak has been recorded, turn off the chart drive motor, put reagent tubes into wash and turn off the oil bath heater. Once the oil bath has cooled below 100 degrees C, the instrument can be entirely shut down.

6.3 Compound Quantification

The data for each sample is recorded in strip-chart form. On a bench sheet is recorded the information for each sample such as Sample ID, Cup #, and Dilution. Then the value for the Peak Ht and Baseline, as read from the strip-chart (to the nearest 0.25 units), is recorded for each sample. This data is then entered into a standard spreadsheet for the calculation of all other values. (An example of the spreadsheet is included in appendix II.)

- Percent transmittance is calculated from the formula: %T = Peak Ht/(Baseline/100) where the ratio Baseline/100 accounts for baseline drift.

- Absorbance is calculated from %T by the formula: Abs = 2-log(%T)

- The spreadsheet performs a linear regression including the calibration standards 0 mg/L up to 0.200 mg/L with x being the true value of the standards, and y being their corresponding absorbances.

- Concentration of CN^- in the samples is calculated from the linear regression curve using the formula y=mx+b. Substituting the sample absorbance as the y value and solving for x (conc. of CN^- in the same units as the standards were entered) and multiplying by the dilution factor, the formula becomes:

  conc. of CN^- = [(Abs. - y intercept) / slope] * dilution

7. Reports

7.1 Data Qualifying Flags

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

CODE	Definition
U	: The analyte of interest was not detected, to the reporting limit.
*	: QC limits were exceeded.

Appendix I
Laucks Testing Laboratories
Method LTL 9104 QA Requirements and Corrective Actions

QA Element	Method Criterion	Laucks Criterion	Frequency	Corrective Action	Documentation
Initial Calibration	7 standards plus a blank. r ≥0.995	7 stds plus a blank, one std distilled as the BS. r ≥0.995	Beginning of run	Recalibrate if r < 0.995	In raw data.
Initial Calibration Verification / LCSW	Verify calibration every 15 samples with an independently prepared check standard.±15%.	Independent source ±15%.	1 per batch or 5% including one immediately after calibration.	Recalibrate	Enter in QC database under SRM.
Initial Calibration Blank	No criteria.	≤0.010 mg/L	1 per run immediately after ICV.	Recalibrate	In raw data.
Continuing Calibration Verification	Verify calibration with the mid-range standard. Must agree within 15% of true value.	Verify calibration with the mid-range standard. Must agree within 15% of true value.	1 after every 10 samples, and at the end of the run.	Perform instrument maintenance and recalibrate.	In raw data.
Continuing Calibration Blank	No criteria.	≤0.010 mg/L or reporting limit	1 after every 10 samples, and at the end of the run.	Check for source of contamination. Reanalyze associated samples.	In raw data.
Preparation Blank Soil: Water:	No criteria. No criteria.	≤0.50 mg/kg DB ≤0.010 mg/L or reporting limit	1 per batch or 5%.	Check for source of contamination. Reanalyze / reprep w/ associated samples.	Enter in QC database under BLANKS.
Matrix Spike (%Rec) Soil: Water:	Run one spike duplicate every 10 samples. No recovery criteria .	Refer to current control limits catalog	1 per batch or 5%	Spike at higher level or choose another sample and reanalyze / reprep w/ associated samples.	Enter in QC database under MSPKDATA.
Matrix Spike Dup. (RPD) Soil: Water:	See MS.	Refer to current control limits catalog	1 per batch or 5%	Same as MS.	Same as MS.
Blank Spike (%Rec) (Manually distilled soils and waters only.)	Two stds distilled and compared to equal values in standard curve.	Refer to current control limits catalog	1 per batch or 5%	Reprep w/ associated samples.	Enter in QC database under BS_CALC.

Appendix II

Excel spreadsheet used for cyanide calculations.
(example)

Cyanide Analysis	ITEM	DATE	METHOD	ANALYST	CHECKER	
JOB#:	prep.		Man. Distill	PL		
MATRIX: water	analysis	1/25/96	EPA 335.3	PL		

STANDARD CURVE LCSW: WP586 (tv=0.500 ppm)

standard stock:
TE4-67-17

T.V. (mg/L CN)	Peak Ht.	Baseline	%T	Absorbance
0.000	94.00	94.50	99.47	0.002
0.010	88.50	94.50	93.65	0.028
0.020	80.50	94.50	83.44	0.055
0.050	67.50	94.50	71.43	0.146
0.100	47.50	94.50	50.26	0.299
0.150	30.50	94.50	36.24	0.512
0.200	17.75	94.50	18.78	0.726
--x value--				--y value--

r = 0.9999 m = 2.9043 y(0) = 0.0022 x(0) = -0.0008

ms/msd=0.100ppm

Sample I.D.	Dilution	Peak Ht.	Baseline	%T	Absorbance	CN (mg/L)	
ICV / LCSW1	5	54.25	95.00	57.11	0.243	0.4151	83% Rec.
ICB	1	94.25	95.00	99.21	0.003	0.0004	ND
MDL tv=0.005	1	91.25	95.00	96.05	0.017	0.0053	105% Rec.
PBW1	1	94.50	95.00	99.47	0.002	0.0000	ND
5555-1	1	94.50	95.00	99.47	0.002	0.0000	ND
5555-1ms	1	48.00	95.00	50.53	0.296	0.1013	101% Rec.
5555-1msd	1	48.25	95.00	50.79	0.294	0.1005	101% Rec.
5555-2	1	94.25	95.00	99.21	0.003	0.0004	ND
5555-3	1	92.00	95.00	96.84	0.014	0.0040	ND
5555-4	1	89.00	95.00	93.68	0.028	0.0090	0.009
5555-5	1	88.50	95.00	93.16	0.031	0.0098	0.010
5555-6	1	65.75	95.00	69.21	0.160	0.0543	0.054
CCV1 tv=0.100	1	45.50	95.00	47.89	0.320	0.1093	109% Rec.
CCB1	1	94.00	95.00	98.95	0.005	0.0008	ND

Appendix III

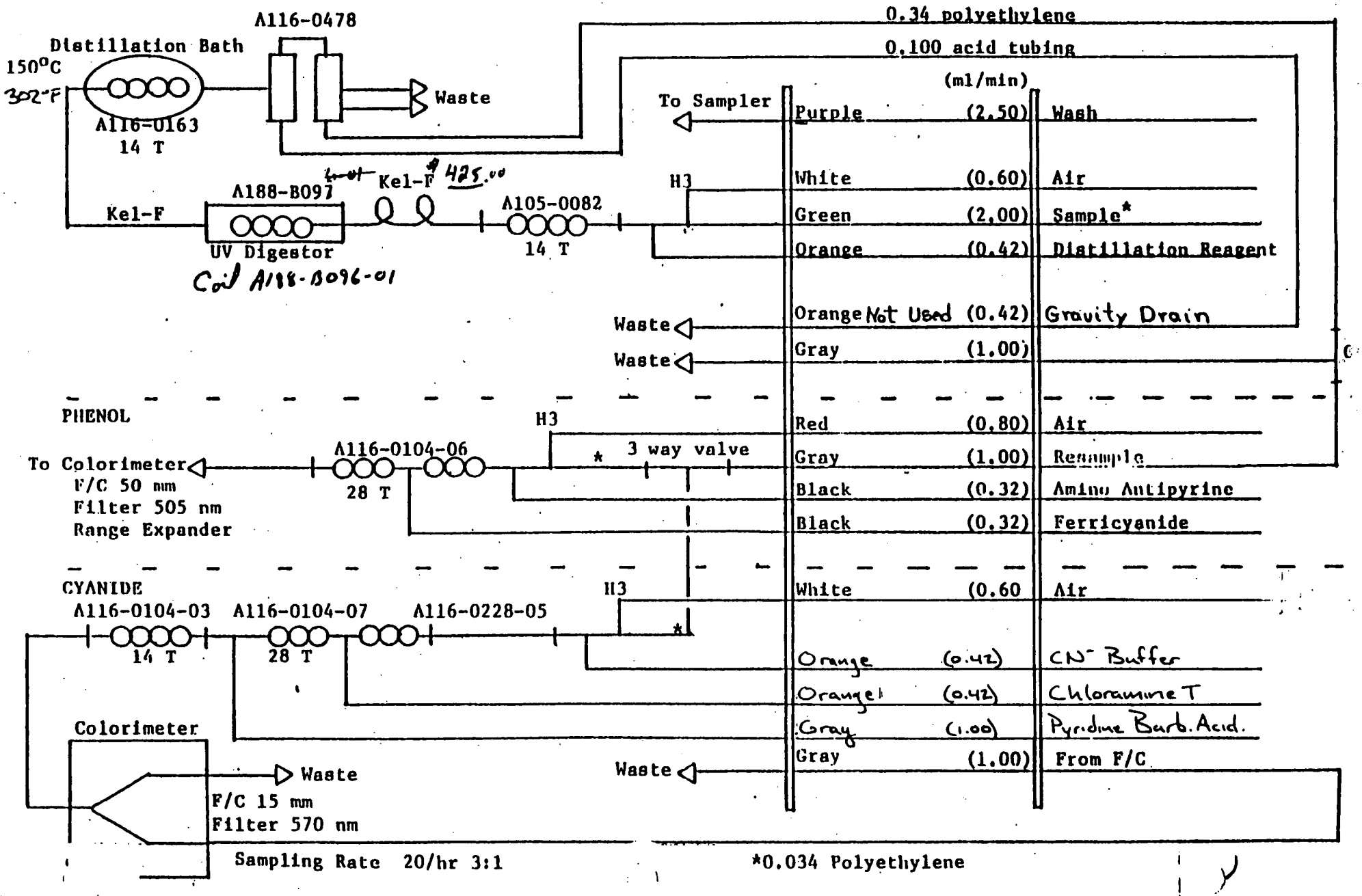
Autoanalyzer Flow Diagram

[illegible] currently in use.
Jan 29, 1991 MS

CYAN[illegible]PHENOL
Comb[illegible]d Manifold
AAI

ALPKEM Corporation
1981
RRP

DISTILLATION



LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #: LTL-9113

Title: **pH in Soil and Waste Samples Using SW846 Method 9045C**

Revision history:

Number	Date
1.0	07/13/94
2.0	01/16/97

UNCONTROLLED

Written by: Bill Lundberg Date: 1/16/97
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Approved by: Karen J Kotz Date: 1/16/97
Karen Kotz, Lab Director

Table of Contents

1. INTRODUCTION AND SCOPE 3

1.1 METHOD DESCRIPTION 3
1.2 INTERFERENCES: 3
1.3 SAMPLE COLLECTION, SAMPLE STORAGE, HOLDING TIMES 3
1.4 DEFINITION OF TERMS 4

2. EQUIPMENT LIST, REAGENTS AND STANDARDS 4

2.1 APPARATUS 4
2.2 STANDARDS 4
2.3 REAGENTS 4

3. SAFETY PRECAUTIONS AND WASTE DISPOSAL 4

3.1 SAFETY PRECAUTIONS 4
3.2 WASTE DISPOSAL 5

4. CALIBRATION AND QUALITY CONTROL 5

4.1 CALIBRATION 5
4.2 SAMPLE DUPLICATE 5

5. OPERATION PROCEDURES 6

5.1 SAMPLE PREPARATION AND pH MEASUREMENT OF SOILS 6
5.2 SAMPLE PREPARATION AND pH MEASUREMENT OF WASTE MATERIAL: 6

6. REPORTS 7

6.1 DATA PACKET ORGANIZATION 7
6.2 QUALITY CONTROL REPORTS 7

APPENDIX I 8

QC SUMMARY TABLE 8

APPENDIX II 10

FLOW CHART 10

1. Introduction and Scope

1.1 Method Description

1.1.1 This method is an electrometric procedure for the determination of pH in soils and waste samples.

1.1.2 The sample is mixed with deionized water (DIW). The pH of the solution is then measured with a pH meter.

1.1.3 This method is restricted to use by, or under the supervision of analysts experienced in the technique described. Each analyst performing this method must have demonstrated the ability to perform the described analysis.

1.2 Interferences:

1.2.1 Samples with very low or very high pH may give incorrect readings on the meter. For samples with a true pH of >10, which is not uncommon for some ash and other waste materials, the measured pH may be incorrectly low. This error can be minimized by using a low-sodium-error electrode. Strong acid solutions, with a true value of <1, an uncommon condition, may give incorrectly high measurements.

1.2.2 Temperature fluctuations will cause measurement errors. Most pH electrodes are calibrated to 25°C. Whereas Laucks does use a temperature correction probe with the pH meter, significant differences in pH can affect the solubility and apparent pH characteristics of the solution itself. Therefore, measurements should be made near the appropriate temperature by allowing the sample and diluent (DIW) to warm to room temperature prior to analysis.

1.2.3 Errors can occur when the pH electrode becomes coated. If this occurs, the electrode must be thoroughly clean and re-buffered prior to measuring the pH of subsequent samples.

1.3 Sample Collection, Sample Storage, Holding Times

1.3.1 There is no official holding time for the analysis of soil pH. The holding time for aqueous samples is to analyze immediately. Soil should not demonstrate significant pH fluctuations if refrigerated and analyzed within a reasonable time but Laucks has chosen to analyze soils samples as soon as is reasonable after collection, not to exceed 7 days from collection.

1.4 Definition of Terms

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

1.4.1.1 **DIW** - Deionized water - Lab reagent water. This water should be free of virtually all analytes.

1.4.1.2 **Sequence** - A set of sample extracts and standard solutions presented to an instrument in a chronologically continuous group.

2. Equipment List, Reagents and Standards

2.1 Apparatus

pH meter with means for temperature correction (temperature correction probe)
Glass electrode
50 ml. beaker
Analytical balance capable of weighing to 0.1 g.

2.2 Standards

pH 4 buffer
pH 7 buffer
pH 10 buffer

2.3 Reagents

Deionized water (DIW)

3. Safety precautions and Waste Disposal

3.1 Safety Precautions

3.1.1 All standards, samples and sample solutions should be handled as if they are hazardous substances.

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

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

3.2 Waste Disposal

3.2.1 There should be no significant waste associated with this procedure which cannot be disposed of in the laboratory sink and/or garbage.

4. Calibration and Quality Control

4.1 Calibration

4.1.1 The pH meter must be calibrated with at least two pH buffers. One is the pH 7 buffer and the other depends upon the samples. If high pH is expected, the second buffer should be at pH 10 or higher. If low pH is expected, the second buffer should be the pH 4 buffer. High pH samples should not be analyzed from a low pH calibration and visa versa.

4.2 Sample Duplicate

4.2.1 Criteria

4.2.1.1 At least one duplicate sample per 10 samples per matrix is required. RPD values are not currently calculated for this analysis. The control limit is considered to be 0.5 pH units.

4.2.2 Corrective action

4.2.2.1 If an out-of-control duplicate pair is observed, the methods used must be examined to determine the source of variance. Once this source is identified, the method must be changed so that samples can be analyzed with a predictable reproducibility. If integrity of reported sample values is in doubt, re-analysis may be called for. Corrective actions should be discussed with the Quality Control Officer.

5. Operation procedures

5.1 Sample preparation and pH measurement of soils

5.1.1 To 20 grams of soil in a 50 ml beaker, add 20 mls of DIW and stir the suspension for 5 minutes. This may also be done with a magnetic stirring bar.

5.1.2 Allow the suspension to stand for about 1 additional hour with no agitation in order to allow most of the suspended particulates to settle out.

5.1.3 Immerse the electrode (including the temperature correction probe) just far enough into the solution to establish good electrical contact. It should not come into contact with the soil itself.

5.1.4 The electrodes should be thoroughly rinsed between samples.

5.1.5 Report the results as "soil pH measured in water @ ____ °C."

5.2 Sample preparation and pH measurement of waste material:

5.2.1 To 20 grams of waste material in a 50 ml beaker, add 20 mls of DIW and stir the suspension for 5 minutes. This may also be done with a magnetic stirring bar.

5.2.2 Allow the suspension to stand for about 15 additional minutes with no agitation in order to allow most of the suspended particulates to settle out.

> Note: If the waste is hygroscopic and absorbs all the DIW, begin the test again using 20 g of waste and 40 ml of DIW.
>
> Note: If the supernatant is multiphasic, decant the oily phase and measure the pH of the aqueous phase.

5.2.3 Immerse the electrode (including the temperature correction probe) just far enough into the solution to establish good electrical contact. It should not come into contact with the waste material itself.

5.2.4 The electrodes should be thoroughly rinsed between samples.

5.2.5 Report the results as "waste pH measured in water @ _____ °C.

6. Reports

6.1 Data Packet Organization

6.1.1 The data package for this analysis consists of the data sheet and a quality control database (QC_DB) report form.

6.2 Quality Control Reports

6.2.1 All results for quality control tests are entered into the lab data base using the QC_DB program. The report form must be filled out in order to indicate that the duplicate QC had been performed and met the criteria specified in this document.

Appendix I

QC Summary Table

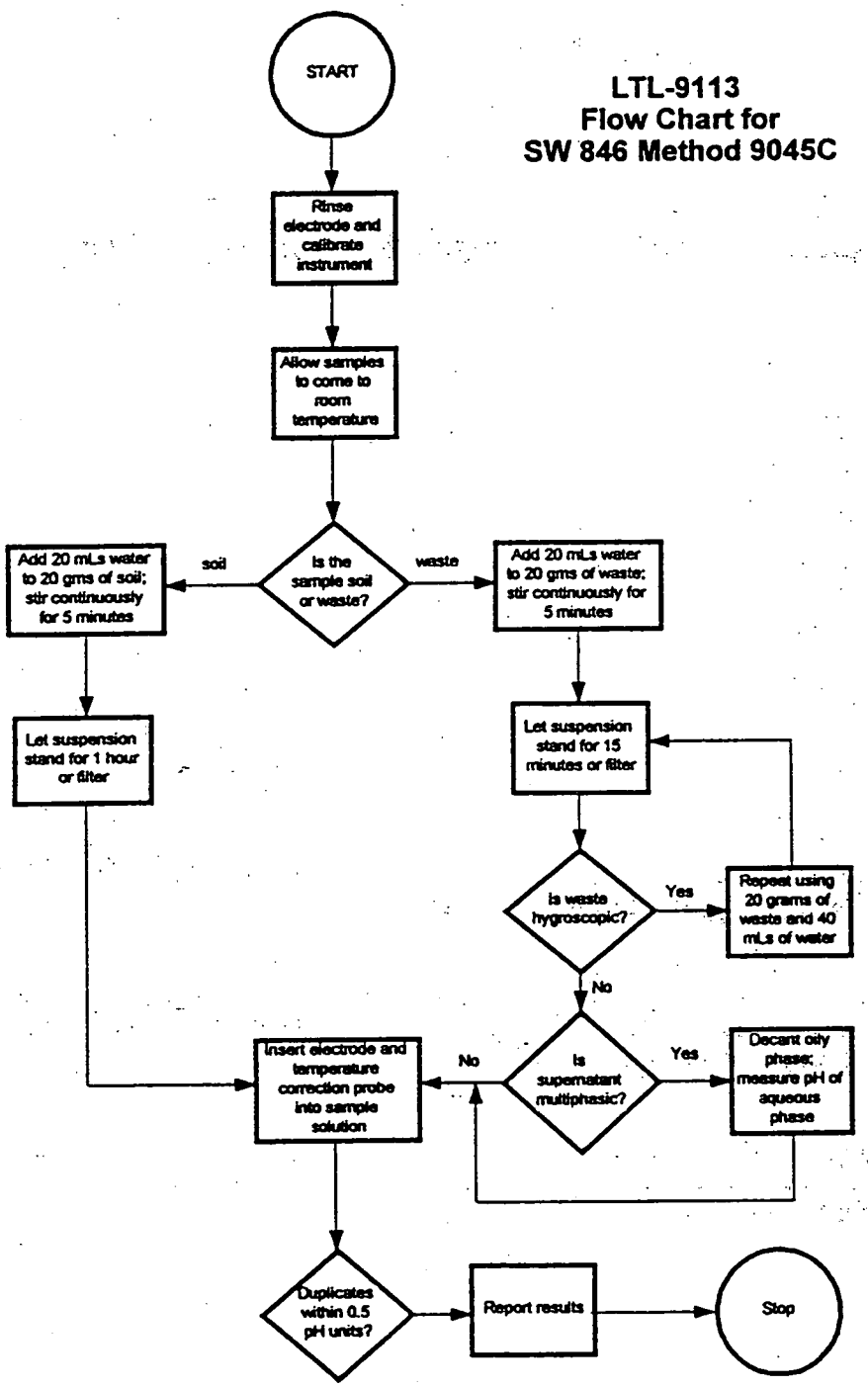
Laucks Testing Laboratories

Method SW 9045C QA Requirements and Corrective Actions

QA Element	Method Criterion	Laucks Criterion	Frequency	Corrective Action	Documentation
Initial Calibration	Buffer pH meter to bracket sample values	Buffer pH meter between pH 7 & 4 or 10 before analyses (to bracket sample values)	Once before analyzing any samples or QC	None	None
Duplicate % Difference	N/A	within 0.5 pH units of one another	10% or once every 10 samples.	Correct problem Consult QC Officer	QC_DB report form with appropriate commentary

Appendix II

Flow Chart


LTL-9113
Flow Chart for
SW 846 Method 9045C
START
Rinse electrode and calibrate instrument
Allow samples to come to room temperature
Is the sample soil or waste?
soil
waste
Add 20 mLs water to 20 gms of soil; stir continuously for 5 minutes
Add 20 mLs water to 20 gms of waste; stir continuously for 5 minutes
Let suspension stand for 1 hour or filter
Let suspension stand for 15 minutes or filter
Is waste hygroscopic?
Yes
Repeat using 20 grams of waste and 40 mLs of water
No
Is supernatant multiphasic?
No
Yes
Decant oily phase; measure pH of aqueous phase
Insert electrode and temperature correction probe into sample solution
Duplicates within 0.5 pH units?
Report results
Stop