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NSWC CRANE
5090.3a

10-1-1989

Corrective Action Work Plan
RCRA Facility Investigation Phase III
Release Characterization, Ground Water
NWSC Crane Ammunition Burning Ground, SWMU #03/10

1. Task A: RFI Phase III Work Plan.

a. RFI Phase III Project Management Plan

The Waterways Experiment Station (USAEWES) will perform a Modified RCRA Facility Investigation (RFI) Phase III Release Characterization for Ground Water at SWMU # 03/10 for the Naval Weapons Support Center (NWSC) Crane, Indiana at the Ammunition Burning Ground Solid Waste Management Unit (SWMU), also known as Corrective Action Plan (CAP) Site Number 03/10 (Figure 1). This document outlines the objectives of the RFI, discusses the technical approach, quality assurance/quality control measures, and personnel to be used in the RFI, and provides a schedule of work to be performed.

Objective. The objective of the Modified Phase III work is to determine the rate and extent of migration of hazardous waste or hazardous constituents in the ground water. The hydrogeology of the ABG was established in the previously submitted report, Geology and Hydrogeology of the Ammunition Burning Grounds Crane Naval Weapons Support Center, Richard W. Hunt, USAE Waterways Experiment Station, Vicksburg, MS, October 1988. A dye tracer test was performed in the ABG study area in January of 1990 to determine flow paths and rate of migration of ground water. A draft technical report was prepared and submitted to EPA. Phase III work will further define ground-water flow patterns, determine rate of flow and ground-water quality, and establish guidelines for long-term monitoring.

Technical Approach. Ground water monitoring wells have been installed, exploratory borings emplaced, and other subsurface investigations conducted to characterize ground water flow and the extent of contamination. A second dye trace was conducted in August of 1990 as proposed earlier. Procedural details are presented in section 1.b. of this document. If pumping tests are needed to determine aquifer parameters, pumping wells and observation wells will be installed. Other options to be considered for determining aquifer parameters include falling head (slug) tests or dye tracer tests. At such time pumping or other tests are deemed necessary, the work plan will be revised and submitted to the EPA for approval.

Quantitative chemical analysis of ground water from existing and proposed monitoring wells and springs are being conducted for specific compounds. Statistical analysis and evaluation of ground-water data from ABG monitoring wells is being conducted for NWSC Crane under contract. The contractor will prepare a report summarizing the extent of suggested ground-water contamination and the wells affected. Summaries will be prepared for data from the last nine sampling quarters and from data gathered in 1981 and 1987. Geologic and ground water profiles and maps showing the orientation of the subsurface geologic units and piezometric surface contour maps were presented in the previously submitted hydrogeology report.

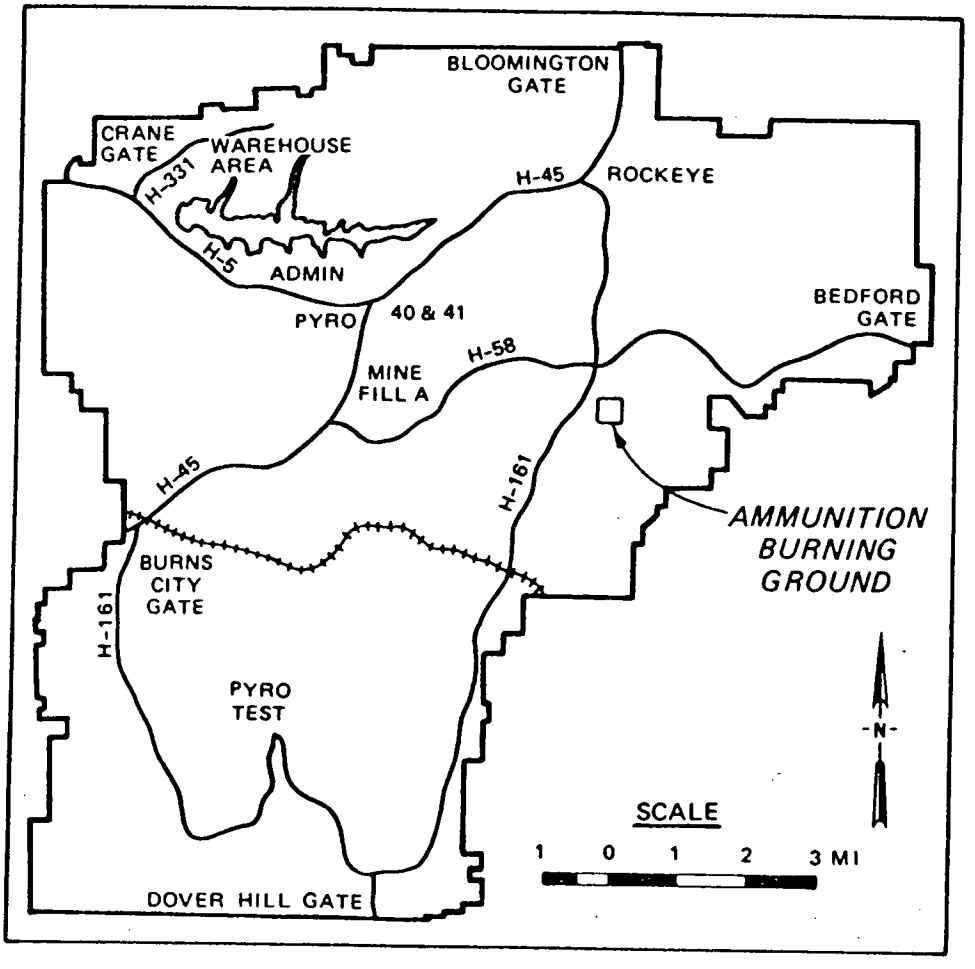

Figure 1. NWSC Crane and location of the Ammunition Burning Ground (ABG)

Personnel. Modified Phase III ground-water work will be conducted by WES and NorthDiv personnel and NWSC contractors. All personnel working at the SWMU will be trained in personal protection and safety and will have had a comprehensive physical examination within the 12 months preceding their involvement in the field work (see Health and Safety Plan, Part f.)

Schedule. The Regional Administrator has provided comments to the Permittee as to the corrections or modifications needed for the RFI Phase III Work Plan. The Work Plan will be initiated within 45 days after it it has been approved. The results of Phase III ground water work will determine whether Corrective Measures are necessary. The schedule for accomplishing the Modified RFI Phase III Release Characterization for Ground Water is presented as a time-line (Figure 2).

b. RFI Phase III Sampling and Analysis Plan

(1) Sampling Objectives. Sampling of the water in monitoring wells has been designed to determine the presence or absence of specified compounds in the ground water of aquifers underlying and adjacent to the ABG area. Monitoring wells were placed and screened to sample water in discrete ground water zones that are isolated from other ground water zones. Screens were placed in the uppermost zone encountered and in subsequent zones (using cluster wells) down to the lowermost aquifer determined to be susceptible to water migrating downward from the surface. The lowermost aquifer at the ABG is the Beaver Bend limestone. the uppermost is the Golconda-Haney limestone. The Big Clifty-Beech Creek formation, positioned between the upper and lower aquifers, is the aquifer of greatest concern for the ABG investigation.

(a). **Dye trace of Beech Creek limestone.** The Beech Creek limestone carries most of the flow in the Big Clifty/Beech Creek aquifer. A dye trace of the Beech Creek aquifer, confined to NWSC Crane boundaries, was performed in January, 1990 by WES and NorthDiv and a Draft Technical Report on its findings submitted to EPA on 29 Jun, 1990. The dye trace identified specific on-post springs as being most likely to serve as outlets to migratory ground water and any associated contaminants leaving the ABG. A proposal for a second dye trace to encompass off-post sampling points in addition to the on-post points was also submitted to EPA on 29 Jun. The second dye trace injection took place 7 August, 1990. Sampling is continuing for the second dye trace.

The area of the proposed second dye trace was expanded to include off-post monitoring points and additional on-post wells. Figure 3 is a topographic map showing the expanded study area (Williams and Indian Springs USGS 7-1/2 minute quadrangles). Springs A through F were monitored in the first dye-trace. The second dye trace included field-mapped springs off-post north and west of the heavy dashed line that roughly follows the axis of the floodplain of Big sulphur Creek. Off-post springs are labeled SP1-90 through SP15-90. Springs SP13-90 through SP15-90 are in the Beaver Bend limestone and were to be monitored to confirm that no karstic hydraulic connection exests between the Beech Creek and Beaver Bend aquifers. Springs SP13-90 through SP15-90 could not be located during the second dye trace because of heavy vegetation and lack of visible signs of flow. The were presumed dry and were not monitored. Springs SP1-90 through SP12-90 issue from the Beech Creek. Dye tracing in those springs will better define the extent of potential migration of contaminants off-post and help establish guidelines for long term water quality sampling. Monitoring stations

SUBJECT: MODIFIED RFI PHASE III RELEASE CHARATERIZATION FOR GROUND WATER: SCHEDULE
NAVAL WEAPONS SUPPORT CENTER CRANE, INDIANA
SWMU: AMMUNITION BURNING GROUND AREA

COMPUTED BY: DATE:

CHECKED BY: DATE:

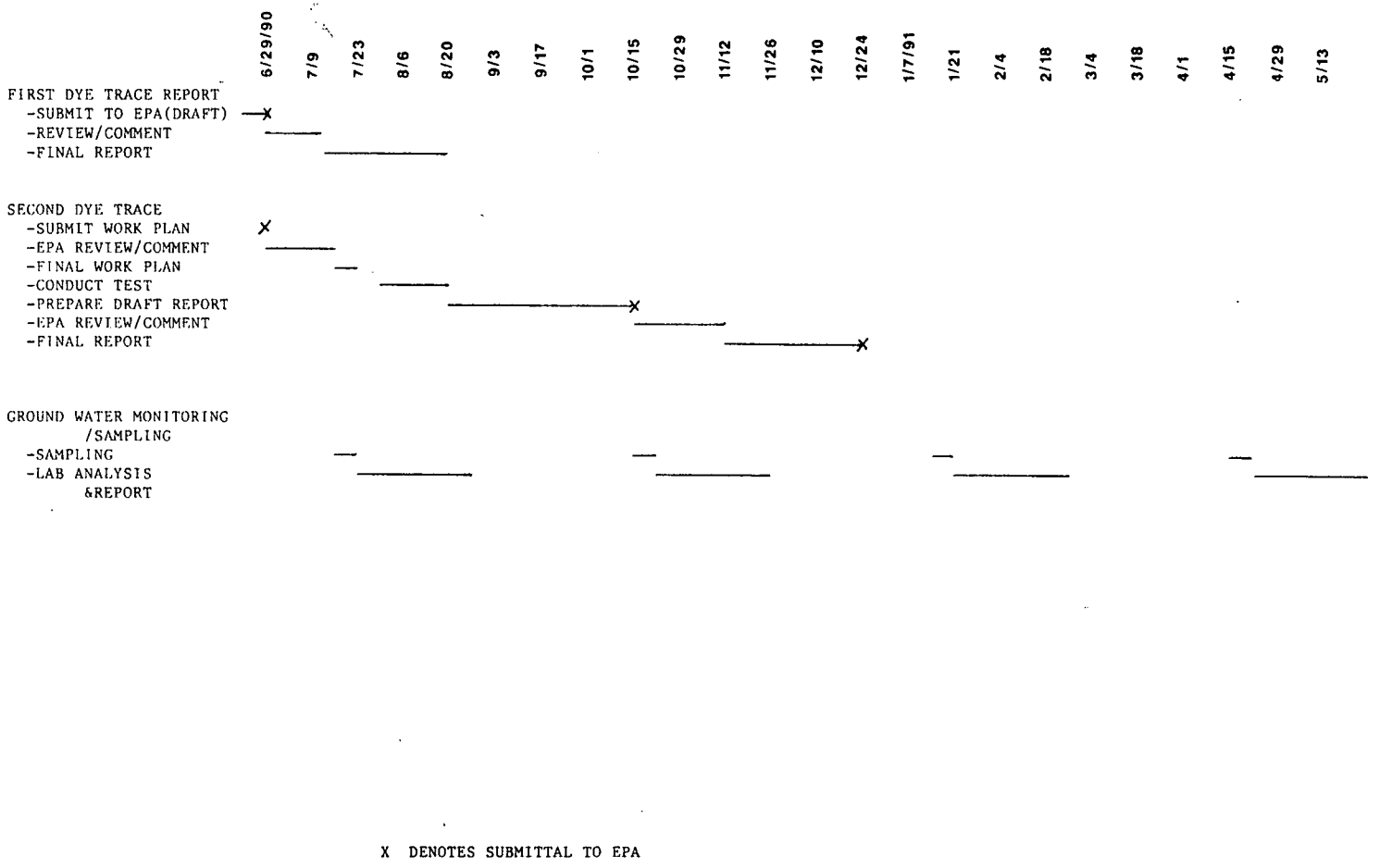

Figure 2. Schedule for Modified RFI Phase III Ground Water, ABG

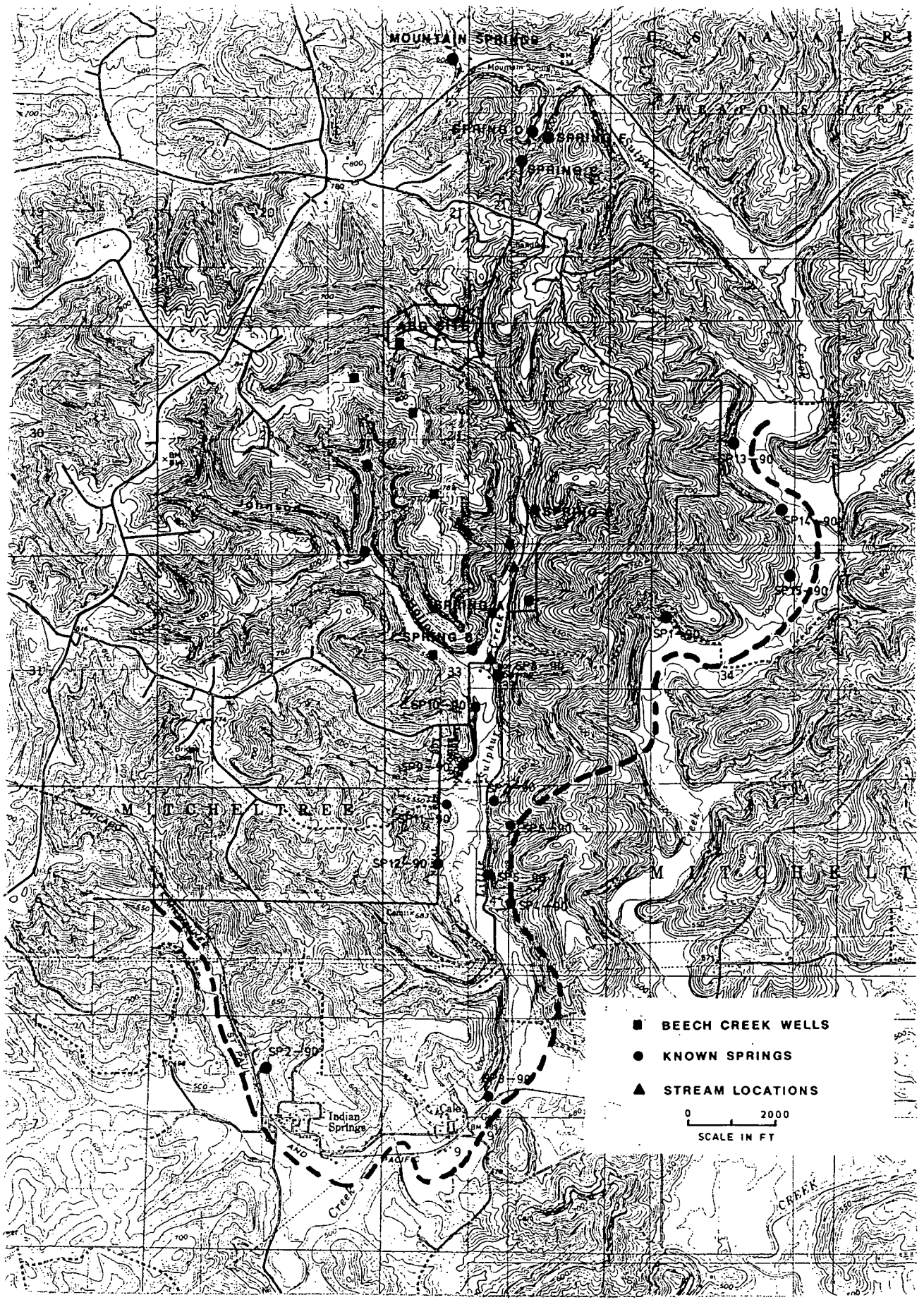

Figure 3. Map of expanded study area for second dye trace, ABG

at several locations within Little Sulphur Creek will determine the extent of surface runoff migration and identify ground-water reentry points to the stream system from springs. Dye injection was in well 03C3P2 at the ABG (for location see Figure 5 of this work plan.

(b) **Ground-water quality sampling.** Eight ground-water monitoring wells were constructed at the ABG and first sampled in the last quarter of 1981. Additional wells have been added at later dates. Constituents originally sampled for were those listed in 40 CFR 265.92. Sampling for explosives began in March 1983, which was the start of the second year of sampling. After sampling results indicated possible ground-water contamination, a more comprehensive ground-water testing program was instituted and additional constituents were tested for. The 40 CFR 261 Appendix VIII parameters were sampled for in 1986. Based on those results, sampling of the wells began in 1987 for 1,1,1-trichloroethane, trichloroethylene, D-N-butyl phthalate, and bis(2-ethylhexyl) phthalate, Appendix III drinking water standards, explosives RDX, HMX and TNT, and volatile organics. Sampling has continued on a quarterly basis to the present. Wells 03C34 through 03C38 were installed in 1989. The first sampling round on the 1989 wells sampled for metals in addition to the Appendix VIII, Appendix III and volatile organics. The seven Beech Creek limestone springs mapped within NWSC boundaries are also being sampled and tested. Results of the statistical analysis of previous and ongoing ground-water quality sampling will guide long term monitoring in and around the ABG regarding selection of wells and parameters to be monitored.

(2) Sampling equipment and containers. Ground-water sampling equipment and containers are discussed in the QA/QC section of this work plan. Sampling equipment used in the dye trace consists of activated coconut charcoal contained in screen wire packets for adsorbing dye which has been injected into the ground water, glass jars used for eluting the dye out of the charcoal, and canning jars used to store duplicates of collected charcoal samples. Water-soluble dyes prepared in the laboratory prior to injection are stored for a short time (one to several hours) in plastic or glass containers. Charcoal packets are stored in a cooler after collection to be transported to the field laboratory for analysis.

(3) Analytical parameters and test methods. Analytical methods in general are described in the QA/QC section. Sampling will continue at all RCRA wells (wells 03-C01 through 03-C38) for explosives compounds, Appendix III ground water quality parameters, the four Appendix VIII compounds listed in section b.(1)(b) above, and volatile organics. Table 1 lists the compounds currently being analyzed for at the ABG. Results of the statistical analysis of sampling results currently being conducted will determine selection of parameters and wells for subsequent sampling and analysis at the ABG, contingent on EPA approval.

(4) Sample Types. Table 2 is a list of the wells currently being sampled quarterly (see paragraph (5) below for well locations). A statistical analysis and evaluation of the ground-water sampling and analysis data is being performed under contract for NWSC Crane. The statistical analysis will first establish background concentrations for specified parameters and determine if there were any increases in concentration levels at any wells over time and if maximum concentration levels were exceeded at any time. Results of the statistical analyses will determine the selection of wells and parameters to be monitored in succeeding sampling rounds, as stated above in paragraph (3).

TABLE 1

SAMPLE ANALYSIS RESULTS

NAVWPNSUPPCEN, BLDG 2516
CRANE, INDIANA 47522
ATTN: JO BELCHER

ANACON# 7120
DATE 12-29-89

SAMPLE DESCRIPTION: 3C1P2, 10-21-89

ALL RESULTS ARE IN MG/L

TEST RESULTS:

pH		7.4		
SP.COND,UM/CM		667		
TOC				
TOX				
FE	<0.05	AS	ENDRIN	
MN		BA	LINDANE	
NA		CD	METHOXYCHLOR	
CA	84	CR	TOXAPHENE	
MG	35	PB	2,4-D	
K	2.2	HG	2,4,5-TP	
SULFATE		SE	RADIUM,pCi/L	
CHLORIDE		AG	GROSS a,pCi/L	
PHENOL		FLUORIDE	GROSS b,pCi/L	
BICARBONATE	381	NITRATE-N	COLIFORM	
AMMONIA-N	<0.01			
TNT	<0.01	1,1,1-TRICHLOROETHANE		<0.001
RDX	<0.01	TRICHLOROETHYLENE		0.070
HMX	<0.02	D-N-BUTYL PHTHALATE		<0.01
PCB		BIS(2-ETHYLHEXYL)PHTHALATE		<0.01

SUBMITTED BY _____

TABLE 1 (concluded)

ANACON, INC.
9001 AIRPORT BLVD, #605
HOUSTON, TEXAS 77061

VOLATILES ORGANIC ANALYSIS DATA SHEET

CUSTOMER: NWSC, CRANE
SAMPLE ID: 3C1P2
ANACON NO: 7120

SAMPLE DATE:
REPORT DATE: 11-21-89
FILE REFERENCE NO: 3C1P2

	CAS #	COMPOUND	AMOUNT (UG/L)	
1.	74-87-3	CHLOROMETHANE	3	U
2.	74-83-9	BROMOMETHANE	3	U
3.	75-01-4	VINYL CHLORIDE	3	U
4.	75-00-3	CHLOROETHANE	0.5	U
5.	75-09-2	METHYLENE CHLORIDE	0.5	U
6.	107-02-8	ACROLEIN	30	U
7.	67-64-1	ACETON	25	U
8.	107-37-1	ACRYLONITRILE	15	U
9.	75-15-0	CARBON DISULFIDE	1.0	U
10.	75-35-4	1,1-DICHLOROETHENE	0.5	U
11.	75-35-3	1,1-DICHLOROETHANE	0.5	U
12.	156-60-5	TRANS-1,2-DICHLOROETHENE	0.5	U
13.	67-66-3	CHLOROFORM	0.5	U
14.	78-93-3	2-BUTANONE	15	U
15.	107-06-2	1,2-DICHLOROETHANE	0.5	U
16.	71-55-6	1,1,1-TRICHLOROETHANE	0.5	U
17.	56-23-5	CARBON TETRACHLORIDE	0.5	U
18.	108-05-4	VINYL ACETATE	5	U
19.	75-27-4	BROMODICHLOROMETHANE	0.5	U
20.	78-87-5	1,2,-DICHLOROPROPANE	0.5	U
21.	10061-02-6	TRANS-1,3-DICHLOROPROPENE	0.5	U
22.	79-01-6	TRICHLOROETHENE	0.5	U
23.	71-43-2	BENZENE	0.5	U
24.	124-48-1	DIBROMOCHLOROMETHANE	0.5	U
25.	79-00-5	1,1,2-TRICHLOROETHANE	0.5	U
26.	10061-01-5	CIS-1,3-DICHLOROPROPENE	0.5	U
27.	110-75-8	2-CHLOROETHYL VINYL ETHER	0.5	U
28.	75-25-2	BROMOFORM	0.5	U
29.	108-10-1	4-METHYL-2-PENTANONE	1.0	U
30.	519-78-6	2-HEXANONE	15	U
31.	127-18-4	TETRACHLOROETHENE	0.5	U
32.	79-34-5	1,1,2,2-TETRACHLOROETHANE	0.5	U
33.	108-88-3	TOLUENE	0.5	U
34.	108-90-7	CHLOROBENZENE	0.5	U
35.	100-41-4	ETHYLBENZENE	0.5	U
36.	100-42-5	STYRENE	0.5	U
37.	108-38-3	XYLENE (total)	0.5	U

CODES: U - COMPOUND WAS ANALYZED FOR BUT NOT DETECTED. THE VALUE REPORTED IS THE METHOD DETECTION LIMIT FOR REAGENT WATER.
J - ESTIMATED VALUE.
SLC - SUSPECTED LABORATORY CONTAMINANT.
SFC - SUSPECTED FIELD CONTAMINANT.

TABLE 2

ABG WELLS CURRENTLY BEING SAMPLED

3C1	3C26
3C1P2	3C27
3C1P3	3C28
3C2	3C29
3C2P2	3C30
3C3	3C31
3C3P2	3C32
3C4	3-34C
3C5	3-37C
3C6	3CB1
3C7	3CB2
3C8A	3CB3
3C8AP2	3CB4
3C8AP3	3CB5
3C9	3CB6
3C9P2	3CB7
3C10	3CB8
3C11	3CB9
3C12	3CB10
3C13	3-1
3C14	3-3
3C15	3-4
3C15P3	3-6
3C16	3-7
3C17	3-9
3C18	3-28
3C19	3-31
3C19P3	3-32
3C20	3-33
3C21	3-34
3C22	3-35
3C23	3-37
3C24	3-38
3C25	3-39

(5) Sample locations, depths and frequency. Approximately 98 monitoring wells have been emplaced and are available for sampling at the ABG. Figure 4 is a topographic map showing the locations of wells outside the ABG site. Figure 5 is a larger scale map of the ABG site well locations. Figure 6 is a large scale map of wells located in the INSET shown on Figure 4. Monitoring well data are presented in Table 3 (5 pages). Total depth of the well boring and depth of screen placement are included in Table 3.

(6) Sampling schedule. The ground water sampling will continue on a quarterly basis until results of the statistical analysis of previous results is complete. At that time negotiations will commence with EPA to revise the schedule or selection of wells and parameters, as discussed above.

(7) Sampling procedures. Monitoring well sampling procedures will be described in detail in Part I of the QA/QC document. Soil and rock sampling were conducted from a truck-mounted rotary drilling rig. Rock cores were collected continuously using a diamond-bit HQ wireline core barrel (HQ wireline retrieves a 2-1/2 in. diameter core from a 3-3/4 in. diameter borehole).

Drilling water is currently taken from the water treatment plant at the Crane installation, which draws its potable water from Lake Greenwood, located within the NWSC boundaries. Prior to the start of drilling, the drill rig and appurtenances were steam-cleaned. Drill cuttings were removed from the boring by circulating clean water from a steel mud pan sealed around the boring top to prevent contamination of the subsurface from surface runoff. The mud pan was cleaned and refilled periodically as conditions warranted. In borings penetrating more than one aquifer the upper zones were sealed before the boring was advanced. PVC casing was grouted 3 to 5 ft. into the confining stratum at the base of the aquifer and allowed to set overnight before advancing the boring. The boring was then advanced through the upper casing and grouted. Smaller casing was set for drilling the next aquifer.

(8) Rationale for sampling location and choice of analytes. The locations of sampling stations for water quality and choice of analytes have been negotiated with USEPA by NWSC Crane previously.

(9) Environmental conditions at the time of sampling. The presence of volatile organic compounds in the air at the well locations will be monitored using a photoionization organic vapor detector and a combustible gas detector prior to removal of the well cap for sampling.

(10) Chain-of-custody forms and procedures. Chain-of-custody is described in the QA/QC document, Section d.

(11) Decontamination Procedures. Decon procedures for water sampling are described in detail in Part I of the accompanying QA/QC document. Decontamination of the drilling and coring equipment consisted of steam cleaning the drilling rig and appurtenances before start of a new boring. In addition, the down-hole equipment such as drill rods, rock bits, and core barrels were steam cleaned after sealing of a ground-water zone and before advancing through the aquiclude to the next aquifer.

(12) Documentation. Ground-water sampling logs will be kept as described in

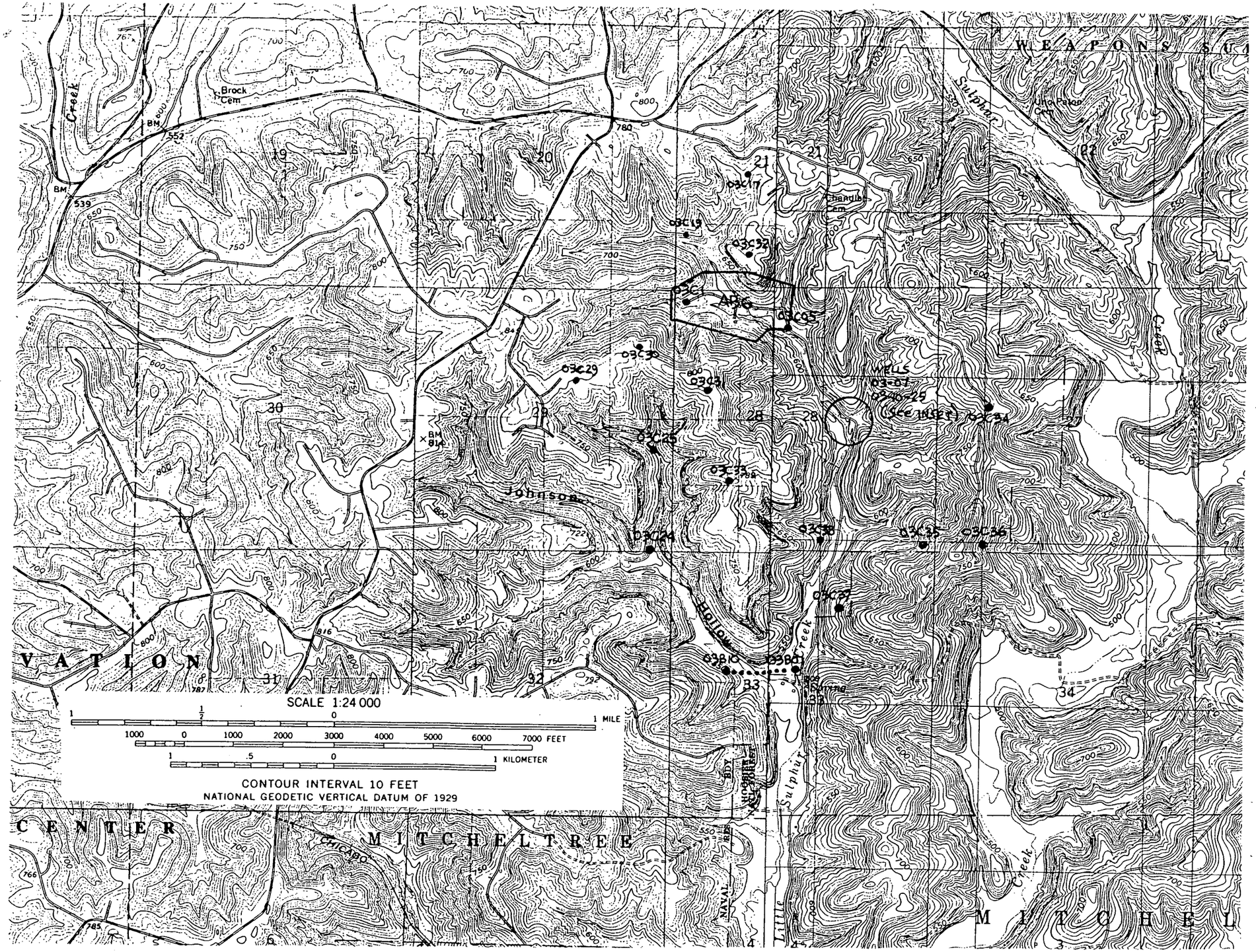
WEAPONS SUA
Brock Cem
Creek
Sulphur
Creek
Johnson
Hollow
Sulphur
Creek
Little
Creek
CENTER
MITCHELTREE
MITCHEL
VATION
CHICAGO
NAVAL
SCALE 1:24 000
1 MILE
7000 FEET
1 KILOMETER
CONTOUR INTERVAL 10 FEET
NATIONAL GEODETIC VERTICAL DATUM OF 1929

Figure 4

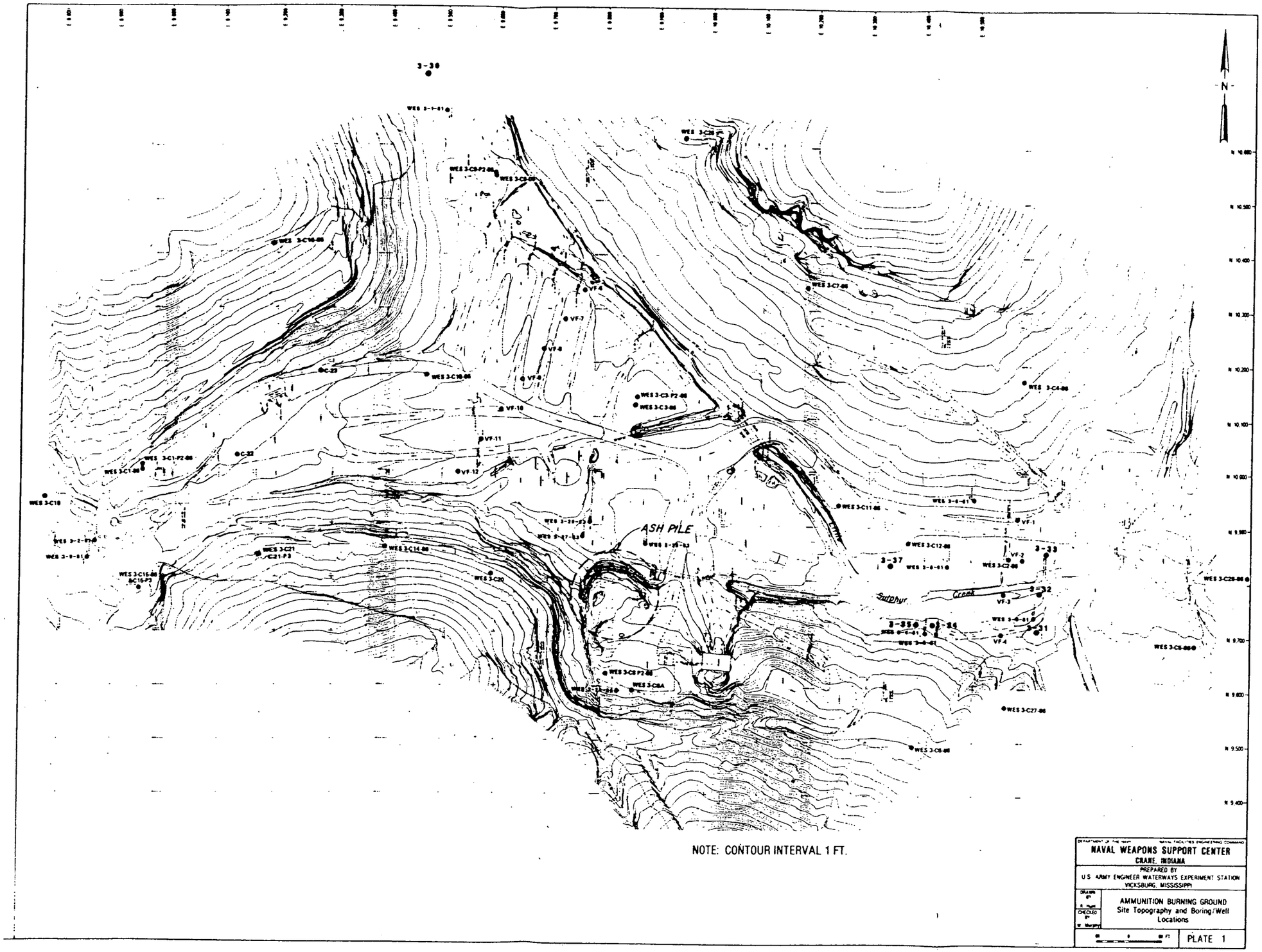
N
ASH PILE
Sulphur Creek
3-33
3-37
3-32
3-35
3-31
NOTE: CONTOUR INTERVAL 1 FT.
NAVAL WEAPONS SUPPORT CENTER
CRANE, INDIANA
PREPARED BY
U.S. ARMY ENGINEER WATERWAYS EXPERIMENT STATION
VICKSBURG, MISSISSIPPI
AMMUNITION BURNING GROUND
Site Topography and Boring/Well Locations
PLATE 1

Figure 5

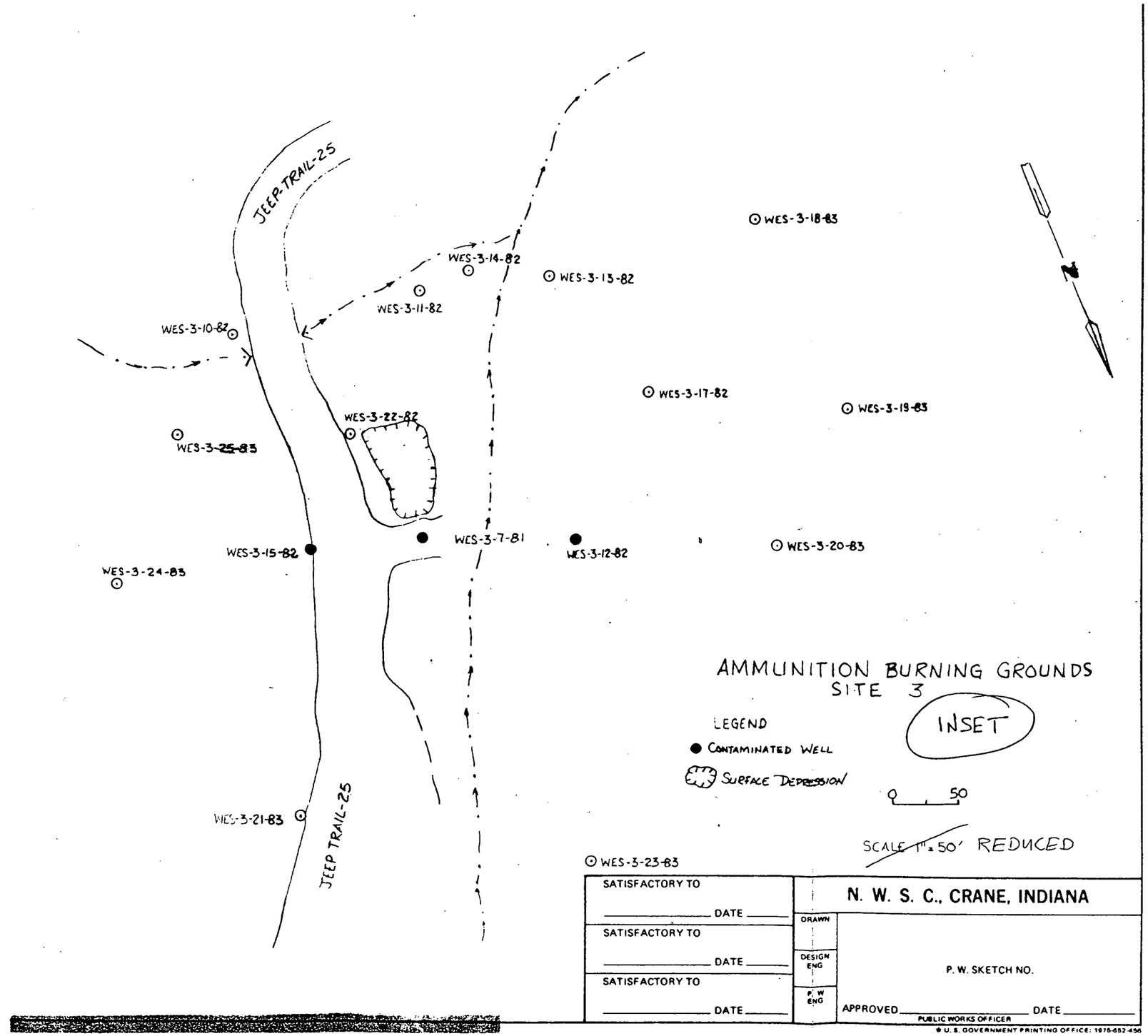
JEEP TRAIL-25
WES-3-18-83
WES-3-14-82
WES-3-13-82
WES-3-11-82
WES-3-10-82
WES-3-17-82
WES-3-19-83
WES-3-22-82
WES-3-25-83
WES-3-7-81
WES-3-15-82
WES-3-12-82
WES-3-20-83
WES-3-24-83
WES-3-21-83
JEEP TRAIL-25
AMMUNITION BURNING GROUNDS
SITE 3
INSET
LEGEND
CONTAMINATED WELL
SURFACE DEPRESSION
0 50
SCALE 1" = 50' REDUCED
WES-3-23-83
SATISFACTORY TO
DATE
SATISFACTORY TO
DATE
SATISFACTORY TO
DATE
N. W. S. C., CRANE, INDIANA
DRAWN
DESIGN ENG
P. W. ENG
P. W. SKETCH NO.
APPROVED
PUBLIC WORKS OFFICER
DATE
U. S. GOVERNMENT PRINTING OFFICE: 1975-652-456

Figure 6

TABLE 3
SUMMARY OF BORING AND MONITORING WELL DATA (COMPILED JULY, 1990)

Boring and Monitoring Well No.'s	Boring Sites (Cluster or Single Mon. Wells)*	Boring Types	Depth (Ft.)	Elev., Ground Surface (Ft.)	Elev., Top of Well Mon. Riser Pipe (Ft.)	Well Screened Interval (Depth Below Ground Surface; Ft.)	Geologic Formation Screened	Date Well Screen Set
WES-3-C1-86		Core	197.2		627.28	174.0 to 184.0	Beaver Bend	12/12/86
WES-3-C1P2-86	Cluster	Core	110.0		626.85	92.0 to 102.0	Beech Creek	09/22/86
WES-3-C1P3-86		Rock Bit	25.6		626.50	19.6 to 24.6	Golconda/Haney	04/ /86
WES-3-C2-86	Cluster	Core	132.7		583.49	119.5 to 129.5	Beaver Bend	10/24/86
WES-3-C2P2-86		Rock Bit	49.0		583.15	38.0 to 48.0	Beech Creek	10/29/86
WES-3-C3-86	Cluster	Core	150.7		600.08	139.0 to 149.0	Beaver Bend	09/13/86
WES-3-C3P2-86		Rock Bit	67.0		600.20	55.8 to 65.8	Beech Creek	10/23/86
WES-3-C4-86	Single	Core	85.3		624.42	73.5 to 83.5	Beech Creek	11/25/86
WES-3-C5-86	Single	Core	44.0		581.36	32.0 to 42.0	Beech Creek	10/29/86
WES-3-C6-86	Single	Core	100.6		625.40	87.0 to 97.0	Beech Creek	10/16/86
WES-3-C7-86	Single	Core	101.6		636.47	88.7 to 98.7	Beech Creek	10/21/86
WES-3-C8-86		Core	187.7		–	Well was lost and replaced by WES-3-C8A-86 (below)		
WES-3-C8P2-86	Cluster	Rock Bit	101.6		629.34	90.0 to 100.0	Beech Creek	11/26/86
WES-3-C8A-86		Rock Bit	186.0		630.50	171.0 to 181.0	Beaver Bend	04/03/87
WES-3-C8AP3-86		Rock Bit	20.5		630.43	14.5 to 19.5	Golconda/Haney	04/05/87

TABLE 3 (continued)

Boring and Monitoring Well No.'s	Boring Sites (Cluster or Single Mon. Wells)*	Boring Types	Depth (Ft.)	Elev., Ground Surface (Ft.)	Elev., Top of Well Mon. Riser Pipe (Ft.)	Well Screened Interval (Depth Below Ground Surface; Ft.)	Geologic Formation Screened	Date Well Screen Set
WES-3-C9-86	Cluster	Core	162.8		603.39	145.0 to 155.0	Beaver Bend	12/05/86
WES-3-C9P2-86		Rock Bit	73.0		603.56	62.0 to 72.0	Beech Creek	12/07/86
WES-3-C10-86	Single	Core	78.8		608.03	66.0 to 76.0	Beech Creek	01/22/87
WES-3-C11-86	Single	Core	59.5		592.36	47.5 to 57.5	Beech Creek	01/28/87
WES-3-C12-86	Single	Core	51.2		587.20	39.0 to 49.0	Beech Creek	02/03/87
WES-3-C13-86	Single	Core	83.2		617.75	70.2 to 80.2	Beech Creek	02/09/87
WES-3-C14-86	Single	Core	115.5		642.79	104.5 to 114.5	Beech Creek	02/20/87
WES-3-C15-86	Cluster	Core	101.1		621.34	88.5 to 98.5	Beech Creek	02/26/87
WES-3-C15P3-86		Rock Bit	22.0		621.11	15.6 to 20.6	Golconda/Haney	04/ /87
WES-3-C16-86	Single	Core	157.0		683.25	146.0 to 156.0	Beech Creek	03/12/87
WES-3-C17-86	Single	Core	149.5		696.15	138.5 to 148.5	Beech Creek	03/19/87
WES-3-C18-86	Single	Core	22.5		625.09	15.5 to 20.5	Golconda/Haney	04/ /87
WES-3-C19-86	Cluster	Core	122.5		650.50	110.8 to 120.8	Beech Creek	05/08/87
WES-3-C19P3-86		Rock Bit	44.0		649.70	37.0 to 42.0	Golconda/Haney	05/13/87
WES-3-C20-86	Single	Core	120.0		646.62	108.0 to 118.0	Beech Creek	05/24/87
WES-3-C21-86	Cluster	Core	123.0		642.22	108.4 to 118.4	Beech Creek	05/28/87
WES-3-C21P~~3~~2-86		Rock Bit	41.0		642.22	33.6 to 38.6	Golconda/Har	05/29/87

TABLE 3 (continued)

Boring and Monitoring Well No.'s	Boring Sites (Cluster or Single Mon. Wells)*	Boring Types	Depth (Ft.)	Elev., Ground Surface (Ft.)	Elev., Top of Well Mon. Riser Pipe (Ft.)	Well Screened Interval (Depth Below Ground Surface; Ft.)	Geologic Formation Screened	Date Well Screen Set
WES-3-C22-86	Single	Rock Bit	16.5		617.58	10.0 to 15.0	Golconda/Haney	06/04/87
WES-3-C23-86	Single	Rock Bit	15.5		618.61	8.5 to 13.5	Golconda/Haney	06/09/87
WES-3-C24-86	Single	Core	28.2		553.24	22.0 to 27.0	Beech Creek	06/16/87
WES-3-C25-86	Single	Core	70.7		597.03	59.7 to 69.7	Beech Creek	06/18/87
WES-3-C26-86	Single	Core	105.7		640.57	94.0 to 104.0	Beech Creek	08/03/87
WES-3-C27-86	Single	Core	73.3		603.84	60.5 to 70.5	Beech Creek	08/14/87
WES-3-C28-86	Single	Core	77.1		612.96	65.0 to 75.0	Beech Creek	08/22/87
WES-3-C29-86	Single	Core	278.8		797.37	267.5 to 277.5	Beech Creek	09/01/87
WES-3-C30-86	Single	Core	263.6		778.91	250.5 to 260.5	Beech Creek	09/17/87
WES-3-C31-86	Single	Core	267.2		792.10	255.0 to 265.0	Beech Creek	11/01/87
WES-3-C32-86	Single	Core	176.9		711.56	164.0 to 174.0	Beech Creek	11/14/87
WES-3-C33-86	Single	Core	249.8		785.03	238.5 to 248.5	Beech Creek	11/31/87
WES-3-B1-86	Single	Split-spoon	23.3		504.47	7.5 to 12.5	Alluvium	06/19/87
WES-3-B2-86	Single	Split-spoon	15.8		503.96	9.6 to 14.6	Alluvium	06/27/87
WES-3-B3-86	Single	Split-spoon	14.5		504.96	8.5 to 13.5	Alluvium	06/29/87
WES-3-B4-86	Single	Split-spoon	16.5		506.47	9.5 to 14.5	Alluvium	06/30/87
WES-3-B5-86	Single	Split-spoon	15.0		507.05	9.0 to 14.0	Alluvium	07/01/87

TABLE 3 (continued)

Boring and Monitoring Well No.'s	Boring Sites (Cluster or Single Mon. Wells)*	Boring Types	Depth (Ft.)	Elev., Ground Surface (Ft.)	Elev., Top of Well Mon. Riser Pipe (Ft.)	Well Screened Interval (Depth Below Ground Surface; Ft.)	Geologic Formation Screened	Date Well Screen Set
WES-3-B6-86	Single	Split-spoon	13.5		509.26	8.0 to 13.0	Alluvium	07/02/87
WES-3-B7-86	Single	Split-spoon	10.5		510.72	4.0 to 9.0	Alluvium	08/04/87
WES-3-B8-86	Single	Split-spoon	12.5		513.40	6.0 to 11.0	Alluvium	08/05/87
WES-3-B9-86	Single	Split-spoon	12.0		516.04	5.5 to 10.5	Alluvium	08/07/87
WES-3-B10-86	Single	Core	58.7		561.96	48.5 to 58.5	Beech Creek	10/22/87
WES-3-C34-89	Single	Core	193.9		727.07	177.4 to 187.4	Beech Creek	/89
WES-3-C35-89	Single	Core	177.7		697.00	164.4 to 174.4	Beech Creek	/89
WES-3-C36-89	Single	Core	183.1		718.24	169.0 to 179.0	Beech Creek	/89
WES-3-C37-89	Single	Core	116.7		620.28	105.5 to 115.5	Beech Creek	/89
WES-3-C38-89	Single	Core	55.5		572.97	42.7 to 52.7	Beech Creek	/89
WES-03-01-81	Single	Rock bit	56.2		604.67	41.6 - 50.8	Big Clifty/Beech Crk	/81
03-02-81	"	"	12.9		622.67	3.5 - 10.0	Mansfield	/81
03-03-81	"	"	100.4		584.29	85.9 - 95.0	Sample Shale	/81
03-04-81	"	"	70.0		594.17 ①	55.1 - 64.5	Beech Creek	/81
03-05-81	"	"	60.0		~595.75	45.4 - 54.7		/81
03-06-81	"	"	52.0		589.71	37.3 - 46.6	Beech Creek	/81
03-07-81	"	"	27.0		556.46	12.4 - 21.7		/81
03-08-81	"	"	47.0		~586.76 ①	32.4 - 41.8		/81
03-09-81	"	"	80.5		624.72	65.9 - 75.1	Big Clifty	/81
03-10-82	"	"	27.0		557.94	12.2 - 21.5		/82
03-11-82	"	"	20.5		553.05	5.7 - 15.1		/82
03-12-82	"	"	27.0		557.99	12.2 - 21.6		/82
03-13-82	"	"	27.0		552.24	12.0 - 21.4		/82

① Earlier survey (1981)

TABLE 3 (continued)

Boring and Monitoring Well No.'s	Boring Sites (Cluster or Single Mon. Wells)*	Boring Types	Depth (Ft.)	Elev., Ground Surface (Ft.)	Elev., Top of Well Mon. Riser Pipe (Ft.)	Well Screened Interval (Depth Below Ground Surface; Ft.)	Geologic Formation Screened	Date Well Screen Set
WES 03-14-82	Single	Rock bit	20.0		552.37	5.2 - 14.7		/82
03-15-82	"	"	30.5		~560.05 ①	15.7 - 25.1		/82
03-16-82	"	"	38.0		559.09	23.2 - 32.6		/82
03-17-82	"	"	27.0		553.44	12.3 - 21.7		/82
03-18-83	"	"	29.5		552.80	14.6 - 23.9		/83
03-19-83	"	"	35.0		~559.37 ①	20.1 - 29.5		/83
03-20-83	"	"	29.3		554.83	14.4 - 23.7		/83
03-21-83	"	"	27.5		560.12	12.8 - 22.1		/83
03-22-83	"	"	29.3		558.72	14.5 - 23.9		/83
03-23-83	"	"	29.7		562.34	14.9 - 23.3		/83
03-24-83	"	"	56.8		589.04	42.0 - 51.4		/83
03-25-83	"	"	39.0		573.64	24.2 - 33.5		/83
03-26-83	"	"	50.4		~595.89	35.9 - 45.0		/83
03-27-83	"	"	33.0		599.08	18.5 - 27.6	Big Clifty	/83
03-28-83	"	"	27.0		627.49	12.5 - 21.6	Mansfield & Golconda	/83
03-29-83	"	"	51.0		598.61	36.6 - 45.6	Big Clifty	/83
03-30-85	"	"	53.5		608.60		Big Clifty	10/04/85
03-31-85	"	"	46.0		582.84		Big Clifty /B.Creek	10/10/85
03-32-85	"	"	45.0		583.54		Big Clifty /B.Creek	10/11/85
03-33-85	"	"	41.0		583.39		Big Clifty, B.Creek	10/13/85
03-34-85	"	"	50.0		585.06		Big Clifty /B.Creek	10/21/85
03-35-85	"	"	51.0		586.60		Beech Creek	10/23/85
03-36-85	"	"	70.0		617.54		Big Clifty	10/25/85
03-37-85	"	"	48.9		586.52		Big Clifty /B.Creek	10/27/85
03-38-85	"	"	57.0		604.79		Big Clifty /B.Creek	11/02/85
03-39-85	"	"	55.0		603.25		Big Clifty /B.Creek	11/05/85

the QA/QC document. Soil and rock sampling logs were kept for each borehole and well as the borehole was advanced. All sampling activities, methods, and analytical results will be presented in written reports at the end of the RFI for the ABG.

(13) Calibration of field devices. Calibration of the air-monitoring devices is described in the Health and Safety Plan (part f. of this document).

(14) Sample preservation. Ground-water sample preservation methods are described in the QA/QC document. Soil and rock core samples collected for geologic analysis are stored in lidded jars and wooden core boxes respectively.

c. RFI Phase III Construction Plan. Construction of the wells occurred previously.

d. Quality Assurance/Quality Control documentation. The QA/QC documentation, Part 1.d., is attached at the end of this Plan.

e. RFI Phase III Data Management Plan.

(1). RFI Phase III Data Record. Records of data collected in the field and in the laboratory will be maintained indefinitely for the Modified ABG RFI in a dedicated file at WES. Some duplicate records will be maintained at the NWSC. Field data records will include logs of all borings and the core or other samples extracted from them, well installation logs, results of dye trace tests and other field investigations, memoranda for record of field visits, raw and final positioning survey data, and monitoring well sampling activities.

Each monitoring well water quality sample taken during the investigation will be assigned a unique number corresponding to the well or boring from which it was obtained. Well numbers will identify the SWMU number (Site 03) and the particular well in a cluster. An example of a well number would be 03C03P2, which identifies well P2 of cluster C03 of site 03. Water samples will be identified by well number, depth and date sampled. Each water sample will be assigned a laboratory analysis ID number by the contract laboratory. Dye detection samples collected during the dye trace will be number consecutively. Data on dye collection and analysis will include date and location dye detector placed, date collected, date analyzed and results of analysis.

(2). RFI Phase III Tabular Displays. Tables of data will be prepared for well information and water sampling and analysis events. Well information tables will list well numbers, dates installed, type of boring, total depth, elevation of well casing and ground, depth to screen and screen length, and identification of aquifer. Water level measurements for dates measured for each well will be tabulated. Tabulated water sampling data will include site, well number, date and depth sampled and laboratory analysis results for each parameter tested. Results of analysis of dye tracers will be tabulated.

(3). RFI Phase III Graphical Displays. Graphical displays will include site maps showing well locations, topographic maps of the site, water table or piezometric surface contour maps of the aquifers encountered, contour maps of pertinent geologic structure, maps of ground-water contamination distribution in sampled aquifers, and other maps as needed. Line or bar graphs will be

constructed to show variations in water or piezometric levels for different aquifers, for different wells, and for different dates. Graphs will also be constructed showing distribution in contaminant concentrations for different aquifers, wells and dates. Other graphs will be constructed as necessary. Photographs of field and laboratory activities will be included in reports of investigation when available.

f. RFI Phase III Health and Safety Plan.

Background. An initial assessment study of the NWSC conducted in 1981 to identify potential contamination sources determined that past activities at the NWSC have resulted in possible contamination of soil and ground water by potentially hazardous chemicals in several areas of the facility. The chemicals, by their presence in the soil and ground water, present a potential hazard to personnel conducting RFIs. RFI activities require drilling of subsurface borings and installation of wells (completed previously), and sampling of soils, rock and ground and surface waters. The health and safety plan is intended to (a) describe field activities to identify potential hazards associated with each activity, (b) insure safety consciousness by each individual involved with the field activities, (c) control exposure to potentially hazardous chemical compounds by specifying protective gear and monitoring procedures commensurate with the anticipated risk, and (d) provide emergency procedures in the event of unanticipated high contaminant concentrations, incident response, control and disposal of generated waste materials.

Other activities. Other field activities that could expose personnel to contaminants include well sampling, well testing, and surface water sampling and flow measurement. Well sampling requires removal of several bore quantities of water from the well, the retention of a sample of the ground water entering the well, and preparation of the sample for shipment to the laboratory for analysis.

Well testing requires measuring the rate of flow of water being withdrawn from or injected into the aquifer through the well, for example by pumping tests or by slug tests, for the purpose of determining aquifer flow parameters. Well testing may also require measurement of water levels in monitoring wells near the pumping well. Because personnel may contact withdrawn ground water and be exposed to vapors emanating from the wells, potential splash and vapor ingestion hazards exist.

Additional flow measurement may be required whereby surface waters in streams and water issuing from springs are sampled and the rate of flow determined or where injected dyes are collected and analyzed. Concentrations of contaminants in surface waters will be decreased by dilution. Some dilution and aeration will also occur in spring fed waters. Field personnel should nonetheless be alert for any signs of contamination such as discoloring of the water or the presence of odors or vapors. Protective rubber gloves will be worn when collecting surface or spring water samples.

General Personnel Safety. The monitoring and precautionary measures specified in the following paragraphs are considered reasonable and prudent for general field investigations planned for the RFI. Each employee will have a comprehensive physical examination within the 12 months preceding his/her field

involvement to provide background information. Upon completion of the project, or annually, whichever occurs first, each employee will have a routine physical examination. Physical examinations will consist of the following:

a. Comprehensive Physical Examination. Basic health history, basic physical examination, routine EKG, chest X-ray, pulmonary function, audio testing, eye evaluation, hemoccult, Chem-screen 26, CBC w/differential, acetylcholinesterase activity, heavy metals in blood to include lead, cadmium, mercury, arsenic, chromium, and strontium; urinalysis.

b. Routine Physical Examination. Basic physical examination, pulmonary function, SMA-26, urinalysis, audiometric.

Air Monitoring. The most common halogenated organic compounds in industrial use and OSHA allowable inhalation limits are listed in Table 4 (OSHA Safety and Health Standards 29CFR 1910).

Table 4. Limits of Exposure and Detection for Common Organic Solvents.

Solvent	Maximum Allowable* Exposure, 8 hrs.	Sensitivity** Photoionization	Detection*** Limit
Benzene	10 ppm	High	5 ppm
1,1 Dichloroethane	100 ppm	High	-
1,2 Dichloroethane	200 ppm	High	-
Methylene Chloride	500 ppm	High	100 ppm
Trichloroethylene	100 ppm	High	2 ppm

* OSHA Safety and Health Standards (29 CFR 1910) OSHA 2206
** HNu Model PI-101 with 11.7 ev lamp, HNu Systems, Newton, Mass.
*** Draeger Detector Tubes, National Draeger Inc., Pittsburg, PA

Air monitoring efforts will concentrate on monitoring for organic solvents and combustible gas. The presence of dust will be determined visually.

The presence of volatile organic compounds in the head space of the monitoring well will be monitored upon removal of the protective cap using a photoionization organic vapor detector (HNu Systems Model 101, Hazardous Waste Detector).

The photoionization unit is a non-specific detector that can semiquantitatively determine the concentration of a broad range of potentially hazardous organic compounds in the air. The unit will be calibrated relative to benzene concentration or to standards traceable to benzene. The unit will be set to provide an audible alarm at 75 percent of the threshold limit value (TLV) for benzene. The photoionization unit will be calibrated in accordance with manufacturer's recommendation and checked periodically for proper operation.

Limiting skin contact. All persons involved in handling soil or water samples or equipment contaminated with soil or water from the work site will wear rubber gloves and safety glasses or face shields. Higher levels of protective clothing, such as Levels A, B, or C, will be issued and worn if or when working conditions require them. Protective equipment will be inspected daily. Worn or damaged equipment will be cleaned and discarded or disposed of as contaminated waste.

1. Task A: RFI Phase III Work Plan, Ammunition Burning Ground, SWMU #03/10

d. Quality Assurance/Quality Control Plan

The QA/QC plan for this work is presented in two parts: Part I discusses Sample Collection and Management; Part II discusses Laboratory Analyses.

QUALITY ASSURANCE/QUALITY CONTROL FOR GROUND-WATER SAMPLING AND ANALYSIS

Part I. SAMPLE COLLECTION AND MANAGEMENT

1. Introduction

The references for this document are the "USATHAMA QA Program", U.S. Army Toxic and Hazardous Materials Agency, Aberdeen Proving Ground, Maryland, Dec, 1985 (2nd Ed Mar, 1987) and "Chemical Analysis Quality Assurance Requirements for the Navy Installation Restoration Program", NEESA 20.2-047B, Naval Energy and Environmental Support Activity, Port Hueneme, California, June 1988. The procedures described in this section are designed to obtain samples which are proper representations of the sampled matrix. Trace levels of contaminants from sources external to the sample must be eliminated through the use of good sampling techniques. Sample management and stringent documentation are the key factors in a successful QA program for sampling.

This document does not discuss sampling of soil, air, biological, or surface matrices or sampling for radiological constituents. When such matrices or analytes are included in a project, detailed requirements and protocols will be provided on a case-by-case basis.

Part I of this document discusses selection and cleaning of containers, field QC samples, the chain of custody, shipping requirements, sample receipt, sampling of volatiles, ground-water sampling equipment, procedures for monitor well sampling, and lists sample preservation procedures and suggested holding times. Part II details laboratory analysis QA/QC.

2. Containers

For water samples, the sample container shall be chosen to be compatible with the analyte(s) of interest. A complete list is provided in Appendix H. In general, however, the following containers should be used: (Exceptions will be noted in the standardized method).

- Septum-sealed glass vials for volatile compounds;

- Amber glass bottles with Teflon-lined lids for organic constituents other than volatiles;

- Polyethylene bottles for inorganic analytes; and

- Wide-mouth amber-glass bottles shall be used for all soil and sediment samples.

2.1 Cleaning Procedures for Glass Bottles

In general, only ICHEM certified-clean containers will be used. When containers must be cleaned, the following procedures will be used.

a. Wash glass bottles, Teflon liners, and caps in hot tap water with laboratory-grade nonphosphate detergent.

b. Rinse three times with tap water.

c. Rinse with 1:1 nitric acid (metals-grade), American Society for Testing Materials (ASTM) Type I deionized water.

d. Rinse three times with ASTM Type I deionized water.

e. Rinse with pesticide-grade methylene chloride using 20 mL for 1/2-gal container and 5 mL for 4- and 8-oz containers.

f. Oven dry at 125°C. Allow to cool to room temperature in an enclosed contaminant-free environment.

g. Place liners in lids and cap containers.

h. Store in contaminant-free area. (Amber glass containers shall not be exposed to sunlight).

2.2 Cleaning Procedure for Bottles Used for Volatile Organics (40-mL Glass)

a. Wash glass vials, Teflon-backed septa, Teflon liners, and caps in hot tap water using laboratory-grade nonphosphate detergent.

b. Rinse three times with tap water.

c. Rinse three times with ASTM Type I deionized water.

d. Oven dry vials, septa, and liners at 125°C.

e. Allow vials, septa, and liners to cool to room temperature in an enclosed contaminant-free environment.

f. Seal 40-mL vials with septa (Teflon side down) and cap.

g. Store in contaminant-free area.

2.3 Cleaning Procedure for Polyethylene Bottles

a. Wash polyethylene bottles and caps in hot tap water with laboratory-grade nonphosphate detergent.

b. Rinse with 1:1 nitric acid (metals-grade), ASTM deionized water.

c. Rinse three times with ASTM Type I deionized water.

d. Invert and air dry in contaminant-free environment.

2.4 All Bottles Will Be

a. Capped and labeled with sample numbers and packed in cooler or box.

b. Stored in contaminant-free area.

3. Field QC Samples

Although the number of QC samples changes, the types of field QC samples remain the same regardless of the level of QC implemented. Table I lists the percentage of field QC samples per level per sample matrix. A sampling event is considered to be from the time the sampling personnel arrive at the site until these personnel leave for more than a day. An example of two events would occur if sampling personnel went to a site for three weeks, drilled borings, and put ground-water wells in place. During this visit, soil and water samples were collected. The sampling crew left the site for two months, thus concluding the first sampling event. The crew later returned to collect another set of ground-water samples over a three-day period. The second visit would constitute the second sampling event.

The following information defines and explains the blanks, duplicates, and referee samples.

a. Trip Blanks

Trip blanks are defined as samples which originate from analyte-free water taken from the laboratory to the sampling site and returned to the laboratory with the volatile organic (VOA) samples. One trip blank should accompany each cooler containing VOAs, should be stored at the laboratory with the samples, and analyzed by the laboratory. Trip blanks are only analyzed for VOAs.

b. Equipment Rinsates

Equipment rinsates are the final analyte-free water rinse from equipment cleaning collected daily during a sampling event. Initially, samples from every other day should be analyzed. If analytes pertinent to the project are found in the rinsate, the remaining samples must be analyzed. The results from the blanks will be used to flag or assess the levels of analytes in the samples.

NEESA

Table 3.4. Field QC samples per sampling event

Type of Sample	Level D		Level C		Level E	
	Metal	Organic	Metal	Organic	Metal	Organic
Trip blank (for volatiles only)	NA[1]	1/cooler	NA[1]	1/cooler	NA[1]	1/cooler
Equipment rinsate[2]	1/day	1/day	1/day	1/day	1/day	1/day
Field blank	1/source/event for all levels and all analytes					
Field duplicates[3]	10%	10%	10%	10%	5%	5%
Referee duplicate[3]	collect at direction of HAZWRAP Project Manager					

[1]NA - Not applicable.

[2]Samples are collected daily; however, only samples from every other day are analyzed. Other samples are held and analyzed only if evidence of contamination exists.

[3]The duplicate must be taken from the same sample which will become the laboratory matrix/spike duplicate for organics or for the sample used as a duplicate in inorganic analysis.

blank should accompany each cooler containing VOAs, should be stored at the laboratory with the samples, and analyzed by the laboratory. Trip blanks are only analyzed for VOAs.

2. Equipment Rinsates

Equipment rinsates are the final analyte-free water rinse from equipment cleaning collected daily during a sampling event. Initially, samples from every other day should be analyzed. If analytes pertinent to the project are found in the rinsate, the remaining samples must be analyzed. The results from the blanks will be used to flag or assess the levels of analytes in the samples. This comparison is made during data validation. The rinsates are analyzed for the same parameters as the related samples.

3. Field Blanks

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

This comparison is made during data validation. The rinsates are analyzed for the same parameters as the related samples.

c. Field Blanks

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

d. Field Duplicates/Splits

Duplicates or splits for soil samples are collected, homogenized, and split. All samples except VOAs are homogenized and split. Volatiles are not mixed, but select segments of soil are taken from the length of the core and placed in 40-mL glass vials. Cores may be sealed and shipped to the laboratory for subsampling if the project deems this appropriate. The duplicates for water samples should be collected simultaneously. Field duplicates should be collected at a frequency of 10% per sample matrix for Levels D and C. For Level E, the duplicates should be analyzed at a frequency of 5%. All the duplicates should be sent to the primary laboratory responsible for analysis. The same samples used for field duplicates shall be split by the laboratory and be used as the laboratory duplicate or matrix spike. This means that for the duplicate sample, there will be analyses of the normal sample, the field duplicate, and the laboratory matrix spike/duplicate.

e. Referee Duplicates

Duplicates/splits shall be sent to the referee QA laboratory if regulators (state or region) collect split samples or if a special problem occurs in sample analysis or collection. These duplicates/splits are collected and analyzed in addition to the field duplicates mentioned in the previous paragraph.

4. Chain of Custody

Samples, other than those collected for in situ field measurements or analyses, are identified by using a standard sample label which is attached to the sample container. The sample labels are sequentially numbered and are accountable. The following information shall be included on the sample label.

a. Site name.

b. Field identification or sample station number.

c. Date and time of sample collection.

d. Designation of the sample as a grab or composite.

e. Type of sample (matrix) and a brief description of the sampling location.

f. The signature of the sampler.

g. Sample preservation and preservative used.

h. The general types of analyses to be conducted.

If a sample is split with another party, sample labels with identical information shall be attached to each of the sample containers.

The COC record is used to record the custody of samples and shall accompany samples at all times. The following information shall be supplied to complete the COC record.

a. Project name.

b. Signature of samplers

c. Sampling station number or sample number, date and time of collection, grab or composite sample designation, and a brief description of the type of sample and sampling location.

d. Signatures of individuals involved in sample transfer (i.e., relinquishing and accepting samples). Individuals receiving the samples shall sign, date, and note the time that they received the samples on the form.

e. Matrix.

Sample analysis request sheets serve as official communication to the laboratory of the particular analyses required for each sample and provide further evidence that the COC is complete.

COC records initiated in the field shall be placed in a plastic cover and taped to the inside of the shipping container used for sample transport from the field to the laboratory.

Examples of chain-of-custody sample tags and record are presented in Figures ~~5~~ 2 and ~~6~~ 4, respectively, of Part II of the QA/QC document.

5. <u>Shipping Requirements</u>

Shipping containers shall be secured using nylon strapping tape and custody seals (tamper-proof seals) to ensure that samples have not been disturbed during transport. The custody seals shall be placed on the containers so they cannot be opened without breaking the seal. An example of a tamper-proof seal is shown in Figure ~~4~~ 3. of Part II of the QA/QC document.

Samples which must be kept at 4°C shall be shipped in insulated containers with either freezer forms or ice. If ice is used, it shall be placed in a container so that the water will not fill the cooler as the ice melts. The samples shall be delivered to the laboratory soon enough to allow the laboratory to meet holding times. The Department of Transportation

regulations shall be used for packaging, quantities of shipment, and the way samples are sent. Each subcontractor responsible for sampling shall become familiar with regulations.

Copies of the signed COC forms shall be delivered with the data packages. The originals shall remain on file with the contractor or with the laboratory.

6. Sample Receipt

Upon receipt, the laboratory shall sign and keep copies of the air bill. The COC shall be signed. The temperature of the cooler shall be measured and documented. The condition of the samples shall be documented. If any breakage or discrepancy arises between COC, sample labels, and requested analysis, the sample custodian will notify the engineering subcontractor. The pH of incoming samples shall be checked and documented upon receipt. Any discrepancy or improper preservation shall be noted by the laboratory as an out-of-control event and shall be documented on an out-of-control form with the corrective action taken. The out-of-control form shall be signed and dated by the custodian and any other person responsible for corrective action.

7. Sampling of Volatiles

When sampling water for volatile compounds, extra care must be exercised to prevent analyte loss by evaporation. Precautionary measures include:

- Acquiring the sample with equipment that minimizes water gas/liquid interphase under pressure or vacuum;
- Avoiding aeration or agitation of the sample to the greatest possible extent;
- Triple rinse sample vial with sample water;
- Filling vials to capacity, taking care that no air bubbles are trapped in the vial;

- Storing the sample at 4°C;
- Analyzing the sample as soon as possible, but never exceeding the prescribed holding time (Appendix H);
- Never allowing a volatile sample to freeze; and
- Never filtering the sample.

8. Ground-water Sampling Equipment

All ground-water sampling will occur after the wells have been developed. Because drilling and well construction disturb the natural ground-water system, the maximum possible length of time (never less than two weeks) shall pass between well development and sampling to allow the ground-water system to return to chemical equilibrium.

All equipment used to measure and sample the ground-water system (e.g., bailers, pumps, tapes, ropes) must be cleaned before use in each well to prevent cross contamination between wells. Equipment that is dedicated to a well site may not require cleaning between sampling events. If the well is free of inflowing sediments, thorough rinsing will be sufficient. When inflowing sediments adhere to equipment, scrubbing may be required in addition to rinsing. In no instance shall detergents, soaps, or solvents be used to clean equipment in the field.

Water used for rinsing field equipment shall be bottled distilled water or water from an approved source. Such approved water should originate from an uncontaminated (background) and untreated source. The water shall be analyzed by an approved laboratory for all project specific analytes prior to collection of field samples. A rinse blank shall be included with the initial lot of samples during the initial and subsequent sampling excursions, defined as the time between mobilization and demobilization of the sampling team. Reanalysis of rinse blanks is not required for sampling excursions separated by less than six months. Water from chemical supply companies or retail

merchants is acceptable, provided that analysis reveals such water is free of interferences. At least one sample must be submitted to the laboratory and be analyzed for all analytes of interest prior to the first use in the field. The initial rinse water analyses may be done prior to certification approval provided that the analytical procedures used are identical to those tested during certification.

Sampling equipment must be protected from ground surface contamination. Clean plastic sheeting spread around the well is one means of protecting the equipment. New protective sheeting should be used at each sampling location. Sampling efforts shall preclude wind-blown particles from contaminating the sample or sampling equipment.

9. Monitor Well Sampling Procedures

The following procedures incorporate the necessary aspects of sampling QA and shall be used each time a monitor well is sampled:

- Measure the depth from the top of the well casing (not protective casing) to the top of the water and record the depth in the sampling logbook;

- Measure and record the depth from the top of the casing to the bottom of the sediment/water interface;

- Subtract the depth to top of the water from the depth to the bottom of the sediment/water interface to determine the height of standing water in the casing and saturated annulus. Remember to have on hand the diameter, height, and porosity of the sand pack, as recorded by the geologists during well construction;

- Obtain a sample of ground-water for temperature, conductivity, and pH measurements. Record these measurements in the sampling logbook;

- Remove a quantity of water from the well equal to three times the calculated volume of water in the well, including the saturated annulus;

- If the well goes dry during pumping or bailing, one is assured of removing all water which had prolonged contact with the well casing or air. If the recovery rate is rapid, allow the well to recover to its original level and evacuate a second time before sampling. If recovery is very slow, samples may be obtained as soon as sufficient water is available;

- Obtain samples for chemical analysis immediately after pumping or bailing is complete. For slow recovery wells, the sample shall be collected immediately after a sufficient volume is available;

- After obtaining chemical analysis samples, draw a second sample for temperature, conductivity, and pH measurement and record results in the sampling logbook;

- Filter samples, as appropriate;

- All samples must be placed in containers that have been cleaned according to the protocols in Section 2. Samples for organic analyses shall be placed in clean amber-glass bottles with Teflon-lined lids. Samples for inorganic chemical analyses shall generally be placed in clean polyethylene bottles. Samples for volatile organics shall be placed in septum-sealed vials. The sample bottle and cap shall be triple rinsed with the water being sampled before filling the bottle with the sample to be analyzed. Bottles for filtered samples shall be rinsed with filtered sample water and bottles for unfiltered samples should be rinsed with unfiltered sample water;

- Add the appropriate preservative and cap securely;

- Label samples; and

- Place sample bottle(s) in a temperature-controlled (4°C) chest immediately after sampling and deliver to the laboratory as soon as possible.

APPENDIX H

SAMPLE PRESERVATION PROCEDURES AND HOLDING TIMES FOR AQUEOUS AND SOLID SAMPLES

After the samples have been taken, they should be sent to the laboratory for analysis as expeditiously as possible in order to insure that the most reliable and accurate answers will be obtained as a result of the analysis. As a general rule, storage at low temperature is the best way to preserve most samples, although the length of time the sample can be held even at low temperatures varies with the analyte and matrix. The bottles should be packaged for shipping in insulated containers, constructed to insure that the bottles will arrive at the laboratory intact.

When the samples are received at the laboratory the time lapse between sample acquisition and analysis may not exceed the times shown in the table. Freezing samples to extend holding shall not be permitted.

The following table summarizes containers, preservation, and holding time requirements by analyte and sample matrix.

Table H-1. Containers, Preservation, Storage, and Holding Times[a]

Parameter	Container[b] Water	Container[b] Soil	Preservative[c,d] Water	Preservative[c,d] Soil	Maximum Holding Time for all Matrices[e]
INORGANIC TESTS					
Acidity	P	G	Cool, 4°C	Cool, 4°C	14 days
Alkalinity	P	G	Cool, 4°C	Cool, 4°C	14 days
Amnonia	P	G	Cool, 4°C H_2SO_4 to pH <2	Cool, 4°C	28 days
Asbestos	P	G	Cool, 4°C	Cool, 4°C	48 hours[f]
Bicarbonate	P	G	None Required	None Required	Analyze Immediately
Biochemical Oxygen Demand (BOD) and Carbonaceous BOD	P	G	Cool, 4°C	Cool, 4°C	48 hours
Bromide	P	G	None Required	None Required	28 days
Carbonate	P	G	None Required	None Required	Analyze Immediately
Chemical Oxygen Demand (COD)	P	G	Cool, 4°C H_2SO_4 to pH <2	Cool, 4°C	28 days
Chloride	P	G	None Required	None Required	28 days
Chlorine, Total Residual	P	N/A	None Required	N/A	Analyze Immediately
Color	P	N/A	Cool, 4°C	N/A	48 hours

Table H-1. (Cont'd.)

Parameter	Container[b] Water	Container[b] Soil	Preservative[c,d] Water	Preservative[c,d] Soil	Maximum Holding Time for all Matrices[e]
Cyanide, Total and Amenable to Chlorination	P	G	Cool, 4°C NaOH to pH >12 0.6 g Ascorbic Acid[g]	Cool, 4°C	14 days [h]
Dissolved Oxygen					
Probe	G Bottle and Top	N/A	None Required	N/A	Analyze Immediately
Winkler	G Bottle and Top	N/A	Fix On Site Store in Dark	N/A	8 hours
Fluoride	P	G	None Required	None Required	28 days
Hardness	P	N/A	HNO_3 or H_2SO_4 to pH<2	N/A	6 months
Hydrazine	P	G	If not analyzed immediately, collect under acid. Add 90 ml of sample to 10 ml HCl.	Cool, 4°C	7 days
Iodide	P	G	Cool, 4°C	Cool, 4°C	24 hours
Iodine	P	G	None Required	None Required	Analyze Immediately
Kjeldahl and Organic Nitrogen	P	G	Cool, 4°C H_2SO_4 to pH <2	Cool, 4°C	28 days

Table H-1. (Cont'd.)

Parameter	Container[b] Water	Container[b] Soil	Preservative[c,d] Water	Preservative[c,d] Soil	Maximum Holding Time for all Matrices[e]
Metals[i]					
Chromium VI	P	G	Cool, 4°C	Cool, 4°C	24 hours
Mercury	P	G	HNO_3 to pH <2	Cool, 4°C	28 days
Others	P	G	HNO_3 to pH <2	Cool, 4°C	6 months
Nitrate	P	G	Cool, 4°C	Cool, 4°C	48 hours
Nitrate plus Nitrite	P	G	Cool, 4°C H_2SO_4 to pH <2	Cool, 4°C	28 days
Nitrite	P	G	Cool, 4°C	Cool, 4°C	48 hours
Oil and Grease	G	G	Cool, 4°C H_2SO_4 to pH <2	Cool, 4°C	28 days
Orthophosphate	P	G	Filter Immediately Cool, 4°C	Cool, 4°C	48 hours
pH	P	G	None Required	None Required	Analyze Immediately
Phenols	G	G	Cool, 4°C H_2SO_4 to pH <2	Cool, 4°C	28 days
Phosphorous, Elemental	G	G	Cool, 4°C	Cool, 4°C	48 hours
Phosphorous, Total	P,G	G	Cool, 4°C H_2SO_4 to pH <2	Cool, 4°C	28 days
Silica, Dissolved or Total	P	G	Cool, 4°C	Cool, 4°C	28 days

Table H-1. (Cont'd.)

Parameter	Container[b] Water	Container[b] Soil	Preservative[c,d] Water	Preservative[c,d] Soil	Maximum Holding Time for all Matrices[e]
Residue					
Filterable	P	N/A	Cool, 4°C	N/A	7 days
Settleable	P	N/A	Cool, 4°C	N/A	48 hours
Nonfilterable (TSS)	P	N/A	Cool, 4°C	N/A	7 days
Total	P	N/A	Cool, 4°C	N/A	7 days
Volatile	P	N/A	Cool, 4°C	N/A	7 days
Specific Conductance	P	G	Cool, 4°C	Cool, 4°C	28 days
Sulfate	P	G	Cool, 4°C	Cool, 4°C	28 days
Sulfide	P	G	Cool, 4°C Add Zinc Acetate plus NaOH to pH >9	Cool, 4°C	7 days
Sulfite	P	G	None Required	None Required	Analyze Immediately
Surfactants	P	G	Cool, 4°C	Cool, 4°C	48 hours
Temperature	P	G	None Required	None Required	Analyze Immediately
Turbidity	P	N/A	Cool, 4°C	N/A	48 hours
ORGANIC TESTS[j]					
Acrolein and Acrylonitrile	S	S	Cool, 4°C 0.008% $Na_2S_2O_3$[g] Adjust pH to 4-5[k]	Cool, 4°C	14 days[k]

Table H-1. (Cont'd.)

Parameter	Container[b] Water	Container[b] Soil	Preservative[c,d] Water	Preservative[c,d] Soil	Maximum Holding Time for all Matrices[e]
Benzidines[1]	G	G	Cool, 4°C[m] 0.008% $Na_2S_2O_3$[g] pH 2-7	Cool, 4°C	7 days until extraction[n]
Chlorinated Hydrocarbons[1]	G	G	Cool, 4°C	Cool, 4°C	7 days until extraction 40 days after extraction
Haloethers[1]	G	G	Cool, 4°C 0.008% $Na_2S_2O_3$[g]	Cool, 4°C	7 days until extraction 40 days after extraction
Nitroaromatics and Isophorone[1]	G	G	Cool, 4°C Store in Dark	Cool, 4°C Store in Dark	7 days until extraction 40 days after extraction
Nitrosamines[1,o]	G	G	Cool, 4°C Store in Dark 0.008% $Na_2S_2O_3$[g]	Cool, 4°C Store in Dark	7 days until extraction 40 days after extraction
PCBs	G	G	Cool, 4°C	Cool, 4°C	7 days until extraction 40 days after extraction
Pesticides[1]	G	G	Cool, 4°C pH 5-9[p]	Cool, 4°C	7 days until extraction 40 days after extraction
Phenols[1]	G	G	Cool, 4°C 0.008% $Na_2S_2O_3$[g]	Cool, 4°C	7 days until extraction 40 days after extraction
Phthalate Esters[1]	G	G	Cool, 4°C	Cool, 4°C	7 days until extraction 40 days after extraction

Table H-1. (Cont'd.)

Parameter	Container[b] Water	Container[b] Soil	Preservative[c,d] Water	Preservative[c,d] Soil	Maximum Holding Time for all Matrices[e]
Polynuclear Aromatic Hydrocarbons[1]	G	G	Cool, 4°C 0.008% $Na_2S_2O_3$[g] Store in Dark	Cool, 4°C Store in Dark	7 days until extraction 40 days after extraction
Purgeable Aromatic Hydrocarbons	S	S	Cool, 4°C 0.008% $Na_2S_2O_3$[g] HCl to pH<2[q]	Cool, 4°C	14 days[q]
Purgeable Halocarbons	S	S	Cool, 4°C 0.008% $Na_2S_2O_3$[g]	Cool, 4°C	14 days
TCDD[1]	G	G	Cool, 4°C 0.008% Na_2SO_3[g]	Cool, 4°C	7 days until extraction 40 days after extraction
Total Organic Carbon	G	G	Cool, 4°C HCl or H_2SO_4 to pH <2	Cool, 4°C	28 days
Total Organic Halogen	G	G	Cool, 4°C 1 ml of 0.1 M sodium sulfite	Cool, 4°C	7 days

Analytes not listed should be preserved at 4°C and held not longer than 7 days.

[a]Preservatives and holding times are from Federal Register, Vol. 49, No. 209, Friday, October 26, 1984, Page 43260 and Characterization of Hazardous Waste Sites: A Methods Manual -- Volume II, Sampling Methods, Second Edition, EPA-600/4-84-076. Container requirements are consistent with these references.

[b]P = Polyethylene
G = Amber Glass with Teflon-lined cap
S = Glass Vial with Teflon-lined septum cap

[c]Sample preservation should be performed immediately upon sample collection. For composite samples, each aliquot should be preserved at the time of collection. When use of an automatic sampler makes it impossible to preserve each aliquot, samples may be preserved by maintaining at 4°C until compositing and sample splitting is completed.

[d]When any sample is to be shipped by common carrier or sent through the U.S. Mail, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172). The person offering such material for transportation is responsible for ensuring such compliance. For the preservation requirements in this table, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation, has determined that the Hazardous Materials Regulations do not apply to the following materials: Hydrochloric acid (HCl) in water solutions at concentrations of 0.04% by weight or less (pH about 1.96 or greater); Nitric acid (HNO_3) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H_2SO_4) in water solutions at concentrations of 0.35% by weight or less (pH about 1.15 or greater); and Sodium hydroxide (NaOH) in water solutions at concentrations of 0.080% by weight or less (pH about 12.3 or less).

[e]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.

Some samples may not be stable for the maximum time period given in the table. A laboratory is obligated to hold the sample for a shorter time if knowledge exists to show this is necessary to maintain sample integrity.

[f]If samples cannot be filtered within 48 hours, add 1 ml of a 2.71% solution of mercuric chloride to inhibit bacterial growth.

[g]Should only be used in the presence of residual chlorine.

[h]Maximum holding time is 24 hours when sulfide is present. Optionally, all samples may be tested with lead acetate paper before pH adjustment in order to determine if sulfide is present. If sulfide is present, it can be removed by addition of cadmium nitrate powder until a negative spot test is obtained. The sample is filtered and then NaOH is added to pH 12.

[i]For dissolved metals, filter immediately on site before adding preservative.

[j]Guidance applies to samples to be analyzed by GC, LC, or GC/MS for specific compounds.

[k]The pH adjustment is not required if acrolein will not be measured. Samples for acrolein receiving no pH adjustment must be analyzed within three days of sampling.

[l]When the extractable analytes of concern fall within a single chemical category, the specified preservative and maximum holding times must 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 pH to 6-9; samples preserved in this manner may be held for 7 days before extraction and 40 days after extraction. Exceptions to this optimal preservation and holding time procedure are noted in footnotes g, m, and n.

[m]If 1,2-diphenylhydrazine is likely to be present, adjust the pH of the sample to 4.0 ± 0.2 to prevent rearrangement to benzidine.

[n]Extracts may be stored up to 7 days before analysis if storage is conducted under an inert (oxidant-free) atmosphere.

[o]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.

[p]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% $Na_2S_2O_3$.

[q]Sample receiving no pH adjustment must be analyzed within 7 days of sampling.

Part II
Quality Assurance for Environmental Chemical Analyses

Prepared by
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ENVIRONMENTAL LABORATORY

US ARMY ENGINEER WATERWAYS EXPERIMENT STATION

QUALITY ASSURANCE PROGRAM FOR ENVIRONMENTAL CHEMICAL ANALYSES

1. PURPOSE AND SCOPE

The purpose of this Quality Assurance (QA) program is to ensure the scientific reliability and compatibility of water quality data at the Environmental Laboratory (EL), US Army Engineer Waterways Experiment Station (WES) by providing QA guidelines for program managers, principal investigators, and analysts in the development of and execution of projects.

Any project requiring sample collection, analysis, data reporting and interpretation must be susceptible to QA if it is to be scientifically valid. Obviously, Quality control (QC) measures are more readily applied to routine monitoring than to state-of-the-art research. However this does not prevent the application of QC procedures and it should be realized that different research projects will involve variations of the QA program to meet specific project needs. These requirements are best defined by the principal investigators. The role of the QA officer is to provide recommendations for appropriate and necessary QA methods and plans to coordinate this effort with the EL staff (managers, investigators, analysts, etc.).

The basic QC measures for analytical testing will be the same for both routine monitoring and Research and Development as long as standard analytical methods are in use. When new procedures must be developed due to matrix interferences, or the the Corps of Engineers (CE) improves of develops a new analytical procedure, these variances

must be validated through extensive testing to assure their reliability. The QA officer will be responsible for this validation to maintain the integrity of EL data.

As the CE lead agency for Civil Works Environmental Quality Research, the WES and EL are obligated to support quality assurance if documentable and reliable research results are to be generated. Specific quality control measures must be developed for each project.

2. References

a. ER 1110-1-8100

b. ER 1110-1-261

c. ER 1110-1-263

d. ER 1110-2-244

e. Handbook for Analytical Quality Control in Water and Wastewater Laboratories. EPA 600/4-79-019, March 1979, US Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, OH 45268.

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Environmental Laboratory, US Army Engineer Waterways Experiment Station, P.O. Box 631, Vicksburg, MS 39180

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m. "Guidance for Contracting Biological and Chemical Engineering Evaluations of Dredged Material", Environmental Research and Simulation Division, Environmental Laboratory, US Army Engineer Waterways Experiment Station, Vicksburg, MS

n. Test Methods for Evaluating Solid Waste, SW-846, Third Edition November 1986, US Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington, D.C. 20460

3. Background

QA is defined as the sum of activities that document and maintain the quality of monitoring data and quality control (QC) is the routine application of procedures to control the measurement process. The objectives of the WES QA program are to assure that the data generated are scientifically sound, defensible, continuously precise and accurate, and to enhance the overall capability and performance of the laboratory. References 2a-2d establish water quality policy for the

CE. In Addition to its function as a major research and developmen center for the Corps of Engineers, the WES also serves the Lowe Mississippi Valley Division as Division Laboratory and is responsibl for ensuring the application of quality control measures.

Water and wastewater monitoring data are collected in response t various Federal regulations and research projects. Included in thes regulations:

a. The National Environmental Policy Act (NEPA) of 1969

b. The Federal Water Pollution Control Act (FWPCA) of 1972

c. The Marine Protection Research and Sanctuaries Act (MPRSA) of 1972

d. The Coastal Zone Management Act of 1972

e. The Safe Drinking Water Act of 1974 and 1986

f. The Toxic Substances Control Act of 1976

g. The Resource Conservation and Recovery Act of 1976

h. The Clean Water Act of 1977

i. The Comprehensive Environmental Response, Compensation and Liability Act of 1980 (CERCLA or Superfund)

j. The Defense Environmental Restoration Program

k. Superfund Amendments and Reauthorization Act of 1986

Much of the preceding legislation requires monitoring programs that fall under the regulatory authority of the Environmental Protection Agency (EPA). On 30 May 1979, the EPA administrator issued the following policy statement, "I am making participation in the Quality Assurance effort mandatory for all EPA supported or required monitoring activities...For the purpose of this policy statement monitoring is defined as all environmentally related measurements which are funded by the EPA or which generate data mandated by the EPA." This program does not attempt to describe in detail all of the QC measures to be applied, but is intended to serve as a general guide for projects in developing specific policies, organizations, objectives, and QC activities to achieve data quality goals. References 2d, 2e, 2f, and 2g provide detailed procedures to be followed in water quality laboratories.

These regulations may also precipitate research projects that supply data for standards setting, environmental assessments, permit applications, etc. The program classification response to government regulation or research project will determine the types and numbers of samples to be collected and the parameters to be measured (physical, chemical, biological, radiological). The minimum precision and accuracy requirements will be determined by the end use of the data. These factors together will determine the level of QA effort.

4. Quality Assurance Goals

QA goals have been established to:

a. Provide an organizational structure defining the basic concepts of QA at WES in the Environmental Laboratory.

b. Establish guidelines to assist project officers and program managers in the logical development of general and specific QA plans for projects.

c. Provide a means for evaluating projects as to appropriate data requirements.

d. Implement a procedure to review data quality aspects of projects.

e. Encourage the use and development of methods of analysis and data treatment that are capable of meeting the data quality or research quality needs required by the project goals-to assure the use for which the data are intended.

f. Monitor the operational performance through appropriate intralaboratory and interlaboratory QC programs.

g. Ensure that program and project officers and contractors develop protocols with approved QA plans and procedures prior to program initiation and that they adhere to them.

h. Identify data quality problem areas and alert management to them.

5. Organization and Responsibility

The quality of data collected by the Environmental Laboratory lies ultimately with the Chief. In order to provide an organizational structure for QA, a QA officer will be designated to develop, coordinate, and direct these

activities. QC must be built into a laboratory program to such an extent that it is a routine part of all other laboratory activities. Management Support for QA program must be visible and active. The QA officer shall be responsible for coordinating a definition of the quality of data required to meet program objectives. Principal investigators or project managers will work with the QA officer to implement the QA plan and assure the documentation of QC measures. The WES functional management structure is shown in Figure 1 - the QA officer being a part of the Environmental Engineering Division with responsibility to the Chief, EL.

6. Quality Assurance

Quality assurance is maintained through the application of QC measures to all phases of the analytical process--from the initial planning and selection of sampling sites to the final interpretation and reporting of data. A brief description of these QC procedures follows:

6.1 Sample Collection

The major objective of a sampling program is to obtain a representative portion of the environment under investigation. Criteria for surface water sampling may be determined by (1) permitting, (2) area wide or basin planning, (3) compliance, (4) enforcement, (5) experimental design, (6) process control, and (7) research and development. They are often tied to the physiographic features of the area under consideration. Groundwater monitoring is usually performed in the vicinity of dredged material solid waste disposal facilities. Factors to be considered in sample collection for monitoring programs are:

WATERWAYS EXPERIMENT STATION
EXECUTIVE OFFICE

ENVIRONMENTAL LABORATORY

CONTINUED FROM PAGE 12

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1 MATHEMATICIAN	GS-11
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1 MECHANICAL ENGINEER	GS-11
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1 PHYSICAL SCIENTIST (V)	GS 11
1 BIOLOGIST	GS-11
1 BIOLOGIST	GS 09
2 MATHEMATICIAN	GS-09
1 ELECTRONICS ENGINEER	GS-09
1 CIVIL ENGINEER	GS-09
1 ELECTRONICS ENGINEER	GS-07
1 CIVIL ENGRG TECH	GS-07
1 ENGRG TECH	GS-04
1 SECRETARY (TYPING)	GS-04
1 ENGRG AID	GS-03

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1 RES BIOLOGIST	GS-13
1 SOIL SCIENTIST	GS-13
1 PHYSICAL SCIENTIST	GS-13
1 AQUATIC BIOLOGIST	GS-13
2 RES BIOLOGIST	GS-12
1 ZOOLOGIST	GS-12
1 PHYSICAL SCIENTIST	GS-12
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1 RES BIOLOGIST	GS-14
1 RES LIMNOLOGIST	GS 14
1 RES CHEMIST	GS-13
1 RES MICROBIOLOGIST	GS-13
1 AQUATIC BIOLOGIST	GS-13
2 RES BIOLOGIST	GS 12
3 HYDROLOGIST	GS-12
1 PLANT PHYSIOLOGIST	GS-11
1 ECOLOGIST	GS-11
2 PHYSICAL SCIENTIST	GS-11
2 BIOLOGIST	GS-11
1 BIOLOGIST	GS-09
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228 GS GM
2 OFFICERS

1 FEBRUARY 1989

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1 RES AQUATIC BIOLOGIST	GS-13
1 RES BIOLOGIST	GS-13
1 BOTANIST	GS-12
1 ECOLOGIST	GS-12
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2 WILDLIFE BIOLOGIST	GS-12
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4 ENVIRON ENGINEER	GS-12
1 RES CIVIL ENGINEER	GS-12
2 CIVIL ENGINEER	GS-11
1 ENVIRON PROTECTION SPEC	GS-11
1 CIVIL ENGINEER	GS-09
1 CHEMICAL ENGINEER	GS 07
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2 RES CIVIL ENGINEER	GS-13
1 CIVIL ENGINEER	GS-12
1 HYDROLOGIST	GS-12
1 MATHEMATICIAN	GS-11
3 CIVIL ENGINEER	GS-11
1 CIVIL ENGINEER	GS-11
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3 CHEMIST	GS-11
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1 CHEMIST	GS-09
2 PHYSICAL SCI TECH	(1 ON CALL) GS-06

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ECOSYSTEM RESEARCH AND SIMULATION DIVISION

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1 FEBRUARY 1983

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LTC JACK R STEPHENS, EXT 2582 — DEPUTY COMMANDER & DIRECTOR, CEWES-ZB
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WES GRADUATE INSTITUTE
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SPECIAL ASSISTANTS
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COMMITTEES
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[illegible] HANDA, EXT [illegible] — ASST CHIEF, CEWES HV A

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[illegible], EXT [illegible] — CHIEF, CEWES [illegible]

WATERWAYS DIVISION
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DWAYNE G LEE
COLONEL EN
COMMANDER & DIRECTOR

[illegible]

a. Site Selection

The selection of sampling sites is critical in obtaining a representative sample. Some areas of consideration are study objectives, accessibility, physical characteristics, inflow, sampling personnel, and facilities.

b. Sample Type Frequency

The primary water sample types are grab samples and composite samples. Composite samples may be conducted manually or automatically and depend on either a continuous or periodic sampling mode using: (1) constant pumping rates, (2) sample pumping rates proportioned to streamflow rates for surface water, (3) constant sample volumes at constant time intervals between samples, (4) constant time intervals between samples and sample volumes proportioned to total streamflow rates, and (5) constant time intervals between samples and sample volume proportioned to total streamflow rates at time of sampling. In many cases automatic samplers help eliminate the element of human error associated with manual sampling; however, automatic samples are susceptible to mechanical problems and should be subject to strict maintenance checks and calibration procedures. Protocol for sampling sediments is given in reference 2h.

c. Flow Measurement (surface water)

Accurate flow measurement is a necessary part of any water or wastewater survey and is required under the National Pollutant Discharge Elimination System. Flow rates are divided into four categories: (1) flow measurement in completely filled pipes under pressure (Venturi

tubes, flow nozzles, Pitot tubes, magnetic flowmeters, etc.), (2) trajectory methods measured at the end of the pipe (California and Purdue Pipe Methods), (3) flow measurement in open channels and sewers (velocity area measurement time or passage measurement or weir and flume level measurements, and (4) miscellaneous flow methods (Manning formula, tracer and salt dilution technique, water meters, pump rates, etc.). A written step-by-step procedure for the use of the sampler shall be available for the personnel involved in sample collection.

d. Sample Handling and Preservation

Proper sample containers and preservation techniques must be employed to maintain the integrity of the samples. Some determinations such as temperature, pH, specific conductance, and dissolved gases should be made in the field. A list of recommended sample containers, preservatives, and holding times for the various analytical parameters are given in Table 1. Instructions for cleaning sample containers are as follows:

(1) Organics: Chromic acid—wash 15 min, rinse thoroughly with tap water, distilled water, and several times with redistilled acetone, hexane petroleum ether, or chloroform, and clean lids and liners in the same manner and store both in a sealed container until needed at the sampling site.

(2) Metals: Soak in 1:1 AR grade nitric acid for 12 hr, then rinse thoroughly with three to five successive portions of high-quality

Table 1

WATER SAMPLES

SAMPLE TYPE	CONTAINER	PRESERVATIVE	HOLDING TIME
Boron[2/]	½ gal. plastic	Refrigeration, 4°C	24 hrs.
Fluoride[2/]	½ gal. plastic	Refrigeration, 4°C	7 days
Settleable Solids[2/]	½ gal. plastic	Refrigeration, 4°C	24 hrs.
Solids, Total & Suspended, etc.[1/]	1 pint plastic	Refrigeration, 4°C	7 days
TKN[2/]	1 pint plastic	Refrigeration, 4°C	24 hrs.
Sulfate[2/]	1 pint plastic	Refrigeration, 4°C	7 days
Chloride[2/]	1 pint plastic	Refrigeration, 4°C	7 days
Color[2/]	1 pint plastic	Refrigeration, 4°C	24 hrs.
Turbidity[2/]	1 pint plastic	Refrigeration, 4°C	7 days
Alkalinity[2/]	1 pint plastic	Refrigeration, 4°C	24 hrs.
Acidity[2/]	1 pint plastic	Refrigeration, 4°C	24 hrs.
pH[2/]	1 pint plastic	Refrigeration, 4°C	6 hrs.
Conductivity[2/]	1 pint plastic	Refrigeration, 4°C	24 hrs.
Chromium, Hexavalent[1/]	1 pint plastic	Refrigeration, 4°C	48 hrs.
NH_3, TKN, NO_3-NO_2, T-P, COD, TOC[2/]	1 pint plastic	Refrigerate, 4°C; H_2SO_4 pH<2	24 hrs. COD (7 Days)
Total Phosphorus, Dissolved[2/]	1 pint plastic	Filter-on-site, Refrigerate, 4°C; H_2SO_4 pH<2	24 hrs.
Ortho-Phosphate	1 pint plastic	Filter-on-site, Refrigerate, 4°C;	24 hrs.
Oil and Grease[3/]	1 qt. glass w/ Teflon Liner	Refrigeration, 4°C H_2SO_4 pH<2	24 hrs.
Pesticides	1 qt. glass w/ Teflon Liner	Refrigeration, 4°C	30 days[6/]
Specific Organics (NPDES Permit)	1 qt. glass w/ Teflon Liner	Refrigeration, 4°C	30 days[6/]
General Organics	1 gal. glass (amber) w/ Teflon Liner	Refrigeration, 4°C	30 days[6/]
Volatile Organics (VOA)	40 ml vials w/Septum caps	Refrigeration, 4°C	14 days
Sulfides[3/]	1 pint plastic	2 ml Zinc Acetate, Refrigerate	24 hrs.
Cyanides[3/]	½ gal. plastic	NaOH pH 12, Refrig.	24 hrs.
Metals[3/4/]	1 pint plastic	HNO_3 pH<2	6 months
Mercury[3/4/]	1 pint glass	HNO_3 pH<2	38 days
Bacteriological	250 ml glass, sterilized	Refrigeration, 4°C	8 hrs.
Phenols[3/]	1 pint glass	0.5 g $CuSO_4$, H_3PO_4 pH<4, Refrigerate	24 hrs.

SEDIMENT SAMPLES

Organics[5/]	1 qt. glass w/ Teflon Liner	Refrigerate, 4°C	ASAP[7/]
Metals[5/]	1 pint plastic	Refrigerate, 4°C	6 months

1/ - If all or part of these analyses are required on the same sample, a one gallon plastic bottle should be used.
2/ - Must be preserved in field if samples are not returned to lab within 24 hours.
3/ - Must be preserved in field at time of collection.
4/ - If a sample is collected for mercury and other metals, only a mercury bottle should be collected.
5/ - If metals and organics are to be analyzed on the sediment, collect a 1 quart glass container only.
6/ - Samples must be extracted within 7 days and analyzed within 30 days.
7/ - "As soon as possible."

distilled water. Seal containers and enclose in a sealed container until needed at the sampling site.

e. Chain-of-Custody

If samples are to be used for compliance purposes, a chain-of-custody procedure must be employed and must begin in the field. This will ensure that the samples are collected, transferred, stored, analyzed, and destroyed only by authorized personnel. The following chain-of-custody procedures are generally considered to be acceptable:

(1) A written procedure is provided to the field and laboratory personnel to assure that sample possession is traceable.

(2) Involve a minimum number of trained persons in sample collection and handling.

(3) Provide guidelines to be followed for sample collection, including number of samples, sampling method to be used, preservation, and handling

(3) Provide guidelines to be followed for sample collection, including number of samples, sampling method to be used, preservation, and handling.

(4) At the time the sample is collected a chain-of-custody tag (Figure 2) with tamperproof seal (Figure 3) is attached to the

CHAIN OF CUSTODY

U. S. ARMY CORPS OF ENGINEERS
WATERWAYS EXPERIMENT STATION
P.O. DRAWER 631
VICKSBURG, MS 39180

United States Army
Corps of Engineers
...Serving the Army
...Serving the Nation

U.S. ARMY CORPS OF ENGINEERS

Station No.	Date	Time	Sequence No.
Station Location			____ Grab ____

____ BOD
____ Solids
____ COD
____ Nutrients
____ Metals
____ Oil & Grease
____ D.O.
____ Bact.
____ Other

Remarks/Preservative:

Samplers:

Serial Tag No. ____

Figure 2. Example of chain-of-custody sample tag. (a) Front. (b) Back.

U.S. ARMY CORPS OF ENGINEERS
OFFICIAL SAMPLE SEAL

Sample No. Date
Signature
Print Name and Title (Inspector, Analyst, Tech.)

SEAL BROKEN BY

Figure 3. Example of tamper-proof seal.

sample. Information on the tage should be written legibly with waterproof ink. A chain-of-custody record (Figure 4) should accompany the samples.

(5) Bound field notebooks are used to record field measurements and information necessary to reconstruct the sample collection process.

f. QA in the Field

Procedures and equipment shall meet project requirements. The QA coordinator shall be provided documentation to:

(1) Assure approved methodology.

(2) Assure the percentage of QC analyses and results.

(3) Validate the procedures used for recording, processing, and reporting data.

(4) Ensure the calibration and maintenance of field instruments.

(5) Ensure qualifications of field personnel.

For each project type a minimum of seven sets of split samples, spiked samples, and sample preservation blanks shall be collected to define acceptable estimates of precision and accuracy criteria for data validation.

CHAIN OF CUSTODY RECORD

PROJECT NAME

SAMPLERS: (Signature)

DATE	TIME	SAMPLE ID	NO. OF CONTAINERS							REMARKS

Relinquished by: (Signature)	Date /Time	Received by: (Signature)	Relinquished by: (Signature)	Date /Time	Received by: (Signature)
Relinquished by: (Signature)	Date /Time	Received by: (Signature)	Relinquished by: (Signature)	Date /Time	Received by: (Signature)
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6.2 Facilities

Laboratory facilities must include sufficient space to process and examine the samples for the required parameters during peak work loads (ν 200 ft^2 and 15 linear ft of bench space per analyst). Storage space must also be provided for samples, glassware, equipment, and refrigeration.

Hot and cold running water, high capacity sinks and drains capable of accepting acid waste, distilled/deionized water, and ultra-pure water for trace analyses should be readily available. Provisions should be made for adequate electrical outlets and vacuum systems.

Exhaust hoods capable of venting organic solvents and acid fumes should be provided as well as laminar flow hoods for trace analyses and carcinogens and special purpose hoods for perchloric acid.

Cleanliness in the laboratory should be emphasized to reduce possible contamination.

Safety features to comply with Federal Occupational Safety and Health Administration regulations should include fire extinguishers, safety showers, eyewash stations, mandatory eye protection requirements, first aid equipment, protective garments, chemical dispensing devices, and safety education.

6.3 Laboratory Personnel

QA can only be as good as the personnel involved in the analysis. Educational and/or experience requirements are determined by the nature and complexity of the analysis. A skill-time rating for some standard analytical operations is given in Table 2.

Table 2*

SKILL-TIME RATING OF STANDARD ANALYTICAL OPERATIONS

Measurement	Skill Required (Rating No.)[1]	Number[2] Per Day
Simple Instrumental		
pH	1	100-125
Conductivity	1	100-125
Turbicity	1	75-100
Color	1	60-75
Dissolved Oxygen (Probe)	1,2	100-125
Fluroide (Probe)	1,2	100-125
Simple Volumetric:		
Alkalinity (Potentiometric)	1	50-75
Acidity (Potentiometric)	1	50-75
Chloride	1	100-125
Hardness	1	100-125
Dissolved Oxygen (Winkler)	1,2	75-100
Simple Gravemetric:		
Solids, Suspended	1,2	20-25
Solids, Dissolved	1,2	20-25
Solids, Total	1,2	25-30
Solids, Volatile	1,2	25-30
Simple Colorimetric:		
Nitrate N (Manual)	2	75-100
Nitrate N (Manual)	2	40-50.
Sulfate (Turbidimetric)	2	70-80
Silica	2	70-80
Arsenic	2,3	20-30
Complex, Volumetric, or Colorimetric:		
BOD	2,3	[2]15-20
COD	2,3	25-30
TIN	2,3	25-30
Ammonia	2,3	25-30

(Continued)

[1]Skill-required rating numbers are defined as follows:

1-aide who is a semiskilled subprofessional with minimum background or training, comparable to GS-3 through GS-5.

2-aide with special training or professional with minimum training with background in general laboratory techniques and some knowledge of chemistry, compatable to GS-5 through GS-7.

3-experienced analyst capable of following complex procedures with good background in analytical techniques, professional, compatable to GS-9 through GS-12.

4-experienced analyst specialized in highly complex procedures, professional, comparable to GS-11 through GS-13.

[2]Rate depends on type of samples.

Table 2 (Continued)*

Measurement	Skill Required (Rating No.)[1]	Number[2] Per Day
Phenol (Distillation Included)	2,3	20-30
Oil and Grease	2,3	25-30
Fluoride (Distillation Included)	2,3	25-30
Cyanide	2,3	8-10
Special Instrumental:		
TOC	2,3	75-100
Metals (by AA), No Preliminary Treatment	2,3	150
Metals (by AA), With Preliminary Treatment	2,3	60-80
Organics (by GC), Pesticides Without Cleanup	3,4	3-4
Organics (by GC), Pesticides, With Cleanup	3,4	2-4

*Source, U. S. Environmental Protection Agency, 1979, Handbook for Analytical Quality Control in Water and Wastewater Laboratories, EPA-600/4-79-019.

The time limits presented in Table 2 are based on use of approved methodology. A tacit assumption has been made that multiple analytical units are available for measurements requiring special equipment, as for cyanides, phenols, ammonia, nitrogen, and COD. For some of the simple instrumental or simple volumetric measurements, it is assumed that other operations such as filtration, dilution, or duplicate readings are required; in such cases the number of measurements performed per day may appear to be fewer than one would normally anticipate.

Training is an essential part of any program measuring water quality. For more experienced, higher grade personnel, formal training in special fields, possibly leading to specialization will be a part of each individual's career development plan. Such training is fostered through local institutions and through the training courses developed by the Corps, EPA, and other government agencies.

Formalized training for lower grade personnel, comparable to GS-3 to GS-5, is relatively scarce. However, skills can be most efficiently improved at the bench level on a personal informal basis by more experienced analysts working in the same area. Exposure of personnel to pertinent literature will also be a definite program policy.

Personnel will be indoctrinated into QA procedures and their importance to the Corps. Laboratory supervisors will periodically review basic techniques and policies with the analysts. Recognition of excellent performance

as well as tactful assessment of areas needing improvement will aid in overall laboratory performance.

6.4 Instrumentation

Instrumentation is an integral part of all water and wastewater laboratories and is constantly being improved and upgraded. "State of the art" equipment and practices are essential if laboratories are to obtain data that will meet the accuracy, precision, and sensitivity required by today's monitoring programs. A routine maintenance and calibration program will be provided and documented. Table 3 presents calibration recommendations for some of the major instruments in the laboratory. Assignment of responsibility will be clearly defined and records will be maintained in a bound notebook or equivalent permanent records management file. Some considerations to be made when planning the purchase of new equipment are:

a. Is there a need, present or future for the item, i.e., does present or projected regulations specify tests that this equipment will be used for?

b. Does the purchaser have equipment in-house that can be modified or adapted to perform the necessary function at a lesser cost?

c. Will the purchaser have the necessary auxiliary input, e.g., if G.C. - Mass spectroscopy unit is requested, will library facilities be available?

d. Are there technically competent personnel available to operate the equipment? If not, what plans are available for hiring or training such personnel?

6.5 Methodology

Analytical methods for routine environmental testing of water samples should be selected using the following criteria:

a. The selected methods should measure desired constituents of water samples in the presence of normal interferences with sufficient precision and accuracy to meet the water data needs.

b. The selected procedures should use equipment and skills ordinarily available in the average water pollution control laboratory or water supply laboratory.

c. The selected methods should be sufficiently tested to have established their validity.

d. The selected methods should be sufficiently rapid to permit repetitive routine use in the examination of large numbers of water samples.

More complex matrices such as those encountered in sediment, soil and plant tissue may require extensive sample preparation and variance in procedures to compensate for analytical interferences. Procedural development is

TABLE 3. INSTRUMENT CALIBRATIONS*

Instrument	Procedure	Frequency
1) Analytical Balances	(a) Zero	Before each use
	(b) Standard weights	Monthly
	(c) Full adjustment adjustment	Annually
2) pH Meters	At pH 4,7, and 10	Daily
3) Conductivity Meters	(a) Obtain cell constant with potassium chloride reference solutions	Daily
	(b) Construct temperature curve if measurements are to be made other than at 25 ± 0.5°	Monthly
4) Nephelometer/ Turbidimeters	(a) Check instrument scales or develop calibration curve with formazin stds (≤40NTU)	Monthly
	(b) If manufacturer's stds are not formazine, check against formazine stds. (≤40NTU)	Annually
5) Colorimeters/Filter Photometers	Curves determined with 5-6 laboratory-prepared std. solutions for each parameter in conc. range of samples	Daily
6) UV/Visible Specrophotmeters	(a) Wavelength calibration with holmium oxide glass or solution, low-pressure mercury arc, benzene vapor (UV), or hydrogen arc (visible)	Quarterly
	(b) Absorbance vs. concentration curves with 5-6 std. Solutions for each parameter at analytical wavelength in conc. range of samples	Daily
	(c) Full servicing and adjustment	Annually

* Source, U. S. Environmental Protection Agency, 1978, "Procedures for the Evaluation of Environmental Monitoring Laboratories," EPA-600/4-78-017.

TABLE 3. (Continued)

Instrument	Procedure	Frequency
7) Infrared Spectrophotometers	(a) Wavelength calibration with polystyrene or indene	Daily
	(b) Absorbance vs. concentration curves with 5-6 std. solutions for each parameter at analytical wavelength in conc. range of samples	Daily
	(c) Full servicing and adjustment	Semi-annually
8) Atomic Absorption Spectrophotmeters	(a) Response vs. concentration curves with 6-8 std. solutions for each metal (std. mixtures are acceptable, but wih same acid as samples to be run) in conc. range of samples	Daily
	(b) Full servicing and adjustment	Annually
9) Carbon Analyzers	Curves determined with 5-6 std. solutions in conc. range of samples	Daily
10) DO Meters	Calibrated against modified Winkler method on aerated distilled or tap water	Daily
11) Other Selective Ion Electrodes and Electromenters	Curves determined with 5-6 std. solutions in conc. range of samples	Daily
12) Thermometers	Calibrate in constant temperature baths at two temperatures against precision thermometers certified by NBS	Quarterly
13) Technicon Auto Analyzers	(a) Curves determined with std. solutions for each parameter	Each set of samples
	(b) Full service and adjustment (esp. colorimeter)	Annually

TABLE 3. (Continued)

Instrument	Procedure	Frequency
14) Gas Chromatographs	(a) Retention times and detector response checked with std. solutions	Daily
	(b) Response curves for each parameter determined with std. solutions	Monthly
15) Radiological Equipment	(See Standard Methods, Sect. 300)	

frequently necessary for samples of this type. Whenever possible a standard reference material in a similar matrix should be analyzed with the sample to assure method reliability.

When Federal regulations must be met, such as for Section 304(h) of Public Law 92-500 and for the Interim Primary Drinking Water Regulations, EPA approved methods will be used. The WES QA officer will coordinate any variances. Methodology for implementation of Section 404(b)(1) of PL 92-500 is provided in Reference 2h.

Methodologies must be carefully documented and readily available to each analyst. Laboratory inspections will be made to assure adherence to the written procedure. Method documentation will include the following items for QA review:

a. Parameter that the method measures.

b. Principle - A brief description of the method.

c. Optimum Concentration Range - The analytical range from the lowest concentration to the highest concentration in which a substance is measured. The sample may be concentrated or diluted so that the substance can be detected within this range.

d. Sensitivity - The slope of a curve of concentration versus instrument response (such as absorbance).

e. Detection Limit - The lowest quantity which may be distinguished from background with an acceptable degree of confidence.

f. Reference - The source of the analytical method. In addition all variances of the original procedure are documented here.

g. Matrix - The general composition of the sample that the method is capable of handling, e.g., water (potable, ambient, wastewater), solids (leachates, sediments, sludges), air (filter particulates, bubbler solutions, casette trap), fluids (solvents, hydrocarbons, oils).

h. Analysis Procedure

(1) Description - The analytical procedure is described for normal conditions. Sample pretreatment (if required) and preparation protocols are also described here. The language used to describe the method is to be detailed enough (cook-book fashion) so that a technician with experience in the respective type of analysis would clearly understand every step of the procedure. Analytical techniques that employ a great deal of instrumentation such as atomic absorption and automated analyzers are briefly described since instrument manuals are available which detail the use of the instrument. However, auto analyzer manifolds are to be depicted.

(2) Instrument Parameters - A description of the instrument and all the instrument settings that are necessary to set up the instrument for normal conditions.

(3) Routine Performance Tests - A test of the instrument performance which is separate from a calibration procedure and is a gross indication of the instrument's response. This test is performed and documented each time a batch of samples is processed or else on a daily basis. The frequency chosen for instrument response check is dependent on the analyst's confidence of instrument stability.

(4) Calibration Standards - The calibration standards are described in terms of the range of concentrations used in the normal procedure and in terms of composition (preparation of standard solutions) employed for various matrices.

6.6 Reagents

Reagents are available in a wide range of grades; the purity requirement varies with the type of analysis being conducted. For many analyses American Chemical Society (ACS) reagent-grade quality is acceptable, and use of reagents of lesser purity should be prohibited. Where special high-purity grades, such as spectro grade or reference grade, are required, the analytical

procedure should specify the special grade or the required in-house purification, such as distillation or recrystallization.

Upon receipt, a reagent should be labeled with the date of receipt, who received it, and an expiration date if it is unstable. When reagent quality is critical and variation is expected in the quality by reagent lot or source, initial testing of the reagent may be advisable before placing it in laboratory stores.

An effective inventory control program should be used to assure that reagent stocks are rotated, out-of-date reagents are discarded, and necessary stocks of reagents are maintained. Reagents must be stored in accordance with manufacturer's directions to assure reagent integrity, such as storage of light-sensitive reagents in dark bottles, protection from excessive heat, and so on.

For most reagents, verification of reagent suitabilty is conducted by analyzing a reagent blank with the series of analyses. Charting of the reagent blank determinations may be advisable.

Distilled or deionized water should be used in all analyses and for final rinsing of glassware. As a minimum, laboratory water should meet American Society for Testing and Materials (ASTM) specifications for either Type I or Type II reagent water (see Table 4). Type I reagent water is filtered deionized water and is the most common grade of water used in industrial hygiene laboratories. Type II reagent water is distilled water (usually double

TABLE 4. AMERICAN SOCIETY FOR TESTING AND MATERIALS SPECIFICATIONS FOR REAGENT WATER[a]

	Type I	Type II	Type III	Type IV
Total matter, maximum mg/l	0.1	0.1	1.0	2.0
Electrical conductivity, maximum micromho/cm at 25°C	0.06	1.0	1.0	5.0
Electrical resistivity, minimum MΩ • cm at 25°C	16.66	1.0	1.0	0.20
pH at 25°C			6.2-7.5	5.0-8.0
Minimum color retention time (min)	60	60	10	10

[a]Source: American Society for Testing and Materials[67]

distilled) and is used for most analytical methods requiring organic-free water.

Laboratory water should be periodically tested for specific conductance, and corrective actions should be taken as indicated. The analyst should be aware of possible sources of contamination from leaching, especially when water is stored for extended periods of time after deionization or distillation.

6.7 Analytical Performance

Valid precision and accuracy data must be maintained for each method and analyst by initial and systematic checks. Generally, control charts such as those developed by Dr. Walter A. Shewhart are used to show standard deviations (precision) and ranges (accuracy). Details for preparing these charts are presented in Appendix A and are taken from EPA's Handbook for Analytical Quality Control in Water and Wastewater Laboratories. When either the

(2) Participate in performance evaluation and method studies as available from EPA (audit-sample program), American Standard for Testing and Materials (ASTM), and other agencies.

Performance checks should be computerized wherever possible to facilitate evaluation.

6.8 Data Handling and Reporting

After the laboratory has established control over sample collection, instrumentation, and analytical methods, the data must be handled and reported in order to be useful and accurate. Prescribed procedures must be established for controlling the passage of samples through the laboratory.

a. Analytical Process

(1) Samples are recorded in a logbook upon arrival and assigned a number. All descriptive information concerning the sample, source, quantity, collection time, and sample collector is entered. This information ~~may~~ is also [redacted] entered into a computer data storage and retrieval system.

(2) An analytical request sheet ~~(or IBM card)~~ is prepared which lists the sample number, descriptive information, and all necessary pretreatments and analyses. Space is provided for completion

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dates. ~~If information is computerized, a~~ flag date for expected completion time ~~may~~ is also be added.

(3) As results are completed, the name of the analyst, calculation, and all pertinent data are noted on the results sheets (which may be attached to the request sheets or made as part of the request sheet).

(4) After all analyses are completed, the results are sent to the laboratory supervisor or person responsible for reporting results. After review, they are put into final report form which may be a typed copy ~~or~~ computer output, or floppy disk.

(5) Analysis request forms and results are filed for future reference.

b. Data Control

(1) The proper use of significant figures should be emphasized.

(2) Data should be rounded properly.

(3) Bound books should be used at the bench to maintain permanent records.

(4) Care must be taken to avoid transfer errors when entering data ~~onto cards or~~ into a computer.

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(5) Automation of instrumentation in many instances will eliminate errors in the transfer process and speed up data availability.

A flow chart showing the sequence of events in the laboratory is presented in Figure 5.

If sample data are to be used for compliance or regulatory purposes, a chain of custody record such as that shown in Figure 6 may be used to accompany the sample.

6.9 Standards

Data generated are only as good as the standards used for instrument and method calibrations. All measurements or calibrations shall be traceable directly or indirectly to an acceptable reference standard. Both EPA and NBS are excellent sources for these materials.

When standard solutions are prepared, records must be maintained to assure traceability of calibrations to the primary standard. For standard solutions that are prepared just prior to use, traceabilty is easily accomplished by including the standard solution preparation data in the analyst's log book. For previously prepared working or stock standard solutions that are available for use by several analysts, traceability can be accomplished by use of a standard solution log. The log book should contain raw data on solution preparation or a reference to the data's location (i.e., analyst's laboratory book number and page), as well as the information placed on the standard

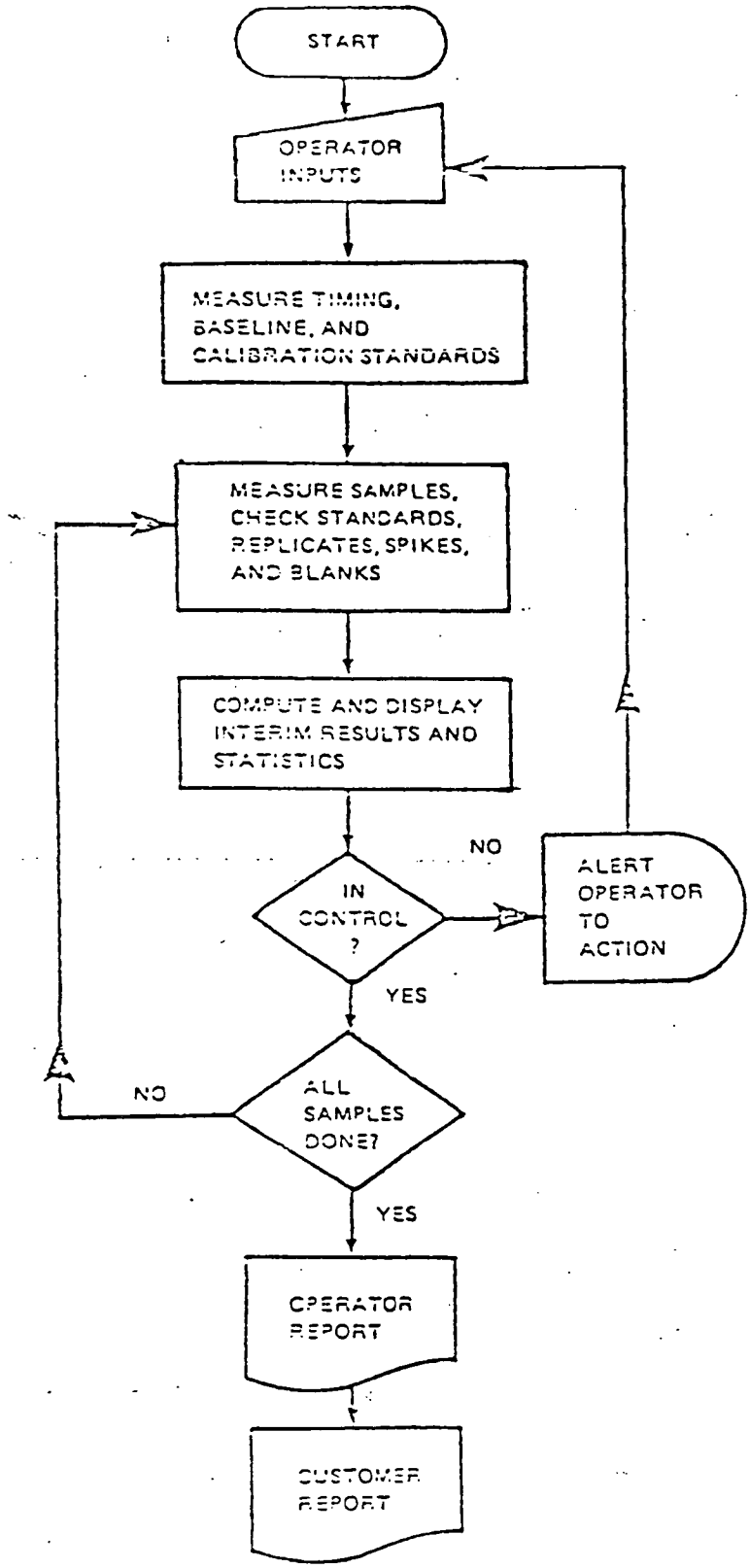

Figure 5. Flow chart of the sequence of events during a controlled series of laboratory measurements.

CHAIN OF CUSTODY RECORD

PROJECT NAME

SAMPLERS: (Signature)

DATE	TIME	SAMPLE ID	NO. OF CONTAINERS							REMARKS

Relinquished by: (Signature)	Date /Time	Received by: (Signature)	Relinquished by: (Signature)	Date /Time	Received by: (Signature)
Relinquished by: (Signature)	Date /Time	Received by: (Signature)	Relinquished by: (Signature)	Date /Time	Received by: (Signature)
Relinquished by: (Signature)	Date /Time	Received by: (Signature)	Date /Time	Remarks	

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Figure 6. Example of chain-of-custody record

solution label (i.e., identification, solution number, date prepared, expiration date, and analyst's initials). Traceability is completed by including the standard solution number in the analyst's laboratory book when the standard solution is used.

When standard solutions are stored for extended periods of time, verification of solution stability may be possible by requiring at least one fresh standard to be prepared at each time of use, or by comparing the standard solution against a freshly prepared solution at the end of the expiration dating period. Storage of dilute solutions or extended storage when less than one-fourth of the container is full should be avioded whenever possible.

6.10 Supervisory Control

Behind every good analytical laboratory is a good supervisor who maintains a continuing interest in the quality of data produced. A QA program provides the management mechanism to document precision, accuracy, completeness, and comparability of data. In this regard, the Quality Assurance Office will check on a weekly basis to review analytical laboratory operations and QC with analysts. Management will be provided with a status report on a regular (monthly) basis.

7.0 Contractor Laboratories

Quality Assurance begins with and is most directly controlled in the procurement process. If the contracting officer and project officer fail to procure high quality sampling and analytical services, the project is subject

to inferior data from the beginning. For this reason, all contracts, grants, and interagency agreements (IAG's) developed by EL primarily for the purpose of measuring chemical components in water and wastewater shall be subject to the following QA requirements:

a. All requests for Proposals (RFP's) grant and IAG application shall include a separate clause dealing with QA requirements.

b. As part of the selection process, technical evaluation criteria shall be applied to the proposed contractor QA program and the percentage of effort to be devoted to QA activities shall be specifically stated.

The IERL-CI laboratory of the EPA has put together a checklist for evaluation criteria to be used in rating quality assurance programs submitted as part of a contract proposal or grant application. These are given in Appendix B. Reference 21 also supplies excellent guidance for selecting a contractor for biological and chemical analyses.

The QA section of the Technical proposal shall address the following items:

(1) Goal of the QA program.

(2) Organization and responsibility of the QA program.

(3) Sample collection procedures including site selection, frequency, flow measurements, sample handling and preservation, chain of custody, and field analyses.

(4) Facilities, personnel, and equipment.

(5) Calibration procedures--standards and verification.

(6) Analytical procedures.

(7) Internal and external QC practices.

(8) Data handling and reporting.

(9) Documentation of QA practices.

(10) Percentage effort devoted to QA (cost and time).

c. Before selection and award of a contract, and at the option of WES, bidders may be required to show:

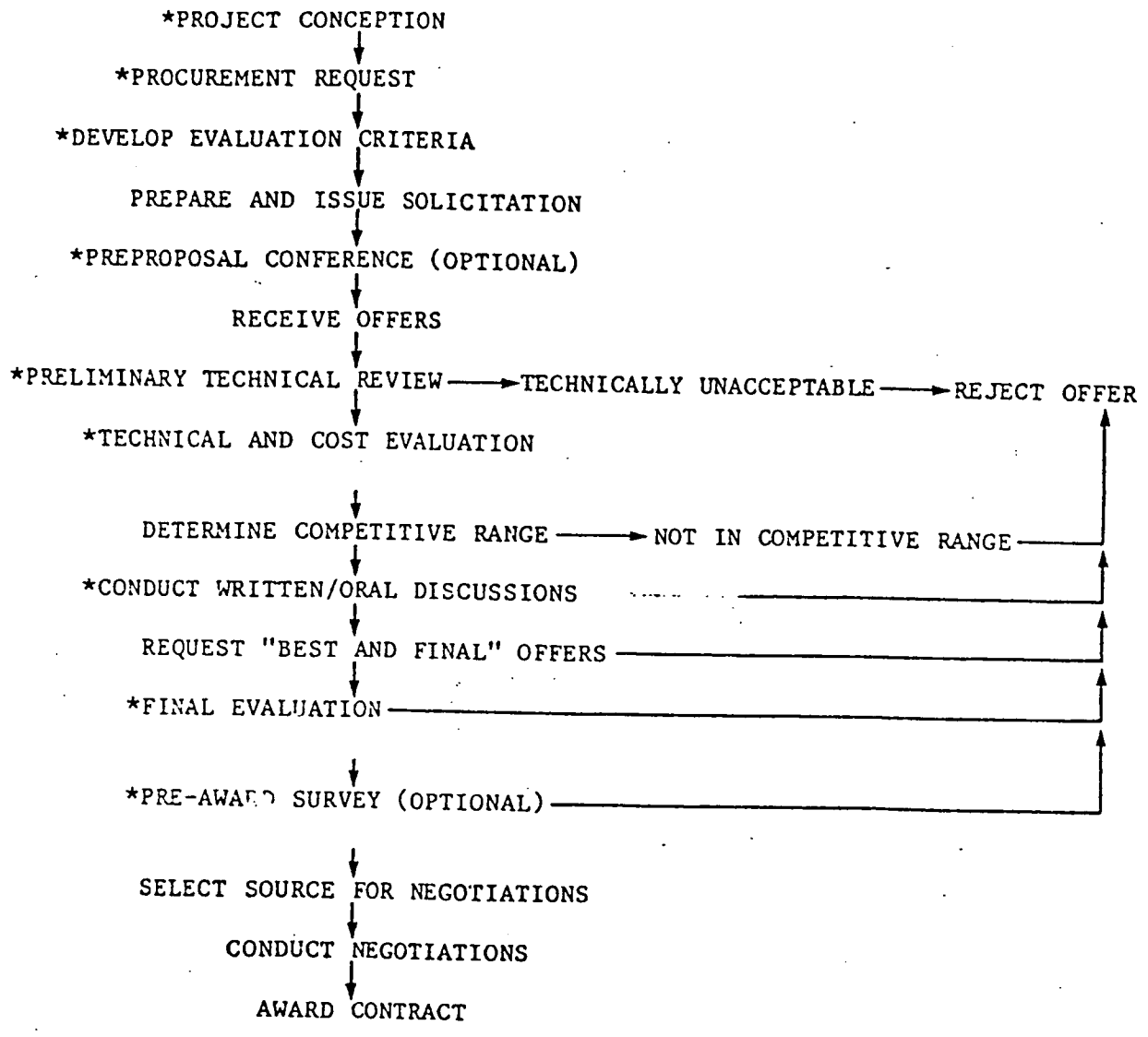
(1) Acceptable performance on audit samples.

(2) By on-site evaluation (laboratory inspection by WES) that manpower, equipment, and supplies necessary for completion of the project are available.

d. EL may also require that a specified number of split samples be analyzed by both the Contractor and EL.

Figure 7 shows the sequence of events in awarding a contract and the areas where quality assurance should be injected. It is the responsiblity of the WES project officer to see that QA requirements which are a part of the contract, grant, or IAG are met during the course of the monitoring project. Prior to contract awards the EL QA officer should be available to provide technical advice to the project officer and should work with him in the initial development of the contract, grant, or IAG. The QA program developed by the contract, grant, or IAG must be coordinated with the EL QA officer before award.

If the contractor laboratory performs analyses for projects generated by the Districts, an inspection is required prior to initiation of testing and at least every two years thereafter (Reference 2b). Guidance for these inspections is provided in Appendix C.



*QA considerations are important at these points in the process.

Figure 7. Processing sequence for contract source evaluation and selection

APPENDIX A

QUALITY CONTROL FOR ANALYTICAL PERFORMANCE

A.1 Introduction

Previous chapters discussed basic elements of quality control (QC) pertaining to laboratory services, instrumentation, glassware, reagents, solvents, and gases; the reader should refer to the appropriate sections to determine necessary specifications and requirements for QC. Assuming that these basic variables are under QC, that approved methods are being used, and that the complete ststem is initially under QC, valid precision and accuracy data must initially be developed for each method and analyst. Then, to insure that valid data continue to be produced, systematic daily checks must show that the test results remain reproducible, and that the methodology is actually measuring the quantity in each sample. In addition, QC must begin with sample collection and must not end until the resulting data have been reported. QC of analytical performance within the laboratory is thus but one vital link in the dissemination of valid data to the public. Understanding and conscientious use of QC among all field sampling personnel, analytical personnel, and management personnel is imperative. Technical approaches are discussed in the following sections.

A.2 The Industrial Approach to QC

In the 1920's, Dr. Walter A. Shewhart of Bell Telephone Laboratories, Inc. developed the theory of control charts as a basic method for evaluating the quality of products from manufacturing processes. His book (1) on statistical QC grew out of this original work. Later, acceptance of his concepts and related statistical techniques within industry led to refined, quantitative evaluations of product quality in manufacturing. Dr. Shewhart's work on production processes assumed a uniform product manufactured in large numbers and inspected

on a continuous basis through the periodic analysis of samples of n production units. The resulting data, x_1, x_2, ...x_n, were then used to estimate precision, as the standard deviation S or range R, and accuracy, as the arithmetic mean X. These statistics were calculated as follows:

$$S = \sqrt{\frac{\sum_{i=1}^{n} X_i^2 - \left(\sum_{i=1}^{n} X_i\right)^2 \Big/ n}{n - 1}}$$

R = the largest of the X_i - the smallest of the X_i

$$\overline{X} = \frac{\sum_{i=1}^{n} X_i}{n}$$

These statistics were evaluated by plotting them on control charts developed from similar statistics taken while the process was under properly controlled operation. The elements common to such control charts are represented in Figure A-1. They include an expected value (the central line) and an acceptable range of occurrence (the region between upper and lower control limits).

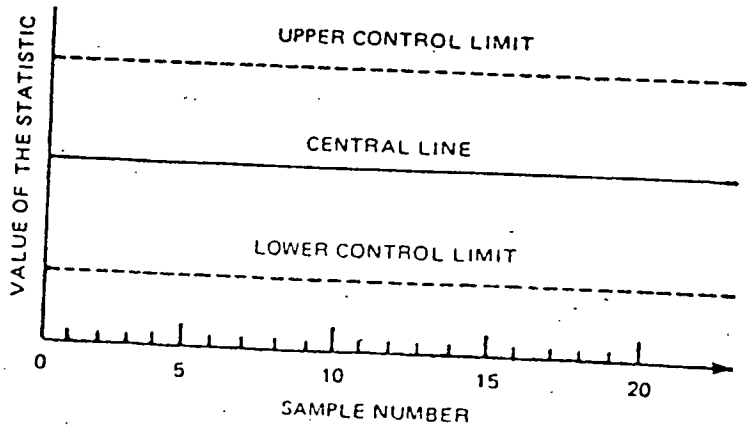


Figure A-1. Essentials of a control chart.

There are many reference sources available that discuss in great detail the classic Shewhart control charts and related statistics that have since been developed for specific industrial applications (2-4). In addition, many authors have discussed applications of a related type of control chart called a cumulative-summation (cusum) chart (2,4). Rather than evaluating each sample independently, the cusum chart evaluates the cumulative trend of the statistics from a series of samples. Because each successive point is based upon a cumulative data trend, cusum charts are often considered more effective than control charts in recognizing process changes and, therefore, may minimize losses from production of unacceptable units; however, cusum charts require the more difficult calculations, and optimally designed Shewhart techniques have been found to be almost as effective (2,4) so there is no universal agreement on the choice between them.

A.3 Applying Control Charts in Environmental Laboratories

In industrial applications, separate control charts are recommended for each product, each machine, and each operator. Analogous system variables in an environmental laboratory are the parameter, the instrument, and the analyst. However, environmental laboratories routinely have to contend with a variable that has no industrial counterpart--the true concentration level of the investigated parameter, which may vary considerably among samples. Unfortunately, the statistics that work well for industry are sensitive to the variability in true concentration that is common in environmental analysis; e.g., the classic $\bar{X}$ and R statistic values increase substantially as concentration increases. This variability in true concentration means there are no expected values for randomly selected samples, so that the accuracy of testing methodology must be evaluated indirectly through the recovery of standards and spikes. As a result, it has been difficult for environmental laboratories to satisfactorily apply industrial QC techniques.

There are two possible approaches to the solution of the problem of variation in the true concentration level; either use of a statistic that is not sensitive to this variation or application of the industrial techniques within restricted concentration ranges. Obviously, the former should be preferred because it actually solves the problem and does not require the development and maintenance of a series of charts for each parameter.

A.3.1 Quality Control Charts for Accuracy

Two replacements for the Shewhart $\bar{X}$ control chart have been suggested for evaluating the recovery of a series of different standards or spikes. One of these, a cusum chart using the square of the difference between the observed and true values, is described in an EPA Region VI QC manual (5). The other alternative uses the classic Shewhart technique to evaluate the percent recovery instead of $\bar{X}$. It is recommended that the percent recovery be calculated as

$$P = 100 \frac{\text{observed}}{\text{known}}$$

for standards, or

$$P = 100 \frac{\text{observed} - \text{background}}{\text{spike}}$$

for recovery of spikes into natural water backgrounds. An example of the linear relationship between percent recovery and the known concentration of standards and spikes is demonstrated in the accuracy plots of a recent EPA method study report on analysis of mercury (6). Both approaches are being used on a daily basis by various environmental laboratories.

The data in Table A-1 were used in the EPA Region VI manual (5) to illustrate the development of a cusum chart. The actual data have been reordered here to appear in ascending order of the known values. Note that the mean and the range of the d_i^2 values increase with increasing concentration level, and this

violates a basic premise for acceptable control chart statistics. Because the percent recovery data do not show any such trend, it is the recommended control chart statistic for controlling accuracy.

From the data in Table A-1, a Shewhart control chart for percent recovery can be calculated in the following way:

Average percent recovery

$$\bar{P} = \frac{\sum_{i=1}^{23} P_i}{23}$$

$$= \frac{2310}{23}$$

$$= 100.4$$

The standard deviation for percent recovery

$$S_P = \sqrt{\frac{\sum_{i=1}^{23} P_i^2 - \sum_{i=1}^{23} P_i^{\,2} \quad 23}{22}}$$

$$= \frac{234{,}074 - (2{,}310)^2/23}{22}$$

$$= \sqrt{94.0751}$$

$$= 9.70$$

Therefore, the upper control limit becomes the following:

$$UCL = \bar{P} + 3S_P$$

$$= 100.4 + 3(9.70)$$

$$= 129.5$$

Table A.1

Analysis[1] of Total Phosphate-Phosphorus Standards, in mg/l

Total PO_4 -P

Point	Known	Obtained	Difference d_i	d_i^2	Percent Recovery P_i	P_i^2
1	0.34	0.33	0.01	0.0001	97	9,409
2	0.34	0.34	0.00	0.0000	100	10,000
3	0.40	0.40	0.00	0.0000	100	10,000
4	0.49	0.49	0.00	0.0000	100	10,000
5	0.49	0.49	0.00	0.0000	100	10,000
6	0.49	0.63	-0.14	0.0196	129	16,641
7	0.50	0.47	0.03	0.0009	94	8,836
8	0.50	0.53	-0.03	0.0009	106	11,236
9	0.50	0.56	-0.06	0.0036	112	12,544
10	0.52	0.59	-0.07	0.0049	113	12,769
11	0.66	0.70	-0.04	0.0016	106	11,236
12	0.66	0.60	0.06	0.0036	91	8,281
13	0.67	0.65	0.02	0.0004	97	9,409
14	0.68	0.65	0.03	0.0009	96	9,216
15	0.83	0.80	0.03	0.0009	96	9,216
16	0.98	0.75	0.23	0.0529	77	5,929
17	1.3	1.2	0.10	0.0100	92	8,464
18	1.3	1.3	0.00	0.0000	100	10,000
19	1.6	1.7	-0.10	0.0100	106	11,236
20	2.3	2.3	0.00	0.0000	100	10,000
21	2.3	2.4	-0.10	0.0100	104	10,816
22	3.3	3.3	0.00	0.0000	100	10,000
23	4.9	4.6	0.30	0.0900	94	8,836
Totals					2310	234,074

[1] Using a colorimetric method with persulfate digestion.

and the lower control limit becomes

$$LCL = 100.4 - 29.1$$

$$= 7.13$$

The completed control chart is shown in Figure A.2.

Following normal procedures, the control chart must indicate the conditions under which it was developed; i.e., laboratory name, parameter, method of

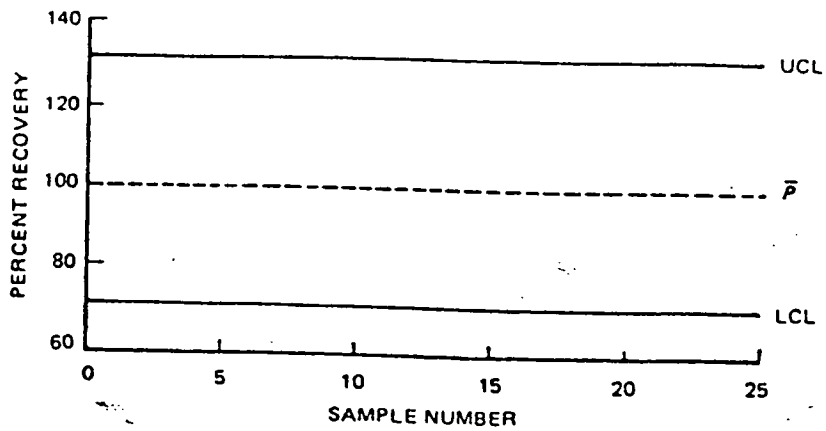


Figure A-2. Shewhart control chart for percent recovery data.

analysis, date of preparation, and any other information unique to the initializing data, such as range of concentration and identification of analyst(s). A control chart is not generally applicable under other conditions.

To verify the control chart, the initializing data should be checked to be sure that none of the values exceeds these new control limits. In addition, if its distribution is proper, about 68 percent of the initializing data should fall within the interval $\bar{P} \pm S_p$. It has been suggested that the control chart is not valid if less than 50 percent of the initializing data falls within this interval.

In applying the control chart, either of the following two conditions would indicate an out-of-control situation:

a. Any point beyond the conrol limits.

b. Seven successive points on the same side of the value $\bar{P}$ of the central line.

When an out-of-control situation occurs, analyses must be stopped until the

problem has been identified and resolved, after which the frequency should be increased for the next few percent-recovery QC checks. The problem and its solution must be documented, and all analyses since the last in-control point must be repeated or discarded.

A final note of caution regarding use of a single percent-recovery P control chart over a broad concentration range is necessary. As noted earlier for the analysis of mercury, a good linear relationship of the form

$$\bar{X} = P \text{ (known concentration)} + K$$

where K is a constant, seems appropriate for many parameters. However, to justify use of a single percent-recovery control chart, K must be small enough relative to the P (known concentration) term that it has little or no practical effect upon the value of $\bar{X}$. This will usually be true for moderate or high concentration levels, but may not be true at very low concentration levels. As a result, for some parameters it may be necessary to develop a separate percent-recovery or Shewhart $\bar{X}$ chart for each standardized low concentration level sample.

A.3.2 Quality Control Charts for Precision

Because the characteristics of the range statistic change as concentration changes, two alternatives to Shewhart's R chart have been used in environmental laboratories to evaluate the precision of routine sample analyses.

One alternative is a cusum chart using the sum of the squared difference between duplicate determinations on randomly selected routine samples (5). Because the range R for duplicate analyses is equal to the difference between them, the cusum statistic equals the sum of squared ranges $\sum R^2$. However, if R changes significantly as concentration level changes, then R^2 is affected even more and, therefore, is not as good a criterion for judging

whether precision of the system is within acceptable limits.

The other alternate uses a chart similar to the R chart, but the chart statistic is either the percent relative standard deviation ($100S/\bar{X}$), the coefficient of variation (CV or $S/\bar{X}$), or the industrial statistic I. For the duplicate determinations A and V, I equals the absolute value of their difference divided by their sum, or $|A - B| / (A + B)$, and can be shown to be equivalent to the other two statistics:

$$
\begin{aligned}
100(CV) &= 100 \frac{S}{\bar{X}} \\
&= 100 \frac{R/\sqrt{2}}{(A + B)/2} \\
&= 100 \frac{2}{\sqrt{2}} \frac{R}{A + B} \\
&= \frac{200}{\sqrt{2}} \frac{|A - B|}{A + B} \\
&= \frac{200I}{\sqrt{2}}
\end{aligned}
$$

For the sake of computational ease, I seems to be a logical alternative to R. The next concern is whether I is independent of changes in concentration level. Based upon experience with duplicates on routine samples taken during the last 2 years by EPA Region VII, I appears to decrease substantially as concentration increases. In recognition of this possible dependency, control charts for I should only be developed from and applied to results within a limited concentration range. Note that control charts for R could be applied under similar limitations.

As an illustration of the concentration dependency of these precision statistics, Table A-2 provides estimates of R and I for different concentration ranges of three parameters. These parameters were selected because approximately 100 sets of duplicates were available that were well distributed over a

reasonably broad concentration range. The ranges for the sum of duplicates A + B used in Table A-2 were selected because they were convenient and the data tended to be well distributed among them. Data judged to be out of control were discarded before any calculations were made.

Table A-2 indicates the concentration dependence of both the range R and the industrial statistic I for three parameters. Because I is not independent of concentration and is more difficult to calculate and develop control charts

Table A-2

Estimates of the Range (R = A - B) and the Industrial Statistic [I = [A - B|/(A + B)] of Three Different Parameters for Various Concentration Ranges[1]

Parameter	Range of A + B	No. of Sets of Duplicates	[2]A + B	[2]R	[2]I
BOD, 5-day (mg/l)	2 to <20	21	11.7	1.04	0.0888
	20 to <50	30	35.2	1.94	0.0552
	50 to <100	27	72.2	3.33	0.0462
	100 to <300	29	204.1	6.52	0.0319
	300 to <600	17	394.4	11.1	0.0282
	600 to <2000	12	1041	12.1	0.0116
	2000 up	3	6683	177	0.0264
Chromium (μl/l)	10 to <20	32	12.3	0.32	0.0306
	20 to <50	15	33.4	0.57	0.0170
	50 to <100	16	72.4	1.12	0.0155
	100 to <300	15	170.3	3.80	0.0223
	300 to <1000	8	480.3	5.25	0.0109
	1000 up	5	6340	76.0	0.0120
Copper (μg/l)	10 to <30	16	22.2	0.93	0.0617
	30 to <50	23	38.2	1.35	0.0368
	50 to <100	21	70.8	1.14	0.0169
	100 to <200	26	131.9	2.33	0.0177
	200 to <400	10	268.0	2.81	0.0105
	400 up	3	702.0	4.56	0.0065

[1] From EPA Surveillance and Analysis Laboratory, Region VII.
[2] Average values.

for, the use of R charts for a series of sequential concentration ranges for each parameter seems practical. However, because the primary concern when using any range chart is whether the upper control limit has been

Table A-3

Shewhart Upper Control Limits (UCL) and Critical Range R_c Values for the Differences Between Duplicate Analyses Within Specific Concentration Ranges for Three Parameters[1]

Parameter	Concentration Range[2]	UCL	R_c
BOD, 5-day (mg/l)	1 to <10	3.40	3.5
	10 to <25	6.34	6
	25 to <50	10.9	11
	50 to <150	21.3	21
	150 to <300	36.3	36
	300 to <1000	39.6	[3]40
	1000 up	579	[3]579
Chromium (μg/l)	5 to <10	1.05	1
	10 to <25	1.86	2
	25 to <50	3.66	4
	50 to <150	12.4	12
	150 to <500	17.2	[3]17
	500 up	249	[3]249
Copper (μg/l)	5 to <15	3.04	3
	15 to <25	4.41	4
	25 to <50	3.73	5
	50 to <100	7.62	8
	100 to <200	9.19	[3]9
	200 up	14.9	[3]15

[1] From EPA Surveillance and Analysis Laboratory, Region VII.
[2] Equal to half of the range of A + B given in Table A-2.
[3] Based on fewer than 15 sets of duplicate analyses.

exceeded, an even more practical approach would be to develop a table of these limits for all concentration levels of each parameter. As an example, Table A-3 contains the calculated Shewhart upper control limits for the range R from duplicate analyses within the various concentration levels for the three parameters in Table A-2. These limits were calculated, as usual, from the Shewhart factor D_4 for ranges based upon duplicate analyses and the appropriate average value of the range R given in Table A-2. For example, the UCL for 25 to 50 mg/l of BOD was calculated as follows:

$$UCL = D_4R$$
$$= 3.27(3.33)$$
$$= 10.9$$

Table A-3 also contains a critical range R_c column. Because the data from EPA Region VII were almost always whole units with only a very occasional half unit reported, the R_c value is the UCL value rounded to the nearest whole unit at higher concentration levels and to the nearest half unit for the lowest concentration level. However, there is an exception to this rule among the low-concentration R_c values for copper that demonstrates an advantage beyond the simplicity of using such tables. The UCL value for copper at 25 to 50 μg/l is inconsistent with the UCL values for adjacent concentration levels, and the R_c value has been adjusted to resolve this inconsistency. Without the table, such inconsistencies could very easily go unnoticed.

The examples in Table A-4 illustrate how to use the R_c values in Table A-3. This technique, consisting of the development and use of a table of critical-range R_c values at different concentration levels, is recommended to control precision. Normal control chart procedures should be followed as in Section A 3.1 regarding identification and verification of the table. The table should be updated periodically as additional, or more current, data become available, or whenever the basic analytical system undergoes a major change. If any difference between duplicate analyses exceeds the critical-range value for the appropriate concentration level, then analyses must be stopped until the problem is identified and resolved, and the frequency should be increased for the next few precision checks. After resolution, the problem and its solution must be documented, and all analyses since the last in-control check must be repeated or discarded.

A.4 Recommended Laboratory Quality Assurance Program

A minimum laboratory quality assurance program should include control procedures for each parameter as described in the following sections.

A.4.1 Standard Curves

A new standard curve should be established with each new batch of reagents, using at least seven concentration levels.

A.4.2 Quality Control Checks for Each Analytical Run

With each batch of analyses, the following tests should be run:

a. One blank on water and reagents.

Table A-4

Critical Range Values for Varying Concentration Levels

Parameter	Duplicates	R	R_c	$R \leq R_c$	Condition of System
BOD (mg/l)	20 and 24	4	6	Yes	Normal
Chromium (μg/l)	60 and 75	15	12	No	Out-of-control
Copper (μg/l)	46 and 51	5	[1] 5	Yes	Normal

[1] This R_c value is used because (46 + 51)/2 = 48.5, which is between 25 and 50.

b. One midpoint standard.

c. One spike to determine recovery.

d. One set of duplicate analysis.

The results from b through d should be compared with previous in-control data by using the appropriate technique recommended in section A.3.

A.4.3 Interlaboratory QC

An interlaboratory QC program would require each laboratory to do the following:

a. Analyze reference-type samples to provide independent checks on the

analytical system. These may be available from EPA as QC samples, from the National Bureau of Standards as standard reference materials, or from commercial sources. If performance limits are not provided, the results should fall within the routine limits of each laboratory for a standard at a level comparable to the specified true value.

b. Participate in performance evaluation and method studies as available from the American Society for Testing and Materials, and from other agencies.

A.5 Outline of a Comprehensive Quality Assurance Program

In the following discussion the symbols used represent the results of analysis according to the scheme:

A_1 = first replicate of sample A

A_2 = second replicate of sample A

B = sample taken simultaneously with sample A

B_{SF} = field spike into sample B

B_{SL} = laboratory spike into sample B

D_F = field spike into distilled water

D_L = laboratory spike into distilled water

T = true value for all spikes

The laboratory spikes B_{SL} and D_L are the only analyses that may not be necessary. All other analyses must be done simultaneously.

A.5.1 Steps for the Field Personnel

A comprehensive quality assurance program would include the following steps for each parameter in the monitoring study:

a. Take independent simultaneous samples A and B at the same sampling

point. Depending on the parameter, this might involve side-by-side grab samples or composite samplers mounted in parallel.

b. Split sample A into the equal-volume samples A_1 and A_2.

c. Split sample B into equal volumes and add a spike T to one of them; the latter sample becomes sample B_{SF}. As with all spikes, the addition of T should approximately double the anticipated concentration level.

d. Add the same spike T to a distilled water sample furnished by the laboratory and designate this sample as D_F.

These samples must be treated in the same way as routine samples; i.e., the volume, type of container, preservation, labeling, and transportation must be the same for all.

A.5.2 Steps for the Laboratory

The laboratory personnel should perform the following steps for quality assurance:

a. Analyze the blank and midpoint standard recommended in section A.4. If results are unsatisfactory, resolve problems before continuing.

b. Analyze sample D_F. If the percent recovery of T is unsatisfactory (see section A.3.1), create a similarly spiked, distilled-water sample D_L and analyze to test for a systematic error in the laboratory or fundamental problems with the spike. If the percent recovery of T from D_L is satisfactory, any systematic error occurred before the samples reached the laboratory.

c. Analyze samples B and B_{SF}. If B is below the detection limit, or if B is greater than 10T or less than 0.1T, disregard the remainder of this step and proceed to step d. If the percent recovery of T from B_{SF} is unsatisfactory (see section A.3.1), spike an aliquot

of sample B the same way in the laboratory so that a similar recovery can be anticipated. Analyze this sample B_{SL} to test for immediate interferences or a bad background result B . If the percent recovery from B_{SL} is satisfactory, then the interference must require a longer delay before analysis, or other special conditions not present in the laboratory, in order to have a noticeable effect upon recovery of the spike.

d. Analyze A_1 and A_2 . If the absolute (unsigned) difference between these results exceeds the critical value (see section A.3.2), then precision is out of control.

e. Calculate the absolute difference between A_1 and B . If it is unsatisfactory (see section A.3.2), the field sampling procedure did not provide representative samples.

If initial results at each of the laboratory steps were satisfactory, then the validity of the related data has been indisputably established. If results at any step are unsatisfactory, resolution depends upon the problem identified. Laboratory problems may just require that the analyses be repeated, but field problems will usually require new samples. Figure A-3 is intended to clarify the interdependence of the preceding laboratory steps b through e.

In figure A-3 it must be noted that there is no way to identify additive sample interferences; i.e., those that have an equal effect upon the background-plus-spike results (B_{SF} or B_{SL}) and the background result B . Recovery of a spike will not show such interferences.

Problems causing systematic errors that may occur in the field include the following:

a. Contaminated preservative, distilled water, or containers.

b. Contamination by sampling personnel.

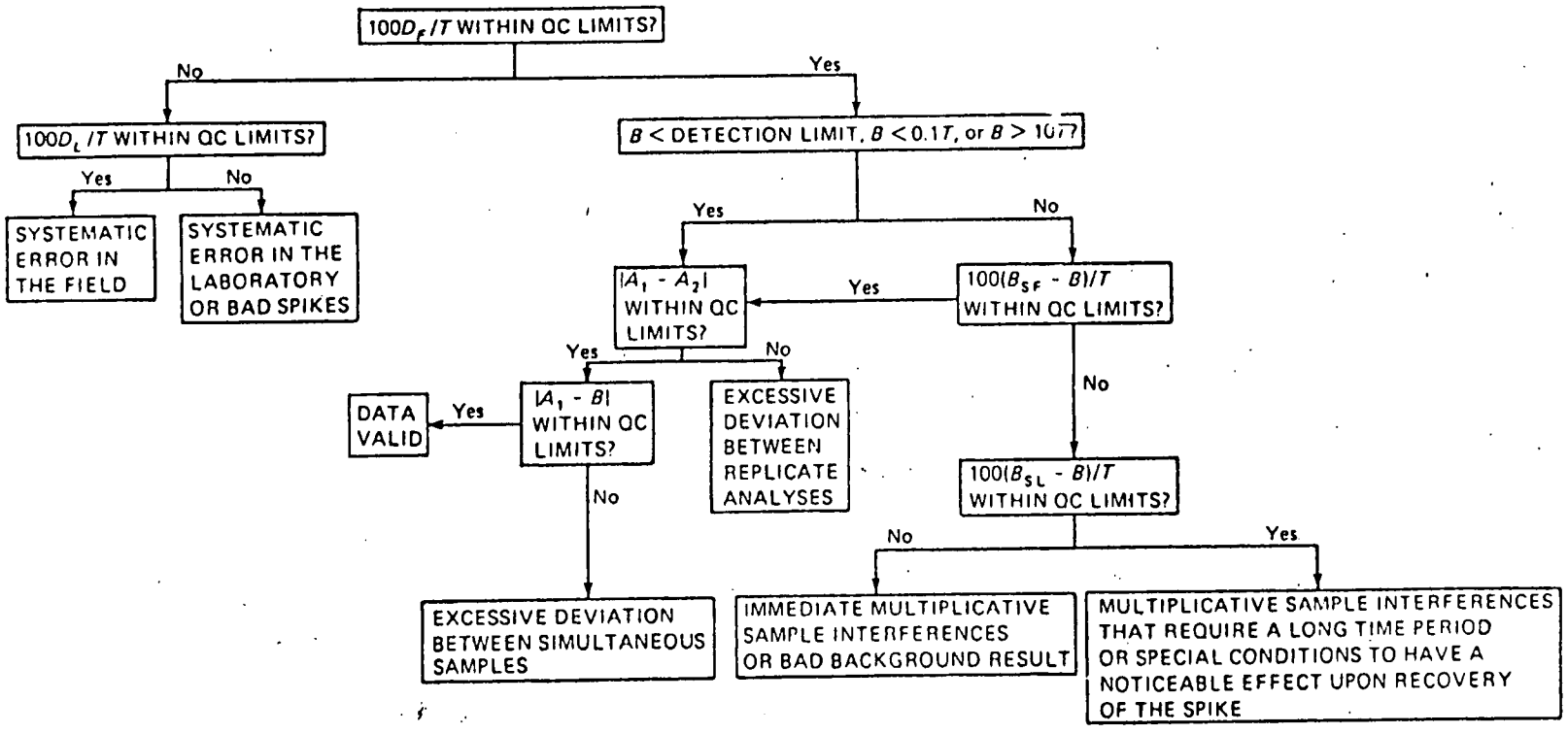


Figure A.3. Procedure for evaluating QC data from a monitoring study.

c. Deterioration through holding time or use of an ineffectual preservation technique.

d. Use of a bad field-spiking procedure.

A.6. Related Topics

A.6.1 Advanced Laboratory Automation and its Effect on QC

Advanced laboratory automation systems under development analyze samples automatically and use a control computer to interpret the resulting data and produce an analytical report. The primary benefits of such a system are not only that the data-recording and calculation errors common to manual analyses have been inherently eliminated, but also that extensive QC can be accomplished quite easily and cheaply. The computer can be programed to automatically recognize different kinds of QC samples and to establish or recall appropriate control limits. Thus the QC overhead is reduced considerably and QC procedures previously too costly or complex become practical.

As an example of a QC procedure that is considered impractical for manual use, regression could be used to determine the relationship between concentration change and the accuracy and precision statistics discussed earlier. Using these relationships, very responsive, single accuracy and precision charts could be developed for each parameter. As computer-assisted analysis becomes common, automated laboratories will very likely replace the manual procedures recommended earlier in this chapter with evaluation criteria based upon regressions.

A.6.2 Method Comparability (Equivalency)

Requirements for method comparability are under development for proposed alternatives to the methodology specified in Public Law 92-500, section 304(g).

A final version of these requirements should be available at a later date.

A.7 References

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APPENDIX B*

QUALITY ASSURANCE EVALUATION CRITERIA CHECKLIST FOR PROPOSALS AND GRANT APPLICATIONS OFFERING SAMPLING AND ANALYSIS SERVICES

Criteria	Scoring Value	×	Numerical Weight	=	Individual Score
A. Quality assurance management policy/written procedures.					
1a. Does the offeror have an on-going QA program?	_____	×	5	=	_____
2a. Does the offeror have a written QA manual that he will make available for review?	_____	×	4	=	_____
3a. Has the offeror designated a QA coordinator or a QA supervisor who reports to senior management levels?	_____	×	3	=	_____
4a. Does the proposed project management structure provide for adequate QA?	_____	×	2	=	_____
5a. Will a project specific QC plan be prepared before commencement of sampling and analysis?	_____	×	1	=	_____

Total Score for Sub-element A. _____

Maximum Possible Score 75

Percent of maximum possible score awarded for Sub-element A. (circle closest value).

0% 20% 40% -- 60% 80% 100%

Score for this sub-element of the proposal evaluation criteria (circle corresponding value).

0 1 2a 2b 3 4 5

* Source: Stratton, C. L. and Bands, J. D., "Quality Assurance Guidelines for IERL-CI Project Officers," EPA-600/19-79-046, U. S. Environmental Protection Agency, Cincinnati, OH 45268, December 1979.

Criteria	Scoring Value	×	Numerical Weight	=	Individual Score
B. Quality assurance procedures for sampling.					
1b. Are sampling locations chosen to assure representative samples will be taken?	____	×	2	=	____
2b. Will the proposed sampling program yield data of statistical significance as appropriate to the objectives of the project (e.g., replicate samples, background samples, etc., should be discussed)?	____	×	2	=	____
3b. Does the offeror show an understanding of the proper techniques used to collect representative samples while avoiding sample contamination?	____	×	3	=	____
4b. Does the offeror have access to the appropriate sampling equipment?	____	×	2	=	____
5b. Are samples to be shipped promptly to the laboratory to meet maximum sample holding time limitations?	____	×	3	=	____
6b. Are appropriate sample preservation methods proposed?	____	×	2	=	____
7b. Are sample chain-of-custody procedures described?	____	×	1	=	____
			Total Score for Sub-element B.		____
			Maximum Possible Score		75

Percent of maximum possible score awarded for Sub-element B. (circle closest value).

0% 20% 40% -- 60% 80% 100%

Score for this sub-element of the proposal evaluation criteria (circle corresponding value).

0 1 2a 2b 3 4 5

Criteria	Scoring Value	×	Numerical Weight	=	Individual Score
C. Quality assurance procedures for analysis.					
1c. Does the offeror intend to use standard analytical methods where available? If standard methods are not available, will the mthods used be documented?	_____	×	3	=	_____
2c. Does the offeror have a laboratory QC program which specifies at least 5-10 percent sample replication and 5 percent spiked sample analysis?	_____	×	4	=	_____
3c. Is high quality analytical instrumentation available for use on the project?	_____	×	2	=	_____
4c. Are laboratory facilities adequate?	_____	×	2	=	_____
5c. Are analytical detection limits adequate for the purposes of the project?	_____	×	2	=	_____
6c. Does the offeror participate in EPA and/or other interlabortory QC programs?	_____	×	2	=	_____
Total Score for Sub-element C.					_____
Maximum Possible Score					75

Percent of maximum possible score awarded for Sub-element C. (circle closest value).

0% 20% 40% -- 60% 80% 100%

Score for this sub-element of the proposal evaluation criteria (circle corresponding value).

0 1 2a 2b 3 4 5

Criteria	Scoring Value	×	Numerical Weight	=	Individual Score
D. Quality assurance procedures for data management.					
1d. Does the offeror possess appropriate data handling, processing, and retrieval capabilites?	_____	×	3	=	_____
2d. Will QC data (e.g., standard curves, duplicate results, spike sample results) be maintained and be accessible to the Project Officer?	_____	×	3	=	_____
3d. Does the organization routinely maintain analytical performance records such as quality control charts?	_____	×	2	=	_____
4d. Are all laboratory results and QC data reviewed by laboratory supervisory personnel?	_____	×	3	=	_____
5d. Are alldata and records retained for a minimum of 3 years:	_____	×	1	=	_____
6d. Are field notebooks used to record sampling and engineering data (e.g., sample number, date/time of collection, flow, operating conditions, etc.)?	_____	×	3	=	_____
			Total Score for Sub-element D.		_____
			Maximum Possible Score		75

Percent of maximum possible score awarded for Sub-element D. (circle closest value).

0% 20% 40% -- 60% 80% 100%

Score for this sub-element of the proposal evaluation criteria (circle corresponding value).

0 1 2a 2b 3 4 5

Appendix C
Revised

EDW

DEPARTMENT OF THE ARMY
U.S. Army Corps of Engineers
Washington, D.C. 20314-1000

ETL 1110-2-309

CEEC-EH-W

Engineer Technical
Letter No. 1110-2-309

5 February 1988

Engineering and Design
WATER AND WASTEWATER LABORATORY INSPECTIONS

1. Purpose. The purpose of this letter is to provide guidance for conducting inspections of commercial laboratories engaged in the chemical and physical analysis of environmental samples of water, wastewater, and related media for the Corps of Engineers.

2. Applicability. This ETL is applicable to all HQUSACE/OCE elements and field operating activities having civil works responsibility for laboratory inspections.

3. References.

a. ER 1110-1-261

b. ER 1110-1-263

c. ER 1110-1-8100

d. USACE Chemical Quality Management Protocol for Evaluation of Contract Laboratories Providing Analyses for Superfund and Defense Environmental Restoration Account (DERA) Projects, 8 April 1986.

4. Policy. The basic policies concerning inspection of laboratories performing analysis for water and wastewater projects are set forth in references 3a and 3c. Activities involving Superfund and DERA projects are governed by references 3b and 3d.

5. Background. The purpose of laboratory inspections is to assure that laboratories performing analyses for water and wastewater projects have the required capability, are following accepted quality control procedures and are using methods consistent with those contained in documents listed in Enclosure 1 or in contract specifications. Difficulties arising during laboratory inspections are frequently a result of deficient program or contract specifications.

6. Laboratory Inspections.

a. General. Inspections should be performed prior to the initiation of laboratory testing (or award of contract) and at appropriate intervals thereafter. Following each inspection, a report covering observations and recommendations should be provided to the district commander and will

This engineer technical letter supersedes ETL 1110-2-268 dated 31 December 1981.

generally include a summary of contract requirements for chemical/physical testing, a completed laboratory evaluation checksheet, and a summary of findings with specific recommendations. If satisfactory performance on audit samples is a contract requirement, an evaluation of the results should be included in the report.

b. Program Specifications. Prior to an inspection, a review of the program and contract specifications should be made to determine requirements for field sampling and analysis, transportation and handling of samples, laboratory analyses, facilities and equipment, personnel, reporting, and quality control. This review will establish the level of detail required in the inspection. Enclosure 2 describes material generally covered in this review.

c. Onsite Laboratory Inspection. When a site visit is planned, arrangements should be made for a schedule that will have a minimum impact on routine activities, allow observation of a variety of tests in actual operation, and assure the presence of the laboratory staff. At the laboratory, an interview is held with the facility director to discuss the purpose of the site visit and to emphasize the Corps requirements for an active quality assurance/quality control program. This is followed by an evaluation survey to determine the laboratory's ability to meet program requirements or contract specifications. A checksheet is presented in Enclosure 3 to aid in this evaluation. It may be necessary to add or delete items based on individual program needs. Note also that much of the information in Enclosure 3 may be obtained before the inspection, either as part of the quality assurance plan required by contract specifications, or by written communication. An exit interview is then held with the laboratory director to discuss observations and make recommendations.

7. Conclusions. The inspection of laboratories to ensure the reliability of environmental data is a very important part of the overall quality assurance plan for the Corps of Engineers. The enclosures provide guidance to assist in carrying out this responsibility. However, variations in funding levels, types of laboratories involved, and analyses required dictate that considerable judgement be exercised by the individuals responsible for the inspection program. All of the information outlined may not be required for each inspection, while additional information may be needed in some cases. Contracts must often be written which require state-of-the-art procedures and this may cause some contracts to be less specific than others. Established methods often allow some discretion on the part of the analyst.

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12. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods. SW-846, November 1986. U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington, D.C. 20460.

Enclosure 1

13. Jones, Jerry N. and Cullinane, M. John. Guidelines for Water Quality Laboratory Operations. July 1985, Environmental Laboratory, USAE Waterways Experiment Station, Vicksburg, MS 39180.

14. Palermo, Michael R., Development of a Modified Elutriate Test for Estimating the Quality of Effluent from Confined Disposal Areas. August 1986, Environmental Laboratory. USAE Waterways Experiment Station, Vicksburg, MS 39180.

15. Waide, Jack B., General Guidelines for Monitoring Contaminants in Reservoirs. February 1986, Environmental Laboratory, USAE Waterways Experiment Station, Vicksburg, MS 39180.

CONTRACT, PROJECT, OR PROGRAM SPECIFICATIONS SUMMARY
(For Each Individual Contract, Project, or Program)

1. Contract, Project, or Program No. _____

2. Reviewed by _____

3. Contract, Project or Program Requirements

a. Field Sampling and Analysis Requirements

(1) Sampling Sites

(2) Methods of Collection and Preservation

(3) Field Analyses-Parameters and Methods

(4) Sampling Schedules

(5) Sample Transportation

b. Laboratory Analysis Requirements

(1) Quality Control (QC) Plan

(2) Personnel Qualifications

(3) Analytical Methods Specified

(4) Instrumentation Required

(5) Detection Limits

(6) Laboratory Data Reporting and Records Keeping

(7) Reporting Requirements (Progress, Interim, Final and QC)

c. Other Requirements (i.e., audit samples)

Note: The above outline is a guide and its contents should not be construed as the only requirements that exist in a contract, project, or program. Any other pertinent information not covered in 3a-3b should be noted under 3c, Other Requirements.

Enclosure 2

In addition, new equipment can make procedures unnecessary that were previously mandatory. In view of these considerations, inspections should be carried out by personnel qualified in laboratory techniques and knowledgeable regarding acceptable alternative methods.

FOR THE COMMANDER:

3 Encls

HERBERT H. KENNON
Chief, Engineering Division
Directorate of Engineering and
Construction

LABORATORY EVALUATION CHECKSHEET

LABORATORY: _____

ADDRESS: _____

DATE: _____

LABORATORY PERSONNEL CONTACTED

NAME	TITLE

EVALUATION PERSONNEL

NAME	TITLE

Enclosure 3

1. ORGANIZATION OF PERSONNEL

Some personnel may occupy more than one position, particularly in small laboratories. The recommended minimum standards for the laboratory director/manager are a bachelor of science degree and 5 years experience. The recommended minimum standards for the chief analyst directing the testing operations are a bachelors degee in chemistry and 2-3 years experience in analyses being performed.

a. Laboratory Director/Manager
   Name: _____
   Education: _____
   Experience: _____

b. Project Manager
   Name: _____
   Education: _____
   Experience: _____

c. Quality Assurance Officer
   Name: _____
   Education: _____
   Experience: _____

d. Chief Classical Inorganic Analyst
   Name: _____
   Education: _____
   Experience: _____

e. Chief Metals Analyst
   Name: _____
   Education: _____
   Experience: _____

f. Chief Organic Analyst
   Name: _____
   Education: _____
   Experience: _____

2. ANALYSTS

Most analysts should have a minimum bachelor of science degree in chemistry or closely related laboratory science and at least one year's experience performing the analyses required in the contract. Personnel interpreting spectra from gas chromatographs or gas chromatograph/mass spectrometers should have a minimum of two and three year's experience, respectively. Some laboratories make extensive use of technicians. Their work should be performed under the direction of a chemist or senior analyst.

a. Name: _____
Education: _____
Experience: _____
Analyses Performed: _____

b. Name: _____
Education: _____
Experience: _____
Analyses Performed: _____

c. Name: _____
Education: _____
Experience: _____
Analyses Performed: _____

d. Name: _____
Education: _____
Experience: _____
Analyses Performed: _____

e. Name: _____
Education: _____
Experience: _____
Analyses Performed: _____

3. SUMMARY OF PERSONNEL

a. Do the personnel assigned to this project have appropriate educational background and experience to successfully fulfill this contract? Explain. _____

_____

b. Is the laboratory adequately staffed to meet time requirements of of the contract? _____

c. Were pertinent personnel available for interview during the inspection? _____

d. Are training programs in effect to keep analysts current in instrumentation, procedures and quality control? _____

e. Are personnel adequately trained in safety procedures? _____

f. Other comments regarding personnel _____

_____

_____

_____

4. SAMPLING

a. Does the contract specify sampling by the laboratory? _____

b. What sampling methods are being used? Do they follow contract specifications? _____

c. What type of sample containers are being used? _____

d. State method used for cleaning containers. _____

e. State preservation methods being used. _____

f. How are samples identified in the field? _____

g. State method used for recording field sampling and analysis. _____

h. What analyses are being performed in the field? _____

i. Are field calibrations required? Explain. _____

j. How are samples transported to the laboratory? Are holding times being met? _____

k. Are blind duplicate and spike samples prepared in the field for laboratory analysis? Are field blanks used? _____

l. Are chain-of-custody procedures being followed if this is required in the contract? _____

m. Other comments regarding sampling _____

5. SAMPLE RECEIPT AT THE LABORATORY

If any analyses are to be subcontracted to another laboratory a separate site inspection is required to evaluate the subcontractor's capabilities.

a. Is a person designated to receive samples into the laboratory?

Name: _____

b. Are written procedures developed for receipt and storage of samples? Are they available at the sample receipt area? _____

c. Is a chain of custody form signed by persons logging in samples if appropriate? _____

d. Is a permanent logbook maintained? Are records kept in ink? _____

e. Are chain-of-custody seals checked for integrity? _____

f. Does the information on the samples match that in the field notebooks or the sample transmittal sheets? _____

g. Are discrepancies noted in the logbook? _____

h. Are adequate facilities available or sample storage, including refrigerator and freezer space? _____

i. Is a system in effect to assure that the proper cold storage temperature is maintained? _____

j. Other comments regarding sample receipt: _____

6. LABORATORY FACILITIES

a. Is the laboratory maintained in a clean and efficient manner? _____

b. Does the laboratory appear to have adequate work space? _____

c. Does the laboratory have sufficient fume hoods? Are they checked periodically for air flow? _____

d. Does the laboratory have safety devices such as eye washes, showers, spill control kits, etc? _____

e. Are laboratory operations adequately separated to avoid cross-contamination? _____

f. Are chemical waste disposal procedures well-defined and followed by the laboratory? _____

g. Is an adequate supply of distilled or deionized water available? Is it checked for purity? _____

h. Is adequate chemical storage available, including solvent cabinets? Are chemicals properly segregated? _____

i. Are chemicals dated upon receipt? _____

j. Are chemicals in use that have passed identified expiration dates? _____

k. Is sufficient glassware available to handle samples specified in the contract? _____

l. Is volumetric glassware of class "A" quality? _____

m. Are instructions for glassware cleaning posted near the wash area? _____

n. Is provision made for standards storage? Inorganics _____ Organics, refrigerated _____

o. Other comments regarding facilities _____

7. INSTRUMENTATION

a. What type of analytical balances are in use? Are they located on vibration-free surfaces away from drafts? _____

b. Are balances checked routinely with class S weights and recorded in a logbook? _____

c. Are balances calibrated and checked annually by a certified technician? _____

d. List other instrumentation.

INSTRUMENT	MODEL	AGE	ANALYSES

e. Are instruments adequate for the analyses to be performed? _____

f. Have any of the instruments been modified in any way? How? _____

g. Are manufacturers operating manuals readily available for each instrument? _____

h. Are calibration protocols available to the operator? _____

i. Are calibration results maintained in a permanent record? _____

j. Are instruments under service contract? Which ones? _____

k. Are permanent service records available? _____

l. Are instruments properly vented? _____

m. Additional comments on instruments _____

8. ANALYTICAL METHODOLOGY

a. List analytical procedures:

ANALYTE	REFERENCE	SPECIFIED IN CONTRACT

b. Do the methods conform to those specified in the contract? _____

c. Are there any deviations to the reference methodology? Explain. _____

d. Are the written procedures readily available to the analysts? _____

e. Do the methods provide the needed detection limits? _____

f. Does the laboratory require strict adherence to specific quality control procedures for each method? _____

g. Are reagent grade or higher purity chemicals used? _____

h. Are fresh analytical standards prepared in keeping with good quality control practices? _____

i. Are the primary standards traceable to NBS or EPA standards? _____

j. Are records of standards preparations maintained in a logbook? _____

k. Do the analysts maintain complete records of analyses with comments and enter data in a neat and accurate manner? _____

l. Additional comments on analyses _____

9. QUALITY ASSURANCE/QUALITY CONTROL

a. Does the laboratory maintain a quality assurance/quality control manuel? _____

b. Does the manual address the following items?

(1) Organization and Personnel
(2) Quality assurance objectives
(3) Sampling procedures
(4) Facilities and equipment
(5) Instrumentation and maintenance
(6) Calibration procedures and frequency
(7) Analytical procedures
(8) Data analysis, validation and reporting
(9) Internal quality control checks
(10) External quality control checks
(11) Corrective action
(12) Record keeping

c. Is this manual readily available to all laboratory personnel? _____

d. Are duplicate and spike analyses performed on a minimum number of samples (i.e. 10%)? _____

e. Are acceptable criteria developed for duplicate and spike analyses? _____

f. Are quality control charts used? _____

g. Are reagent blank analyses run with each set of samples? _____

h. Are a minimum of three and preferably more standards run to produce standard curves? _____

i. Do routine procedures require that sample concentrations fall within the limits of the standard curves? _____

j. Do supervisory personnel audit laboratory procedures on a routine basis? _____

k. Does the laboratory routinely run standard reference materials to evaluate analytical performance? Are these results documented? _____

l. Are data calculations checked by a second person? _____

m. Are recoveries of organic surrogates documented? _____

n. Are tuning records maintained for gas chromatograph/mass spectrometers? _____

o. Are raw data and records maintained for the required peiod of time? _____

p. Are quality control data routinely reported? _____

q. Are external standard quality control samples run at least twice each year? _____

r. Additional comments on quality assurance/quality control: _____

10. REPORTING

a. Is a standard reporting format required? _____

b. Is provision made for the submission of raw data and chromatograms if required? _____

c. Is there a specified time frame for reporting data? _____

d. Additional comments on reporting _____

Appendix D

ALG PROCEDURES- FY 89

References:

1. U. S. Environmental Protection Agency, Methods for Chemical Analysis of Water and Wastes, EPA 600/4-79-020, March 1979 and EPA 600/4-82-055 Dec 1982. Environmental Monitoring and Support Laboratory, Cincinnati, OH 45268.

2. U. S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste, SW-846 Third Edition, November 1986 with December 1988 revisions. Office of Solid Waste and Emergency Response, Washington, DC 20460.

3. U. S. Environmental Protection Agency, Manual of Analytical Methods for the Analysis of Pesticides in Humans and Environmental Samples, EPA-600/8-80-038, June 1980. Health Effects Research Laboratory, Research Triangle Park, NC 27711.

4. Federal Register, "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act", Vol 49, No. 209, Friday, October 26, 1984.

5. Plumb, Russell H.,Jr. Procedures for Handling and Chemical Analysis of Sediment and Water Analysis, EPA/CE-81-1 May 1981. Environmental Laboratory, U. S. Army Engineer Waterways Experiment Station, Vicksburg, MS 39180.

6. American Public Health Association, Standard Methods for the Examination of Water and Wastewater 16th Edition, 1985. 1015 Fifteenth Street NW, Washington, DC 20005.

7. ASTM, 1987 Annual Book of ASTM Standards, Water and Environmental Technology Vol 11.01 & 11.02. 1987. 1916 Race Street, Philadelphia, PA 19103.

8. U. S. Environmental Protection Agency, Chemistry Laboratory Manual for Bottom Sediments and Elutriate Testing March 1979. Region V Central Regional Laboratory, 536 Clark Street, Chicago, IL 60605

9. Palermo, Michael R. "Development of a Modified Elutriate Test for Estimating the Quality of Effluent from Confined Dredged Material Disposal Areas" Technical Report D-86-4. August 1986. Department of the Army Waterways Experiment Station, Corps of Engineers, P. O. Box 631, Vicksburg, Ms 39180.

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Table 1- List of Nonmetal Inorganic Test Procedures

			Reference (Method No. or page)			
	Parameter	Media	Ref 1 EPA 1979	Ref 2 SW-846	Ref 6 Std Meth	Other
1.	Acidity	Water	305.1			
2.	Alkalinity	Water	310.2			
3.	Ammonia-N	Water	350.1			
4.	BOD	Water			507	
5.	Cation Exchange Capacity	Soil				Ref 5, p 3-20
6.	COD	Water	410.4			
7.	COD,	Sediment				Ref 8, p 54
8.	Chloride	Water	325.2			
9.	Cyanide	Water	335.3			
10.	CN amenable to Cl	Water	335.1 & 335.3			
11.	Hardness	Water	130.1			
12.	Kjeldahl N	Water	351.2			
13.	Nitrate-Nitrite-N	Water	353.2			
14.	Nitrite-N	Water	353.2			Ref 5, p 3-163
15.	Oil & Grease	Water	413.1			
16.	TOC	Water & Sed			505C	
17.	Phenols, tot	Water	420.2			
18.	Phosphorus, total	Water	365.4			
19.	Othophosphate	Water	365.1			
20.	Total solids Dried 103-105°C	Water	160.3		209A	
		Soil, Sed			209F	

21.	Total Dissolved Solids Dried 180°C	Water	160.1	209B
22.	Total Suspended Dried 103-105°C	Water	160.2	209C
23.	Settleable solids	Water	160.5	209E
24.	Total Volatile Solids	Water	160.4	209D
25.	Sulfate	Water	375.2	
26.	Sulfide	Water	376.2	
27.	Elutriate test sediments, std			Ref 5 p 2-28
28.	Elutriate test sed, modified			Ref 9
29.	EP Toxicity Liquid & Solid		1310	

Table 2 Analytical Procedures for Metals

Parameter	Media	Ref 1 EPA-1979	Ref 2 SW-846	Other	Routine Meth Det. limits
1. Digest for tot rec met	Water		3005		
2. Digest for tot metals	Water		3010		
3. Digest for Metals-GFAA	Water		3020		
4. Digest for Metals	Sed., Soil & Sludge		3050		
5. Simultaneous anal by DCP				Ref 7, D4190-82	
5a. Simultaneous anal by ICP		200.7	6010		
6. Aluminum, DCP				Ibid	30ug/l
ICP		200.7	6010		100ug/l
7. Antimony, DCP				Ibid	
ICP		200.7	6010		
8. Antimony, GFAA		204.2	7041		5ug/l
9. Arsenic,GFAA		206.2	7060		5ug/l
10. Arsenic,hydride		206.3	7061		5ug/l
11. Barium,AA		208.1	7080		100ug/l
12. Barium,GFAA		208.2			2ug/l
13. Barium, DCP				Ref 7,	10ug/l
ICP		200.7	6010	D4190-82	5ug/l
14. Beryllium, DCP				Ibid	5ug/L
ICP		200.7	6010		5ug/L
15. Boron, DCP				Ibid	10ug/l
ICP		200.7	6010		
16. Cadmium, DCP				Ibid	20ug/L
ICP		200.7	6010		20ug/l
17. Cadmium, GFAA		213.2	7131		0.1ug/l

18. Calcium, DCP				Ibid	100ug/l
ICP		200.7	6010		200ug/l
19 Calcium, AA		215.1	7140		
20. Chromium, DCP				Ibid	20ug/l
ICP		200.7	6010		20ug/l
21. Chromium, GFAA		218.2	7191		1ug/l
22. Chromium VI		218.4	7197		10ug/l
23. Cobalt, DCP				Ibid	30ug/l
ICP		200.7	6010		50ug/l
24. Cobalt, GFAA		219.2	7201		1ug/l
25. Copper, DCP				Ibid	30ug/l
ICP		200.7	6010		20ug/l
26. Copper, GFAA		220.2			1ug/l
27. Iron, DCP				Ibid	30ug/l
ICP		200.7	6010		30ug/l
28. Iron,AA		236.1	7380		30ug/l
29. Iron,GFAA		236.2	7381		1ug/l
30. Lead, DCP				Ibid	200ug/l
ICP		200.7	6010		100ug/l
31. Lead, GFAA		239.2	7421		1ug/l
32. Magnesium, DCP				Ibid	30ug/l
ICP		200.7	6010		30ug/l
33. Magnesium, AA		242.1	7450		
34. Manganese, DCP				Ibid	30ug/l
ICP		200.7	6010		10ug/l
35. Manganese, AA		243.1	7460		
36. Manganese, GFAA		243.2			1ug/l
37. Mercury, Cold	Water	245.1	7470		0.2ug/l
Vapor	Sed.	245.5	7471		0.2ug/g
38. Molydenum, DCP				Ibid	50ug/l
ICP		200.7	6010		30ug/l
39. Molydenum, GFAA		246.2	7481		1ug/l

40. Nickel, DCP			Ibid	30ug/l
ICP	200.7	6010		20ug/l
41. Nickel,GFAA	249.2			1ug/l
42. Potassium, DCP			Ibid	100ug/l
ICP	200.7	6010		
43. Potassium, AA	258.1	7610		10ug/l
44. Selenium, GFAA	270.2	7740		5ug/l
45. Selenium,Hydride	270.3	7741		5ug/l
46. Silver, DCP			Ibid	30ug/l
ICP	200.7	6010		20ug/l
47. Silver, GFAA	272.1	7761		1ug/l
48. Sodium, DCP			Ibid	100ug/l
ICP	200.7	6010		100ug/l
49. Sodium, AA	273.1	7770		5ug/l
50. Thallium, DCP			Ibid	30ug/l
ICP	200.7	6010		30ug/l
51. Thallium, GFAA	279.2	7841		1ug/l
52. Tin, DCP			Ibid	50ug/l
53. Tin, GFAA	282.2			5ug/l
54. Titanium, GFAA	283.2			10ug/l
55. Vanadium, DCP			Ibid	20ug/l
ICP	200.7	6010		20ug/l
56. Vanadium,GFAA	286.2	7911		5ug/l
57. Zinc, DCP			Ibid	30ug/l
ICP	200.7	6010		10ug/l
58. Zinc, GFAA	289.2			5ug/l
59. Silicon, DCP			Ibid	100ug/l

Table 3. Organic Procedures

Parameter	Media	Reference		
		Ref 4 Fed Reg 1984	Ref 2 SW-846	Ref 6 Std Meth
1. Soxhlet Extraction	Soil & Sed.		3540	
2. Sep funnel Liq-Liq extr	Water Soluble		3510	
3. Purge & trap Prep-GC			5030	
4. Sonication Extraction	Soil, Tissue & Sed.		3550	
5. Florisil Cleanup	Pest, PCB, & ClHC		3620	
6. Silica Gel Cleanup	PAH		3630	
7. Sulfur Cleanup	Sed.		3660	
8. Volatile Organics	Water	624		
	Water & Solid Ext.		8260	
9. Semivol. Organics(BNA)	Water	625		
	Water & Solid Ext.		8270	
10. Cl-Pest & PCBs	Water	608		
	Water & Solid Ext.		8080	
11. Polynuclear Aromatic HC	Water	610		
	Water & Solid Ext		9100	
12. Organophos Pesticides	Water & Solid ext.		8140	509A
13. Chlorinated Herbicides	Water & Solid Ext.		8150	509B

Other Methods as Requested Such as Hydrazine, Nitrosoamines, & TNT

Detection limits for organic analysis are typically those given for the methods. They are, however, subject to matrix interferences and may be higher depending on the nature of the interference.

Method 608 Detection Limits

value 2.44 used in the equation in Section 8.3.3 is two times the value 1.22 derived in this report.)

11. ASTM Annual Book of Standards, Part 31, D3370-76, "Standard Practices for Sampling Water," American Society for Testing and Materials, Philadelphia.

12. "Methods 330.4 (Titrimetric, DPD-FAS) and 330.5 (Spectrophotometric, DPD) for Chlorine, Total Residual," Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268, March 1979.

13. Goerlitz, D.F., and Law, L.M. *Bulletin for Environmental Contamination and Toxicology, 6,* 9 (1971).

14. "Manual of Analytical Methods for the Analysis of Pesticides in Human and Environmental Samples," EPA-600/8-80-038, U.S. Environmental Protection Agency, Health Effects Research Laboratory, Research Triangle Park, North Carolina.

15. Burke, J.A. "Gas Chromatography for Pesticide Residue Analysis; Some Practical Aspects," *Journal of the Association of Official Analytical Chemists, 48,* 1037 (1965).

16. Webb, R.G., and McCall, A.C. "Quantitative PCB Standards for Election Capture Gas Chromatography," *Journal of Chromatographic Science, 11,* 366 (1973).

17. "Method Detection Limit and Analytical Curve Studies, EPA Methods 606, 607, and 608," Special letter report for EPA Contract 68-03-2606, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268.

18. "EPA Method Validation Study 18, Method 608 (Organochlorine Pesticides and PCBs)," Report for EPA Contract 68-03-2606 (In preparation).

TABLE 1.—CHROMATOGRAPHIC CONDITIONS AND METHOD DETECTION LIMITS

Parameter	Retention time (min)		Method detection limit (µg/L)
	Col. 1	Col. 2	
α-BHC	1.35	1.82	0.003
γ-BHC	1.70	2.13	0.00
β-BHC	1.90	1.97	0.00
Heptachlor	2.00	3.35	0.003
δ-BHC	2.15	2.20	0.009
Aldrin	2.40	4.10	0.004
Heptachlor epoxide	3.50	5.00	0.083
Endosulfan I	4.50	6.20	0.014
4,4'-DDE	5.13	7.15	0.004
Dieldrin	5.45	7.23	0.002
Endrin	6.55	8.10	0.006
4,4'-DDD	7.83	9.08	0.011
Endosulfan II	8.00	8.28	0.004
4,4'-DDT	9.40	11.75	0.012
Endrin aldehyde	11.82	9.30	0.023
Endosulfan sulfate	14.22	10.70	0.066
Chlordane	mr	mr	0.014
Toxaphene	mr	mr	0.24
PCB-1016	mr	mr	nd
PCB-1221	mr	mr	nd
PCB-1232	mr	mr	nd

TABLE 1.—CHROMATOGRAPHIC CONDITIONS AND METHOD DETECTION LIMITS—Continued

Parameter	Retention time (min)		Method detection limit (µg/L)
	Col. 1	Col. 2	
PCB-1242	mr	mr	0.065
PCB-1248	mr	mr	nd
PCB-1254	mr	mr	nd
PCB-1260	mr	mr	nd

Column 1 conditions: Supelcoport (100/120 mesh) coated with 1.5% SP-2250/1.95% SP-2401 packed in a 1.8 m long × 4 mm ID glass column with 5% methane/95% argon carrier gas at 60 mL/min flow rate. Column temperature held isothermal at 200 °C, except for PCB-1016 through PCB-1248, should be measured at 160 °C.

Column 2 conditions: Supelcoport (100/120 mesh) coated with 3% OV-1 packed in a 1.8 m long × 4 mm ID glass column with 5% methane/95% argon carrier gas at 60 mL/min flow rate. Column temperature held isothermal at 200 °C for the pesticides; at 140 °C for PCB-1221 and 1232; and at 170 °C for PCB-1016 and 1242 to 1268.

mr = Multiple peak response. See Figures 2 thru 10.

nd = Not determined.

TABLE 2.—DISTRIBUTION OF CHLORINATED PESTICIDES AND PCBs INTO FLORISIL COLUMN FRACTIONS [2]

Parameter	Percent recovery by fraction [a]		
	1	2	3
Aldrin	100		
α-BHC	100		
β-BHC	97		
δ-BHC	98		
γ-BHC	100		
Chlordane	100		
4,4'-DDD	99		
4,4'-DDE	98		
4,4'-DDT	100		
Dieldrin	0	100	
Endosulfan I	37	64	
Endosulfan II	0	7	91
Endosulfan sulfate	0	0	106
Endrin	4	96	
Endrin aldehyde	0	68	26
Heptachlor	100		
Heptachlor epoxide	100		
Toxaphene	96		
PCB-1016	97		
PCB-1221	97		
PCB-1232	95	4	
PCB-1242	97		
PCB-1248	103		
PCB-1254	90		
PCB-1260	95		

[a] Eluant composition:
Fraction 1-6% ethyl ether in hexane.
Fraction 2-15% ethyl ether in hexane.
Fraction 3-50% ethyl ether in hexane.

TABLE 3.—QC ACCEPTANCE CRITERIA—METHOD 608

Parameter	Test conc. (µg/L)	Limit for s (µg/L)	Range for X̄ (µg/L)	Range for P, P_s(%)
Aldrin	2.0	0.42	1.08-2.24	42-122
α-BHC	2.0	0.48	.98-2.44	37-134
β-BHC	2.0	0.64	0.78-2.60	17-147
δ-BHC	2.0	0.72	1.01-2.37	19-140
γ-BHC	2.0	0.46	0.86-2.32	32-127
Chlordane	50	10.0	27.6-54.3	45-119
4,4'-DDD	10	2.8	4.8-12.6	31-141
4,4'-DDE	2.0	0.55	1.08-2.60	30-145

TABLE 3.—QC ACCEPTANCE CRITERIA—METHOD 608—Continued

Parameter	Test conc. (µg/L)	Limit for s (µg/L)	Range for X̄ (µg/L)	Range for P, P_s(%)
4,4'-DDT	10	3.6	4.6-13.7	25-160
Dieldrin	2.0	0.76	1.15-2.49	36-146
Endosulfan I	2.0	0.49	1.14-2.82	45-153
Endosulfan II	10	3.1	2.2-17.1	D-202
Endosulfan Sulfate	10	2.7	3.8-13.2	26-144
Endrin	10	3.7	5.1-12.6	30-147
Heptachlor	2.0	0.40	0.86-2.00	34-111
Heptachlor epoxide	2.0	0.41	1.13-2.63	37-142
Toxaphene	50	12.7	27.8-55.6	41-125
PCB-1016	50	10.0	30.5-51.5	50-114
PCB-1221	50	24.4	22.1-75.2	15-178
PCB-1232	50	17.9	14.0-98.5	10-215
PCB-1242	50	12.2	24.8-69.6	39-150
PCB-1248	50	15.9	29.0-70.2	39-158
PCB-1254	50	13.8	22.2-57.9	29-131
PCB-1260	50	10.4	18.7-54.9	8-127

s = Standard deviation of four recovery measurements, in µg/L (Section 8.2.4).

X̄ = Average recovery for four recovery measurements, in µg/L (Section 8.2.4).

P, P_s = Percent recovery measured (Section 8.3.2, Section 8.4.2).

D = Detected; result must be greater than zero.

Note.—These criteria are based directly upon the method performance data in Table 4. Where necessary, the limits for recovery have been broadened to assure applicability of the limits to concentrations below those used to develop Table 4.

TABLE 4. METHOD ACCURACY AND PRECISION AS FUNCTIONS OF CONCENTRATION—METHOD 608

Parameter	Accuracy, as recovery, X' (µg/L)	Single analyst precision, s_r (µg/L)	Overall precision, S' (µg/L)
Aldrin	0.81C + 0.04	0.16X̄ − 0.04	0.20X̄ − 0.01
α-BHC	0.84C + 0.03	0.13X̄ + 0.04	0.23X̄ − 0.00
β-BHC	0.81C + 0.07	0.22X̄ − 0.02	0.33X̄ − 0.95
δ-BHC	0.81C + 0.07	0.18X̄ + 0.09	0.25X̄ + 0.03
γ-BHC	0.82C − 0.05	0.12X̄ + 0.06	0.22X̄ + 0.04
Chlordane	0.82C − 0.04	0.13X̄ + 0.13	0.18X̄ + 0.18
4,4'-DDD	0.84C + 0.30	0.20X̄ − 0.18	0.27X̄ − 0.14
4,4'-DDE	0.85C + 0.14	0.13X̄ + 0.06	0.28X̄ − 0.09
4,4'-DDT	0.93C − 0.13	0.17X̄ + 0.39	0.31X̄ − 0.21
Dieldrin	0.90C + 0.02	0.12X̄ + 0.19	0.16X̄ + 0.16
Endosulfan I	0.97C + 0.04	0.10X̄ + 0.07	0.18X̄ + 0.08
Endosulfan II	0.93C + 0.34	0.41X̄ − 0.65	0.47X̄ − 0.20
Endosulfan Sulfate	0.89C − 0.37	0.13X̄ + 0.33	0.24X̄ + 0.35
Endrin	0.89C − 0.04	0.20X̄ + 0.25	0.24X̄ + 0.25
Heptachlor	0.69C + 0.04	0.06X̄ + 0.13	0.16X̄ + 0.08
Heptachlor epoxide	0.89C + 0.10	0.18X̄ − 0.11	0.25X̄ − 0.08
Toxaphene	0.80C + 1.74	0.09X̄ + 3.20	0.20X̄ + 0.22
PCB-1016	0.81C + 0.50	0.13X̄ + 0.15	0.15X̄ + 0.45
PCB-1221	0.96C + 0.65	0.29X̄ − 0.76	0.35X̄ − 0.62
PCB-1232	0.91C + 10.79	0.21X̄ − 1.93	0.31X̄ + 3.50
PCB-1242	0.93C + 0.70	0.11X̄ + 1.40	0.21X̄ + 1.52
PCB-1248	0.97C + 1.06	0.17X̄ + 0.41	0.25X̄ − 0.37
PCB-1254	0.76C + 2.07	0.15X̄ + 1.66	0.17X̄ + 3.62
PCB-1260	0.66C + 3.76	0.22X̄ − 2.37	0.39X̄ − 4.86

X' = Expected recovery for one or more measurements of a sample containing a concentration of C, in µg/L.

s_r' = Expected single analyst standard deviation of measurements at an average concentration found of X̄, in µg/L.

S' = Expected interlaboratory standard deviation of measurements at an average concentration found of X̄, in µg/L.

C = True value for the concentration, in µg/L.

X̄ = Average recovery found for measurements of samples containing a concentration of C, in µg/L.

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Method 624 Detection Limits

from the EICP of the primary characteristic m/z given in Table 4. If the sample produces an interference for the primary m/z, use a secondary characteristic m/z to quantitate.

Calculate the concentration in the sample using the response factor (RF) determined in Section 7.3.3 and Equation 2.

Equation 2.

$$\text{Concentration } (\mu g/L) = \frac{(A_s)(C_{is})}{(A_{is})(RF)}$$

where:

A_s = Area of the characteristic m/z for the parameter or surrogate standard to be measured.

A_{is} = Area of the characteristic m/z for the internal standard.

C_{is} = Concentration of the internal standard.

13.2 Report results in µg/L without correction for recovery data. All QC data obtained should be reported with the sample results.

14. Method Performance

14.1 The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero.[1] The MDL concentrations listed in Table 1 were obtained using reagent water.[11] Similar results were achieved using representative wastewaters. The MDL actually achieved in a given analysis will vary depending on instrument sensitivity and matrix effects.

14.2 This method was tested by 15 laboratories using reagent water, drinking water, surface water, and industrial wastewaters spiked at six concentrations over the range 5–600 µg/L.[12] Single operator precision, overall precision, and method accuracy were found to be directly related to the concentration of the parameter and essentially independent of the sample matrix. Linear equations to describe these relationships are presented in Table 5.

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1. 40 CFR Part 136, Appendix B.
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6. "Safety in Academic Chemistry Laboratories," American Chemical Society Publication, Committee on Chemical Safety, 3rd Edition, 1979.
7. Provost, L.P., and Elder, R.S. "Interpretation of Percent Recovery Data," *American Laboratory, 15*, 58–63 (1983). (The value 2.44 used in the equation in Section 8.2.3 is two times the value 1.22 derived in this report.)
8. "Methods 330.4 (Titrimetric, DPD-FAS) and 330.5 (Spectrophotometric, DPD) for Chlorine, Total Residual," Methods for Chemical Analysis of Water and Wastes, EPA–600/4–79–020, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268, March 1979.
9. Budde, W.L., and Eichelberger, J.W. "Performance Tests for the Evaluation of Computerized Gas Chromatography/Mass Spectrometry Equipment and Laboratories," EPA–600/4–80–025, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268, April 1980.
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11. "Method Detection Limit for Methods 624 and 625," Olynyk, P., Budde, W.L., and Eichelberger, J.W. Unpublished report, October 1980.
12. "Interlaboratory Method Study for EPA Method 624—Purgeables," Final Report for EPA Contract 68–03–3102.
13. "Method Performance Data for Method 624," Memorandum from R. Slater and T. Pressley, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268, January 17, 1984.

TABLE 1.—CHROMATOGRAPHIC CONDITIONS AND METHOD DETECTION LIMITS

Parameter	Retention time (min)	Method detection limit (µg/L)
Chloromethane	2.3	nd
Bromomethane	3.1	nd
Vinyl chloride	3.8	nd
Chloroethane	4.6	nd
Methylene chloride	6.4	2.8
Trichlorofluoromethane	8.3	nd
1,1-Dichloroethene	9.0	2.8
1,1-Dichloroethane	10.1	4.7
trans-1,2-Dichloroethene	10.8	1.6
Chloroform	11.4	1.6
1,2-Dichloroethane	12.1	2.8
1,1,1-Trichloroethane	13.4	3.8
Carbon tetrachloride	13.7	2.8
Bromodichloromethane	14.3	2.2
1,2-Dichloroproane	15.7	6.0
cis-1,3-Dichloropropene	15.9	5.0
Trichloroethene	16.5	1.9
Benzene	17.0	4.4
Dibromochloromethane	17.1	3.1
1,1,2-Trichloroethane	17.2	5.0
trans-1,3-Dichloropropene	17.2	nd
2-Chloroethylvinyl ether	18.6	nd
Bromoform	19.8	4.7
1,1,2,2-Tetrachloroethane	22.1	6.9
Tetrachloroethene	22.2	4.1

TABLE 1.—CHROMATOGRAPHIC CONDITIONS AND METHOD DETECTION LIMITS—Continued

Parameter	Retention time (min)	Method detection limit (µg/L)
Toluene	23.5	
Chlorobenzene	24.6	
Ethyl benzene	26.4	
1,3-Dichlorobenzene	33.9	
1,2-Dichlorobenzene	35.0	
1,4-Dichlorobenzene	35.4	

Column conditions: Carbopak B (60/80 mesh) coated with 1% SP-1000 packed in a 6 ft by 0.1 in. ID glass column with helium carrier gas at 30 mL/min flow rate. Column temperature held at 45°C for 3 min, then programmed at 8°C/min to 220°C and held for 15 min.

nd = not determined.

TABLE 2.—BFB KEY M/Z ABUNDANCE CRITERIA

Mass	m/z Abundance criteria
50	15 to 40% of mass 95.
75	30 to 60% of mass 95.
95	Base Peak, 100% Relative Abundance.
96	5 to 9% of mass 95.
173	<2% of mass 174.
174	>50% of mass 95.
175	5 to 9% of mass 174.
176	>95% but <101% of mass 174.
177	5 to 9% of mass 176.

TABLE 3.—SUGGESTED SURROGATE AND INTERNAL STANDARDS

Compound	Retention time (min)*	Primary m/z	Secondary masses
Benzene d-6	17.0	84	
4-Bromofluorobenzene	28.3	95	174, 17
1,2-Dichloroethane d-4	12.1	102	
1,4-Difluorobenzene	19.6	114	63, 8
Ethylbenzene d-5	26.4	111	
Ethylbenzene d-10	26.4	98	
Fluorobenzene	18.4	96	7
Pentafluorobenzene	23.5	168	
Bromochloromethane	9.3	128	49, 130, 5
2-Bromo-1-chloropropane	19.2	77	79, 15
1, 4-Dichlorobutane	25.8	55	90, 9

*For chromatographic conditions, see Table 1.

TABLE 4.—CHARACTERISTIC MASSES FOR PURGEABLE ORGANICS

Parameter	Primary	Secondary
Chloromethane	50	52.
Bromomethane	94	96.
Vinyl chloride	62	64.
Chloroethane	64	66.
Methylene chloride	84	49, 51, and 86.
Trichlorofluoromethane	101	103.
1,1-Dichloroethene	96	61 and 98.
1,1-Dichloroethane	63	65, 83, 85, 98, and 100.
trans-1,2-Dichloroethene	96	61 and 98.
Chloroform	83	85.
1,2-Dichloroethane	98	62, 64, and 100.
1,1,1-Trichloroethane	97	99, 117, and 119.
Carbon tetrachloride	117	119 and 121.
Bromodichloromethane	127	83, 85, and 129.
1,2-Dichloropropane	112	63, 65, and 114.
trans-1,3-Dichloropropene	75	77.
Trichloroethene	130	95, 97, and 132.
Benzene	78	
Dibromochloromethane	127	129, 208, and 206.
1,1,2-Trichloroethane	97	83, 85, 99, 132, and 134.
cis-1,3-Dichloropropene	75	77.
2-Chloroethylvinyl ether	106	63 and 65

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Method 625 Detection Limits

be obtained only from a properly equipped laboratory through the use of EPA Method 613 or other approved alternate test procedures.

References

1. 40 CFR Part 136, Appendix B.
2. "Sampling and Analysis Procedures for Screening of Industrial Effluents for Priority Pollutants." U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268, March 1977. Revised April 1977. Available from Effluent Guidelines Division, Washington, DC 20460.
3. ASTM Annual Book of Standards, Part 31, D3694–78, "Standard Practices for Preparation of Sample Containers and for Preservation of Organic Constituents," American Society for Testing and Materials, Philadelphia.
4. "Carcinogens—Working With Carcinogens," Department of Health, Education, and Welfare, Public Health Service, Center for Disease Control, National Institute for Occupational Safety and Health, Publication No. 77–206, August 1977.
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10. Eichelberger, J.W., Harris, L.E., and Budde, W.L. "Reference Compound to Calibrate Ion Abundance Measurement in Gas Chromatography-Mass Spectometry," *Analytical Chemistry*, 47, 995 (1975).
11. McNair, N.M. and Bonelli, E.J. "Basic Chromatography," Consolidated Printing, Berkeley, California, p. 52, 1969.
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14. "Interlaboratory Method Study for EPA Method 625—Base/Neutrals, Acids, and Pesticides," Final report for EPA Contract 68–03–3102 (In preparation).

TABLE 1.—BASE/NEUTRAL EXTRACTABLES

Parameter	STORET No.	CAS No.
Acenaphthene	34205	83-32-9
Acenaphthylene	34200	208-96-8
Anthracene	34220	120-12-7
Aldrin	39330	309-00-2
Benzo(a)anthracene	34526	56-55-3
Benzo(b)fluoranthene	34230	205-99-2
Benzo(k)fluoranthene	34242	207-08-9
Benzo(a)pyrene	34247	50-32-8
Benzo(ghi)perylene	34521	191-24-2
Benzyl butyl phthalate	34292	85-68-7
β-BHC	39338	319-85-7
δ-BHC	34259	319-86-8
Bis(2-chloroethyl)ether	34273	111-44-4
Bis(2-chloroethoxy)methane	34278	111-91-1
Bis(2-ethylhexyl)phthalate	39100	117-81-7
Bis(2-chloroisopropyl)ether	34283	108-60-1
4-Bromophenyl phenyl ether	34636	101-55-3
Chlordane	39350	57-74-9
2-Chloronaphthalene	34581	91-58-7
4-Chlorophenyl phenyl ether	34641	7005-72-3
Chrysene	34320	218-01-9
4,4'-DDD	39310	72-54-8
4,4'-DDE	39320	72-55-9
4,4'-DDT	39300	50-29-3
Dibenzo(a,h)anthracene	34556	53-70-3
Di-n-butylphthalate	39110	84-74-2
1,3-Dichlorobenzene	34566	541-73-1
1,2-Dichlorobenzene	34536	95-50-1
1,4-Dichlorobenzene	34571	106-46-7
3,3'-Dichlorobenzidine	34631	91-94-1
Dieldrin	39380	60-57-1
Diethyl phthalate	34336	84-66-2
Dimethyl phthalate	34341	131-11-3
2,4-Dinitrotoluene	34611	121-14-2
2,6-Dinitrotoluene	34626	606-20-2
Di-n-octylphthalate	34596	117-84-0
Endosulfan sulfate	34351	1031-07-8

TABLE 1.—BASE/NEUTRAL EXTRACTABLES—Continued

Parameter	STORET No.	CAS No.
Endrin aldehyde	34366	7421-93-4
Fluoranthene	34376	206-44-0
Fluorene	34381	86-73-7
Heptachlor	39410	76-44-8
Heptchlor epoxide	39420	1024-57-3
Hexachlorobenzene	39700	118-74-1
Hexachlorobutadiene	34391	87-68-3
Hexachloroethane	34396	67-72-1
Indeno(1,2,3-cd)pyrene	34403	193-39-5
Isophorone	34408	78-59-1
Naphthalene	34696	91-20-3
Nitrobenzene	34447	98-95-3
N-Nitrosodi-n-propylamine	34428	621-64-7
PCB-1016	34671	12674-11-2
PCB-1221	39488	11104-28-2
PCB-1232	39492	11141-16-5
PCB-1242	39496	53469-21-9
PCB-1248	39500	12672-29-6
PCB-1254	39504	11097-69-1
PCB-1260	39508	11096-82-5
Phenanthrene	34461	85-01-8
Pyrene	34469	129-00-0
Toxaphene	39400	8001-35-2
1,2,4-Trichlorobenzene	34551	120-82-1

TABLE 2.—ACID EXTRACTABLES

Parameter	STORET No.	CAS No.
4-Chloro-3-methylphenol	34452	59-50-7
2-Chlorophenol	34586	95-57-8
2,4-Dichlorophenol	34601	120-83-2
2,4-Dimethylphenol	34606	105-67-9
2,4-Dinitrophenol	34616	51-28-5
2-Methyl-4,6-dinitrophenol	34657	534-52-1
2-Nitrophenol	34591	88-75-5
4-Nitrophenol	34646	100-02-7
Pentachlorophenol	39032	87-86-5
Phenol	34694	108-95-2
2,4,6-Trichlorophenol	34621	88-06-2

TABLE 3.—ADDITIONAL EXTRACTABLE PARAMETERS*

Parameter	STORET No.	CAS No.	Method
Benzidine	39120	92-87-5	605
β-BHC	39337	319-84-6	608
δ-BHC	39340	58-89-8	608
Endosulfan I	34361	959-98-8	608
Endosulfan II	34356	33213-65-9	608
Endrin	39390	72-20-8	608
Hexachlorocyclopentadiene	34386	77-47-4	612
N-Nitrosodimethylamine	34438	62-75-9	607
N-Nitrosodiphenylamine	34433	86-30-6	607

* See Section 1.2.

TABLE 4.—CHROMATOGRAPHIC CONDITIONS, METHOD DETECTION LIMITS, AND CHARACTERISTIC MASSES FOR BASE/NEUTRAL EXTRACTABLES

Parameter	Retention time (min)	Method detection limit (µg/L)	Characteristic masses					
			Electron impact			Chemical ionization		
			Primary	Secondary	Secondary	Methane	Methane	Methane
1,3-Dichlorobenzene	7.4	1.9	146	148	113	146	148	150
1,4-Dichlorobenzene	7.8	4.4	146	148	113	146	148	150
Hexachloroethane	8.4	1.6	117	201	199	199	201	203
Bis(2-chloroethyl) ether	8.4	5.7	93	63	95	63	107	109
1,2-Dichlorobenzene	8.4	1.9	146	148	113	146	148	150
Bis(2-chloroisopropyl) ether	9.3	5.7	45	77	79	77	135	137
N-Nitrosodi-n-propylamine			130	42	101			
Nitrobenzene	11.1	1.9	77	123	65	124	152	164
Hexachlorobutadiene	11.4	0.9	225	223	227	223	225	227
1,2,4-Trichlorobenzene	11.6	1.9	180	182	145	181	183	209
Isophorone	11.9	2.2	82	95	138	139	167	178
Naphthalene	12.1	1.6	128	129	127	129	157	169
Bis(2-chloroethoxy) methane	12.2	5.3	93	95	123	65	107	137
Hexachlorocyclopentadiene*	13.9		237	235	272	235	237	239
2-Chloronaphthalene	15.9	1.9	162	164	127	163	191	203
Acenaphthylene	17.4	3.5	152	151	153	152	153	181

TABLE 4.—CHROMATOGRAPHIC CONDITIONS, METHOD DETECTION LIMITS, AND CHARACTERISTIC MASSES FOR BASE/NEUTRAL EXTRACTABLES—Continued

Parameter	Retention time (min)	Method detection limit (µg/L)	Characteristic masses: Electron impact: Primary	Electron impact: Secondary	Electron impact: Secondary	Chemical ionization: Methane	Chemical ionization: Methane	Chemical ionization: Methane
Acenaphthene	17.8	1.9	154	153	152	154	155	153
Dimethyl phthalate	18.3	1.6	163	194	164	151	163	164
2,6-Dinitrotoluene	18.7	1.9	165	89	121	193	211	223
Fluorene	19.5	1.9	166	165	167	166	167	195
4-Chlorophenyl phenyl ether	19.5	4.2	204	206	141			
2,4-Dinitrotoluene	19.8	5.7	165	63	182	183	211	223
Diethylphthalate	20.1	1.9	149	177	150	177	223	251
N-Nitrosodiphenylamine*	20.5	1.9	169	168	167	169	170	198
Hexachlorobenzene	21.0	1.9	284	142	249	284	286	288
β-BHC*	21.1		183	181	109			
4-Bromophenyl phenyl ether	21.2	1.9	248	250	141	249	251	277
δ-BHC*	22.4		183	181	109			
Phenanthrene	22.8	5.4	178	179	176	178	179	207
Anthracene	22.8	1.9	178	179	176	178	179	207
β-BHC	23.4	4.2	181	183	109			
Heptachlor	23.4	1.9	100	272	274			
δ-BHC	23.7	3.1	183	109	181			
Aldrin	24.0	1.9	66	263	220			
Dibutyl phthalate	24.7	2.5	149	150	104	149	205	279
Heptachlor epoxide	25.6	2.2	353	355	351			
Endosulfan I*	26.4		237	338	341			
Fluoranthene	26.5	2.2	202	101	100	203	231	243
Dieldrin	27.2	2.5	79	263	279			
4,4'-DDE	27.2	5.6	246	248	176			
Pyrene	27.3	1.9	202	101	100	203	231	243
Endrin*	27.9		81	263	82			
Endosulfan II*	28.6		237	339	341			
4,4'-DDD	28.6	2.8	235	237	165			
Benzidine*	28.8	44	184	92	185	185	213	225
4,4'-DDT	29.3	4.7	235	237	165			
Endosulfan sulfate	29.8	5.6	272	387	422			
Endrin aldehyde			67	345	250			
Butyl benzyl phthalate	29.9	2.5	149	91	206	149	299	327
Bis(2-ethylhexyl) phthalate	30.6	2.5	149	167	279	149		
Chrysene	31.5	2.5	228	226	229	228	229	257
Benzo(a)anthracene	31.5	7.8	228	229	226	228	229	257
3,3'-Dichlorobenzidine	32.2	16.5	252	254	126			
Di-n-octyl phthalate	32.5	2.5	149					
Benzo(b)fluoranthene	34.9	4.8	252	253	125	252	253	281
Benzo(k)fluoranthene	34.9	2.5	252	253	125	252	253	281
Benzo(a)pyrene	36.4	2.5	252	253	125	252	253	281
Indeno(1,2,3-c,d)pyrene	42.7	3.7	276	138	277	276	277	305
Dibenzo(a,h)anthracene	43.2	2.5	278	139	279	278	279	307
Benzo(ghi)perylene	45.1	4.1	276	138	277	276	277	305
N-Nitrosodimethylamine*			42	74	44			
Chlordane*	19–30		373	375	377			
Toxaphene*	25–34		159	231	233			
PCB 1016*	18–30		224	260	294			
PCB 1221*	15–30	30	190	224	260			
PCB 1232*	15–32		190	224	260			
PCB 1242*	15–32		224	260	294			
PCB 1248*	12–34		294	330	262			
PCB 1254*	22–34	36	294	330	362			
PCB 1260*	23–32		330	362	394			

* See Section 1.2.
* These compounds are mixtures of various isomers. (See figures 2 thru 12.)

Column conditions: Supecoport (100/120 mesh) coated with 3% SP-2250 packed in a 1.8 m long × 2mm ID glass column with helium carrier gas at 30 mL/min flow rate. Column temperature held isothermal at 50 °C for 4 min, then programmed at 8 °C/min to 270 °C and held for 30 min.

TABLE 5.—CHROMATOGRAPHIC CONDITIONS, METHOD DETECTION LIMITS, AND CHARACTERISTIC MASSES FOR ACID EXTRACTABLES

Parameter	Retention time (min)	Method detection limit (µg/L)	Characteristic masses: Electron Impact: Primary	Electron Impact: Secondary	Electron Impact: Secondary	Chemical ionization: Methane	Chemical ionization: Methane	Chemical ionization: Methane
2-Chlorophenol	5.9	3.3	128	64	130	129	131	157
2-Nitrophenol	6.5	3.6	139	65	109	140	168	122
Phenol	8.0	1.5	94	65	66	95	123	135
2,4-Dimethylphenol	9.4	2.7	122	107	121	123	151	163
2,4-Dichlorophenol	9.8	2.7	162	164	98	163	165	167
2,4,6-Trichlorophenol	11.8	2.7	196	198	200	197	199	201
4-Chloro-3-methylphenol	13.2	3.0	142	107	144	143	171	183
2,4-Dinitrophenol	15.9	42	184	63	154	185	213	225
2-Methyl-4,6-dinitrophenol	16.2	24	198	182	77	199	227	239
Pentachlorophenol	17.5	3.6	266	264	268	267	265	269
4-Nitrophenol	20.3	2.4	65	139	109	140	168	122

Column conditions: Supelcoport (100/120 mesh) coated with 1% SP-1240DA packed in a 1.8 m long x 2mm ID glass column with helium carrier gas at 30 mL/min flow rate. Column temperature held isothermal at 70 °C for 2 min then programmed at 8 °C/min to 200 °C.

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CLP Quantitation Limits

Target Compound List (TCL) and
Contract Required Quantitation Limits (CRQL)*

Volatiles	CAS Number	Quantitation Limits** Water ug/L	Low Soil/Sediment[a] ug/Kg
1. Chloromethane	74-87-3	10	10
2. Bromomethane	74-83-9	10	10
3. Vinyl Chloride	75-01-4	10	10
4. Chloroethane	75-00-3	10	10
5. Methylene Chloride	75-09-2	5	5
6. Acetone	67-64-1	10	10
7. Carbon Disulfide	75-15-0	5	5
8. 1,1-Dichloroethene	75-35-4	5	5
9. 1,1-Dichloroethane	75-34-3	5	5
10. 1,2-Dichloroethene (total)	540-59-0	5	5
11. Chloroform	67-66-3	5	5
12. 1,2-Dichloroethane	107-06-2	5	5
13. 2-Butanone	78-93-3	10	10
14. 1,1,1-Trichloroethane	71-55-6	5	5
15. Carbon Tetrachloride	56-23-5	5	5
16. Vinyl Acetate	108-05-4	10	10
17. Bromodichloromethane	75-27-4	5	5
18. 1,2-Dichloropropane	78-87-5	5	5
19. cis-1,3-Dichloropropene	10061-01-5	5	5
20. Trichloroethene	79-01-6	5	5
21. Dibromochloromethane	124-48-1	5	5
22. 1,1,2-Trichloroethane	79-00-5	5	5
23. Benzene	71-43-2	5	5
24. trans-1,3-Dichloropropene	10061-02-6	5	5
25. Bromoform	75-25-2	5	5
26. 4-Methyl-2-pentanone	108-10-1	10	10
27. 2-Hexanone	591-78-6	10	10
28. Tetrachloroethene	127-18-4	5	5
29. Toluene	108-88-3	5	5
30. 1,1,2,2-Tetrachloroethane	79-34-5	5	5

(continued)

Volatiles	CAS Number	Quantitation Limits** Water ug/L	Low Soil/Sediment[a] ug/Kg
31. Chlorobenzene	108-90-7	5	5
32. Ethyl Benzene	100-41-4	5	5
33. Styrene	100-42-5	5	5
34. Xylenes (Total)	1330-20-7	5	5

[a] Medium Soil/Sediment Contract Required Quantitation Limits (CRQL) for Volatile TCL Compounds are 125 times the individual Low Soil/Sediment CRQL.

* Specific quantitation limits are highly matrix dependent. The quantitation limits listed herein are provided for guidance and may not always be achievable.

** Quantitation limits listed for soil/sediment are based on wet weight. The quantitation limits calculated by the laboratory for soil/sediment, calculated on dry weight basis as required by the contract, will be higher.

Target Compound List (TCL) and
Contract Required Quantitation Limits (CRQL)*

Semivolatiles	CAS Number	Quantitation Limits** Water ug/L	Low Soil/Sediment[5] ug/Kg
35. Phenol	108-95-2	10	330
36. bis(2-Chloroethyl) ether	111-44-4	10	330
37. 2-Chlorophenol	95-57-8	10	330
38. 1,3-Dichlorobenzene	541-73-1	10	330
39. 1,4-Dichlorobenzene	106-46-7	10	330
40. Benzyl alcohol	100-51-6	10	330
41. 1,2-Dichlorobenzene	95-50-1	10	330
42. 2-Methylphenol	95-48-7	10	330
43. bis(2-Chloroisopropyl) ether	108-60-1	10	330
44. 4-Methylphenol	106-44-5	10	330
45. N-Nitroso-di-n-dipropylamine	621-64-7	10	330
46. Hexachloroethane	67-72-1	10	330
47. Nitrobenzene	98-95-3	10	330
48. Isophorone	78-59-1	10	330
49. 2-Nitrophenol	88-75-5	10	330
50. 2,4-Dimethylphenol	105-67-9	10	330
51. Benzoic acid	65-85-0	50	1600
52. bis(2-Chloroethoxy) methane	111-91-1	10	330
53 2,4-Dichlorophenol	120-83-2	10	330
54. 1,2,4-Trichlorobenzene	120-82-1	10	330
55. Naphthalene	91-20-3	10	330
56. 4-Chloroaniline	106-47-8	10	330
57. Hexachlorobutadiene	87-68-3	10	330
58. 4-Chloro-3-methylphenol (para-chloro-meta-cresol)	59-50-7	10	330
59. 2-Methylnaphthalene	91-57-6	10	330
60. Hexachlorocyclopentadiene	77-47-4	10	330
61. 2,4,6-Trichlorophenol	88-06-2	10	330
62. 2,4,5-Trichlorophenol	95-95-4	50	1600
63. 2-Chloronaphthalene	91-58-7	10	330
64. 2-Nitroaniline	88-74-4	50	1600
65. Dimethylphthalate	131-11-3	10	330
66. Acenaphthylene	208-96-8	10	330
67. 2,6-Dinitrotoluene	606-20-2	10	330
68. 3-Nitroaniline	99-09-2	50	1600
69. Acenaphthene	83-32-9	10	330

(continued)

Semivolatiles	CAS Number	Quantitation Limits** Water ug/L	Low Soil/Sediment[b] ug/Kg
70. 2,4-Dinitrophenol	51-28-5	50	1600
71. 4-Nitrophenol	100-02-7	50	1600
72. Dibenzofuran	132-64-9	10	330
73. 2,4-Dinitrotoluene	121-14-2	10	330
74. Diethylphthalate	84-66-2	10	330
75. 4-Chlorophenyl-phenyl ether	7005-72-3	10	330
76. Fluorene	86-73-7	10	330
77. 4-Nitroaniline	100-01-6	50	1600
78. 4,6-Dinitro-2-methylphenol	534-52-1	50	1600
79. N-nitrosodiphenylamine	86-30-6	10	330
80. 4-Bromophenyl-phenylether	101-55-3	10	330
81. Hexachlorobenzene	118-74-1	10	330
82. Pentachlorophenol	87-86-5	50	1600
83. Phenanthrene	85-01-8	10	330
84. Anthracene	120-12-7	10	330
85. Di-n-butylphthalate	84-74-2	10	330
86. Fluoranthene	206-44-0	10	330
87. Pyrene	129-00-0	10	330
88. Butylbenzylphthalate	85-68-7	10	330
89. 3,3'-Dichlorobenzidine	91-94-1	20	660
90. Benzo(a)anthracene	56-55-3	10	330
91. Chrysene	218-01-9	10	330
92. bis(2-Ethylhexyl)phthalate	117-81-7	10	330
93. Di-n-octylphthalate	117-84-0	10	330
94. Benzo(b)fluoranthene	205-99-2	10	330
95. Benzo(k)fluoranthene	207-08-9	10	330
96. Benzo(a)pyrene	50-32-8	10	330
97. Indeno(1,2,3-cd)pyrene	193-39-5	10	330
98. Dibenz(a,h)anthracene	53-70-3	10	330
99. Benzo(g,h,i)perylene	191-24-2	10	330

b Medium Soil/Sediment Contract Required Quantitation Limits (CRQL) for SemiVolatile TCL Compounds are 60 times the individual Low Soil/Sediment CRQL.

* Specific quantitation limits are highly matrix dependent. The quantitation limits listed herein are provided for guidance and may not always be achievable.

** Quantitation limits listed for soil/sediment are based on wet weight. The quantitation limits calculated by the laboratory for soil/sediment, calculated on dry weight basis as required by the contract, will be higher.

Target Compound List (TCL) and
Contract Required Quantitation Limits (CRQL)*

Pesticides/PCBs	CAS Number	Quantitation Limits** Water ug/L	Low Soil/Sediment[c] ug/Kg
100. alpha-BHC	319-84-6	0.05	8.0
101. beta-BHC	319-85-7	0.05	8.0
102. delta-BHC	319-86-8	0.05	8.0
103. gamma-BHC (Lindane)	58-89-9	0.05	8.0
104. Heptachlor	76-44-8	0.05	8.0
105. Aldrin	309-00-2	0.05	8.0
106. Heptachlor epoxide	1024-57-3	0.05	8.0
107. Endosulfan I	959-98-8	0.05	8.0
108. Dieldrin	60-57-1	0.10	16.0
109. 4,4'-DDE	72-55-9	0.10	16.0
110. Endrin	72-20-8	0.10	16.0
111. Endosulfan II	33213-65-9	0.10	16.0
112. 4,4'-DDD	72-54-8	0.10	16.0
113. Endosulfan sulfate	1031-07-8	0.10	16.0
114. 4,4'-DDT	50-29-3	0.10	16.0
115. Methoxychlor	72-43-5	0.5	80.0
116. Endrin ketone	53494-70-5	0.10	16.0
117. alpha-Chlordane	5103-71-9	0.5	80.0
118. gamma-Chlordane	5103-74-2	0.5	80.0
119. Toxaphene	8001-35-2	1.0	160.0
120. Aroclor-1016	12674-11-2	0.5	80.0
121. Aroclor-1221	11104-28-2	0.5	80.0
122. Aroclor-1232	11141-16-5	0.5	80.0
123. Aroclor-1242	53469-21-9	0.5	80.0
124. Aroclor-1248	12672-29-6	0.5	80.0
125. Aroclor-1254	11097-69-1	1.0	160.0
126. Aroclor-1260	11096-82-5	1.0	160.0

[c] Medium Soil/Sediment Contract Required Quantitation Limits (CRQL) for Pesticide/PCB TCL compounds are 15 times the individual Low Soil/Sediment CRQL.

* Specific quantitation limits are highly matrix dependent. The quantitation limits listed herein are provided for guidance and may not always be achievable.

** Quantitation limits listed for soil/sediment are based on wet weight. The quantitation Limits calculated by the laboratory for soil/sediment, calculated on dry weight basis as required by the contract, will be higher.

SW-846 Limits

TABLE 1. GAS CHROMATOGRAPHY OF PESTICIDES AND PCBs[a]

Compound	Retention time (min) Col. 1	Retention time (min) Col. 2	Method Detection limit (ug/L)
Aldrin	2.40	4.10	0.004
α-BHC	1.35	1.82	0.003
β-BHC	1.90	1.97	0.006
δ-BHC	2.15	2.20	0.009
γ-BHC (Lindane)	1.70	2.13	0.004
Chlordane (technical)	e	e	0.014
4,4'-DDD	7.83	9.08	0.011
4,4'-DDE	5.13	7.15	0.004
4,4'-DDT	9.40	11.75	0.012
Dieldrin	5.45	7.23	0.002
Endosulfan I	4.50	6.20	0.014
Endosulfan II	8.00	8.28	0.004
Endosulfan sulfate	14.22	10.70	0.066
Endrin	6.55	8.10	0.006
Endrin aldehyde	11.82	9.30	0.023
Heptachlor	2.00	3.35	0.003
Heptachlor epoxide	3.50	5.00	0.083
Methoxychlor	18.20	26.60	0.176
Toxaphene	e	e	0.24
PCB-1016	e	e	nd
PCB-1221	e	e	nd
PCB-1232	e	e	nd
PCB-1242	e	e	0.065
PCB-1248	e	e	nd
PCB-1254	e	e	nd
PCB-1260	e	e	nd

[a]U.S. EPA. Method 617. Organochloride Pesticides and PCBs. Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268.

e = Multiple peak response.

nd = not determined.

TABLE 2.
PRACTICAL QUATITATION LIMITS (PQL) FOR VOLATILE ORGANICS[a]

Volatiles	Practical Quantitation Limits[b] Ground water ug/L	Low Soil/Sediment ug/kg
Acetone	100	100
Acetonitrile	100	100
Allyl chloride	5	5
Benzene	5	5
Benzyl chloride	100	100
Bromodichloromethane	5	5
Bromoform	5	5
Bromomethane	10	10
2-Butanone	100	100
Carbon disulfide	100	100
Carbon tetrachloride	5	5
Chlorobenzene	5	5
Chlorodibromomethane	5	5
Chloroethane	10	10
2-Chloroethyl vinyl ether	10	10
Chloroform	5	5
Chloromethane	10	10
Chloroprene	5	5
1,2-Dibromo-3-chloropropane	100	100
1,2-Dibromoethane	5	5
Dibromomethane	5	5
1,4-Dichloro-2-butene	100	100
Dichlorodifluoromethane	5	5
1,1-Dichloroethane	5	5
1,2-Dichloroethane	5	5
1,1 Dichloroethene	5	5
trans-1,2-Dichloroethene	5	5
1,2-Dichloropropane	5	5
cis-1,3-Dichloropropene	5	5
trans-1,3-Dichloropropene	5	5
Ethylbenzene	5	5
Ethyl methacrylate	5	5
2-Hexanone	50	50
Isobutyl alcohol	100	100
Methacrylonitrile	100	100
Methylene chloride	5	5
Methyl iodide	5	5
Methyl methacrylate	5	50
4-Methyl-2-pentanone	50	50
Pentachloroethane	10	10

TABLE 2.
(Continued)

Volatiles	Practical Quantitation Limits[b] Ground water ug/L	Low Soil/Sediment ug/kg
Propionitrile	100	100
Styrene	5	5
1,1,1,2-Tetrachloroethane	5	5
1,1,2,2-Tetrachloroethane	5	5
Tetrachloroethene	5	5
Toluene	5	5
1,1,1-Trichloroethane	5	5
1,1,2-Trichloroethane	5	5
Trichloroethene	5	5
1,2,3-Trichloropropane	5	5
Vinyl acetate	50	50
Vinyl chloride	10	10
Xylene (Total)	5	5

[a]Sample PQLs are highly matrix-dependent. The PQLs listed herein are provided for guidance and may not always be achieveable. See the following information for further guidance on matrix-dependent PQLs.

[b]PQLs listed for soil/sediment are based on wet weight. Normally data is reported on a dry_weight basis; therefore, PQLs will be higher, based on the percent moisture in each sample.

Other Matrices:	Factor[1]
Water miscible liquid waste	50
High-level soil & sludges	125
Non-water miscible waste	500

[1]PQL = [PQL for ground water (Table 2)] X [Factor]. For non-aqueous samples, the factor is on a wet-weight basis.

TABLE 1.
CHROMATOGRAPHIC RETENTION TIMES AND METHOD DETECTION LIMITS (MDL)
FOR VOLATILE ORGANIC COMPOUNDS ON WIDE BORE CAPILLARY COLUMNS

ANALYTE	RETENTION TIME (minutes) Column 1[a]	Column 2[b]	Column 3[c]	MDL[d] (ug/L)
Dichlorodifluoromethane	1.55	0.70	--	0.10
Chloromethane	1.63	0.73	2.07	0.13
Vinyl Chloride	1.71	0.79	2.12	0.17
Bromomethane	2.01	0.96	2.26	0.11
Chloroethane	2.09	1.02	2.31	0.10
Trichlorofluoromethane	2.27	1.19	2.42	0.08
1,1-Dichloroethane	2.89	1.57	3.08	0.12
Methylene chloride	3.60	2.06	3.32	0.03
trans-1,2-Dichloroethene	3.98	2.36	3.48	0.06
1,1-Dichloroethane	4.85	2.93	4.10	0.04
2,2-Dichloropropane	6.01	3.80	4.43	0.35
cis-1,2-Dichloroethene	6.19	3.90	4.42	0.12
Chloroform	6.40	4.80	4.58	0.03
Bromochloromethane	6.74	4.38	4.54	0.04
1,1,1-Trichloroethane	7.27	4.84	5.09	0.08
Carbon tetrachloride	7.61	5.26	5.18	0.21
1,1-Dichloropropene	7.68	5.29	5.18	0.10
Benzene	8.23	5.67	5.29	0.04
1,2-Dichloroethane	8.40	5.83	5.29	0.06
Trichloroethene	9.59	7.27	6.07	0.19
1,2-Dichloropropane	10.09	7.66	6.20	0.04
Bromodichloromethane	10.59	8.49	6.39	0.08
Dibromomethane	10.65	7.93	6.27	0.24
Toluene	12.43	10.00	7.36	0.11
1,1,2-Trichloroethane	13.41	11.05	8.07	0.10
Tetrachloroethene	13.74	11.15	8.21	0.14
1,3-Dichloropropane	14.04	11.31	8.20	0.04
Dibromochloromethane	14.39	11.85	8.39	0.05
1,2-Dibromoethane	14.73	11.83	--	0.06
1-Chlorohexane	15.46	13.29	--	0.05
Chlorobenzene	15.76	13.01	9.33	0.04
1,1,1,2-Tetrachloroethane	15.94	13.33	9.41	0.05
Ethylbenzene	15.99	13.39	9.44	0.06
p-Xylene	16.12	13.69	9.56	0.13
m-Xylene	16.17	13.68	9.56	0.05
o-Xylene	17.11	14.52	10.32	0.11
Styrene	17.31	14.60	10.33	0.04
Bromoform	17.93	14.88	10.48	0.12
Isopropylbenzene	18.06	15.46	--	0.15
1,1,2,2-Tetrachloroethane	18.72	16.35	11.38	0.04
Bromobenzene	18.95	15.86	11.35	0.03

TABLE 1.
(Continued)

ANALYTE	RETENTION TIME (minutes) Column 1[a]	Column 2[b]	Column 3[c]	MDL[d] (ug/L)
1,2,3-Trichloropropane	19.02	16.23	11.40	0.32
n-Propylbenzene	19.06	16.41	--	0.04
2-Chlorotoluene	19.34	16.42	11.57	0.04
1,3,5-Trimethylbenzene	19.47	16.90	--	0.05
4-Chlorotoluene	19.50	16.72	12.08	0.06
tert-Butylbenzene	20.28	17.57	--	0.14
1,2,4-Trimethylbenzene	20.34	17.70	--	0.13
sec-Butylbenzene	20.79	18.09	--	0.13
p-Isopropyltoluene	21.20	18.52	--	0.12
1,3-Dichlorobenzene	21.22	18.14	13.16	0.12
1,4-Dichlorobenzene	21.55	18.39	13.27	0.03
n-Butylbenzene	22.22	19.49	--	0.11
1,2-Dichlorobenzene	22.52	19.17	14.10	0.03
1,2-Dibromo-3-chloropropane	24.53	21.08	--	0.26
1,2,4-Trichlorobenzene	26.55	23.08	--	0.04
Hexachlorobutadiene	26.99	23.68	--	0.11
Naphthalene	27.17	23.52	--	0.04
1,2,3-Trichlorobenzene	27.78	24.18	--	0.03
INTERNAL STANDARDS/SURROGATES				
4-Bromofluorobenzene	18.63	15.71	11.22	

[a]Column 1 - 60 meter x 0.75 mm i.d. VOCOL capillary. Hold at 10°C for 5 minutes, then program to 160°C at 6°/min.

[b]Column 2 - 30 meter x 0.53 mm i.d. DB-624 wide-bore capillary using cryogenic oven. Hold at 10°C for 5 minutes, then program to 160°C at 6°/min.

[c]Column 3 - 30 meter x 0.53 i.d. DB-624 wide-bore capillary, cooling GC oven to ambient temperatures. Hold at 45°C for 2 minutes, then program to 200°C at 8°/min and hold for 6 minutes.

[d]MDL based on a 25 mL sample volume.

TABLE 2.
PRACTICAL QUANTITATION LIMITS (PQL) FOR SEMIVOLATILE ORGANICS[A]

Semivolatiles	Practical Quantitation Limits[b] Ground Water ug/L	Low Soil/Sediment[1] ug/kg
Acenapthene	10	660
Acenaphthylene	10	660
Acetophenone	10	ND
2-Acetylaminofluorene	20	ND
1-Acetyl-2-thiourea	1000	ND
2-Aminoanthraquinone	20	ND
Aminoazobenzene	10	ND
4-Aminobiphenyl	20	ND
Anilazine	100	ND
o-Anisidine	10	ND
Anthracene	10	660
Aramite	20	ND
Azinphos-methyl	100	ND
Barban	200	ND
Benz(a)anthracene	10	660
Benzo(b)fluoranthene	10	660
Benzo(k)fluoranthene	10	660
Benzoic acid	50	3300
Benzo(g,h,i)perylene	10	660
Benzo(a)pyrene	10	660
p-Benzoquinone	10	ND
Benzyl alcohol	20	1300
Bis(2-chloroethoxy) methane	10	660
Bis(2-chloroethyl) ether	10	660
Bis(2-chloroisopropyl) ether	10	660
4-bromophenyl phenyl ether	10	660
Bromoxynil	10	ND
Butyl benzyl phthalate	10	660
Captafol	20	ND
Captan	50	ND
Carbaryl	10	ND
Carbofuran	10	ND
Carbophenothion	10	ND
Chlorfenvinphos	20	ND
4-Chloroaniline	20	1300
Chlorobenzilate	10	ND
5-Chloro-2-methylaniline	10	ND
4-Chloro-3-methylphenol	20	1300
3-(Chloromethyl) pyridine hydrochloride	100	ND
2-Chloroanaphthalene	10	660
2-Chlorophenol	10	660
4-Chlorophenyl phenyl ether	10	660

TABLE 2.
(Continued)

Semivolatiles	Practical Quantitation Limits[b] Ground Water ug/L	Low Soil/Sediment[1] ug/kg
Chrysene	10	660
Coumaphos	40	ND
p-Cresidine	10	ND
Crotoxyphos	20	ND
2-Cyclohexyl-4,6-dinitrophenol	100	ND
Demeton-o	10	ND
Demeton-s	10	ND
Diallate (cis or trans)	10	ND
Diallate (trans or cis)	10	ND
2,4-Diaminotoluene	20	ND
Dibenz(a,j)acridine	10	ND
Dibenz(a,h)anthracene	10	660
Dibenzofuran	10	660
Dibenzo(a,e)pyrene	10	ND
Di-n-butylphthalate	10	ND
Dichlone	NA	ND
1,2-Dichlorobenzene	10	660
1,3-Dichlorobenzene	10	660
1,4-Dichlorobenzene	10	660
3,3'-Dichlorobenzidine	20	1300
2,4-Dichlorophenol	10	660
2,6-Dichlorophenol	10	ND
Dichlorovos	10	ND
Dicrotophos	10	ND
Diethylphthalate	10	660
Diethylstilbesterol	20	ND
Diethyl sulfate	100	ND
Dimethoate	20	ND
3,3'-Dimethoxybenzidine	100	ND
Dimethylaminoazobenzene	10	ND
7,12-Dimethylbenz(a)anthracene	10	ND
3,3'-Dimethylbenzidine	10	ND
a,a-Dimethylphenethylamine	ND	ND
2,4-Dimethylphenol	10	660
Dimethyl phthalate	10	660
1,2-Dinitrobenzene	40	ND
1,3-Dinitrobenzene	20	ND
1,4-Dinitrobenzene	40	ND
4,6-Dinitro-2-methylphenol	50	3300
2,4-Dinitrophenol	50	3300
2,4-Dinitrotoluene	10	660
2,6-Dinitrotoluene	10	660
Dinocap	100	ND

TABLE 2.
(Continued)

Semivolatiles	Practical Quantitation Limits[b] Ground Water ug/L	Low Soil/Sediment[1] ug/kg
Dinoseb	20	ND
5,5-Diphenylhydantoin	20	ND
Di-n-octyl phthalate	10	660
Disulfoton	10	ND
EPN	10	ND
Ethion	10	ND
Ethyl carbamate	50	ND
Bis(2-ethylhexyl)phthalate	10	660
Ethyl methanesulfonate	20	ND
Famphur	20	ND
Fensulfothion	40	ND
Fenthion	10	ND
Fluchloralin	20	ND
Fluoranthene	10	660
Fluorene	10	660
Hexachlorobenzene	10	660
Hexachlorobutadiene	10	660
Hexachlorocyclopentadiene	10	660
Hexachloroethane	10	660
Hexachlorophene	50	ND
Hexachloropropene	10	ND
Hexamethyl phosphoramide	20	ND
Hydroquinone	ND	ND
Indeno(1,2,3-cd)pyrene	10	660
Isodrin	20	ND
Isophorone	10	660
Isosafrole	10	ND
Kepone	20	ND
Leptophos	10	ND
Malathion	50	ND
Maleic anhydride	NA	ND
Mestranol	20	ND
Methapyrilene	100	ND
Methoxychlor	10	ND
3-Methylcholanthrene	10	ND
4,4'-Methylenebis(2-chloroaniline)	NA	ND
Methylmethanesulfonate	10	ND
2-Methylnaphthalene	10	660
Methyl parathion	10	ND
2-Methylphenol	10	660
3-Methylphenol	10	ND
4-Methylphenol	10	660
Mevinphos	10	ND

TABLE 2.
(Continued)

Semivolatiles	Practical Quantitation Limits[b] Ground Water ug/L	Low Soil/Sediment[1] ug/kg
Mexacarbate	20	ND
Mirex	10	ND
Monocrotophos	40	ND
Naled	20	ND
Naphthalene	10	660
1,4-Naphthoquinone	10	ND
1-Naphthylamine	10	ND
2-Naphthylamine	10	ND
Nicotine	20	ND
5-Nitroacenaphthene	10	ND
2-Nitroaniline	50	3300
3-Nitroaniline	50	3300
4-Nitroaniline	20	ND
5-Nitro-o-anisidine	10	ND
Nitrobenzene	10	660
4-Nitrobiphenyl	10	ND
Nitrofen	20	ND
2-Nitrophenol	10	660
4-Nitrophenol	50	3300
5-Nitro-o-toluidine	10	ND
4-Nitroquinoline-1-oxide	40	ND
N-Nitrosodibutylamine	10	ND
N-Nitrosodiethylamine	20	ND
N-Nitrosodiphenylamine	10	660
N-Nitroso-di-n-propylamine	10	660
N-Nitrosopiperidine	20	ND
N-Nitrosopyrrolidine	40	ND
Octamethyl pyrophosphoramide	200	ND
4,4'-Oxydianiline	20	ND
Parathion	10	ND
Pentachlorobenzene	10	ND
Pentachloronitrobenzene	20	ND
Pentachlorophenol	50	3300
Phenacetin	20	ND
Phenanthrene	10	660
Phenobarbital	10	ND
Phenol	10	660
1,4-Phenylenediamine	10	ND
Phorate	10	ND
Phosalone	100	ND
Phosmet	40	ND
Phosphamidon	100	ND
Phthalic anhydride	100	ND
2-Picoline	ND	ND

TABLE 2.
(Continued)

Semivolatiles	Practical Quantitation Limits[b] Ground Water ug/L	Low Soil/Sediment[1] ug/kg
Piperonyl sulfoxide	100	ND
Pronamide	10	ND
Propylthiouracil	100	ND
Pyrene	10	660
Pyridine	ND	ND
Resorcinol	100	ND
Safrole	10	ND
Strychnine	40	ND
Sulfallate	10	ND
Terbufos	20	ND
1,2,4,5-Tetrachlorobenzene	10	ND
2,3,4,6-Tetrachlorophenol	10	ND
Tetrachlorvinphos	20	ND
Tetraethyl pyrophosphate	40	ND
Thionazine	20	ND
Thiophenol (Benzenethiol)	20	ND
Toluene diisocyanate	100	ND
o-Toluidine	10	ND
1,2,4-Trichlorobenzene	10	660
2,4,5-Trichlorophenol	10	660
2,4,6-Trichlorophenol	10	660
Trifluralin	10	ND
2,4,5-Trimethylaniline	10	ND
Trimethyl phosphate	10	ND
1,3,5-Trinitrobenzene	10	ND
Tris(2,3-dibromopropyl) phosphate	200	ND
Tri-p-tolyl phosphate(h)	10	ND
0,0,0-Triethylphosphorothioate	NT	ND

[a] PQLs listed for soil/sediment are based on wet weight. Normally data is reported on a dry weight basis, therefore, PQLs will be higher based on the % moisture in each sample. This is based on a 30-g sample and gel permeation chromatography cleanup.

[b] Sample PQLs are highly matrix-dependent. The PQLs listed herein are provided for guidance and may not always be achievable.

ND = Not determined.
NA = Not applicable.
NT = Not tested.

Other Matrices	Factor[1]
Medium-level soil and sludges by sonicator	7.5
Non-water miscible waste	75

[1]PQL = [PQL for Low Soil/Sediment (Table 2)] X [Factor].

Appendix E

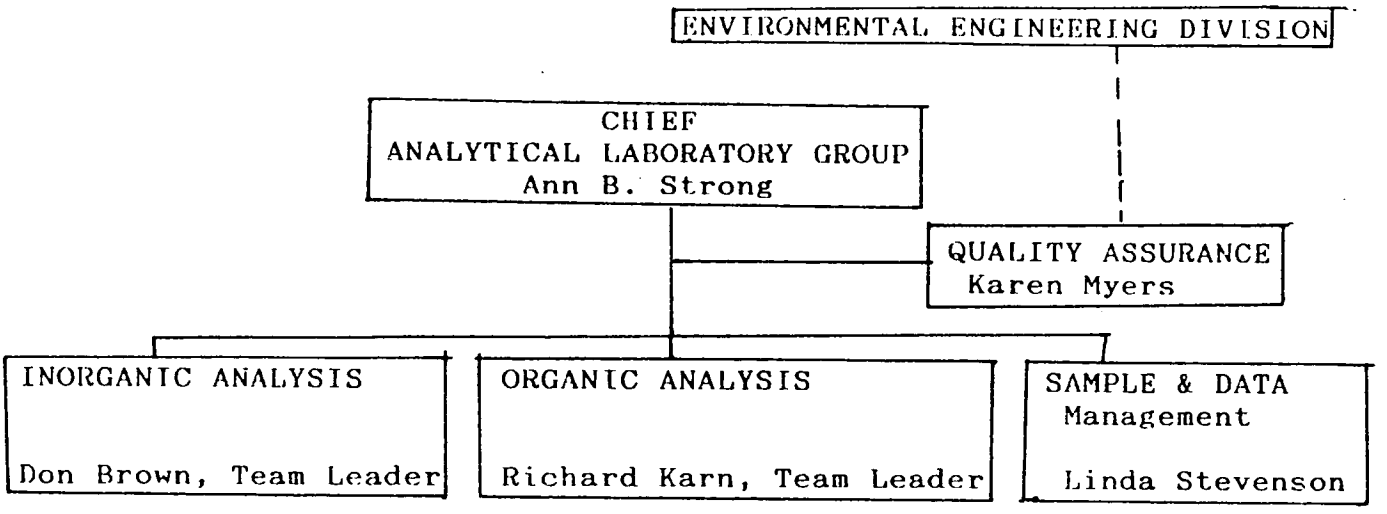
WES ALG Instrumentation

Instrument	Model	Year	Analyses
Autoanalyzers	Technicon GT-PC Models (3) 15 Channels	1986	Nutrients, cyanide, Alkalinity, Phenols Cloride, Fluoride
DC Argon Plasma Emission Spectrometer	Spectra Span IV	1982	Metals (>30 ppb)
ICAP Emmission Spectrometer	Leeman Labs Plasma Spec III Seq/Simul	1988	Metals
Atomic Absorption Spectrophotometers	Perkin-Elmer 5000 with Auto Samplers Model 500 Graphite furnaces (2)	1979 1981	Metals
	Perkin-Elmer 3030 Zeeman with auto-Sampler	1986	Metals
	Perkin-Elmer 5100 with Zeeman Bkg correction, auto-sampler and graphite furnace	1989	Metals
Gas Chromatographs	Hewlett-Packard 5840A with auto-sampler, EC & flame photometric detectors	1978	Pesticides, PCB's, herbicides
	Hewlett-Packard 5880A with auto-sampler, dual EC detectors, dual nitrogen-phosphorus & dual FID detectors	1984	PCB's, Pesticides, herbicides, PAH's,
	Perkin-Elmer Sigma-3 with thermal Conductivity detector	1978	Gases
	Tracor 540 with auto-sampler, dual EC detectors & Nelson Analytical data reduction system	1987	PCBs, pesticides herbicides, PAHs

6/89

Gas Chromatograph/ Mass Spectrometer/ Data Systems	Hewlett-Packard 5985 with auto sampler, EI, CI, both positive and negative capability Upgraded	1980 1988	Organics
GC/MS (cont.)	Hewlett-Packard 5995 with EI, purge and trap Upgraded	1982 1988	Volatile organic compounds
High Performance Liquid Chromatograph	Waters	1978	Organic compounds
FT/IR	Perkin-Elmer Model 1600	1989	Total Recoverable Petroleum Hydro-carbons, Organic identities
Cabon Analyzer	Oceanographics International Model 700	1987	Total carbon, organic and inorganic
Spectrometer, Scanning UV/Vis	Hitachi Model 100-60	1977	Sulfates hydrocarbons Carbohydrates
Gold Foil Mercury Analyzer	Jerome Instrument	1987	Mercury

Appendix F



DUTIES

Ann B. Strong, GM 13, Chemist supervises overall activities of the ALG, including final check of all data prior to submission to customer.

Richard Karn, GS-12, Chemist has oversight for all organic analysis and is GC/MS expert.

Don Brown, GS-11, Chemist schedules all inorganic analysis and is an experienced analyst using the autoanalyzer, the DCP, the ICAP and the GFAA.

Linda Stevenson, GS-6, Physical Science Technician is responsible for sample and data management keeping track of all samples received, computer entry and tracking of all samples, fiscal reports, data reports.

Newberry Brown, GS-11, Chemist is primary gas chromatograph operator.

Glennard Warren, GS-11, Chemist is primary GFAA operator and also performs autoanalyzer analyses.

Karen Myers, GS-11, Biologist is experienced in operation of DCP and GC and serves as QA officer. (on long term leave September 1988-August 1989)

Agnes Morrow, GS-09, Chemist is experienced in organic extractions, and is secondary GC operator, also performs IR analyses.

Jeffretha Christian, GS-06, Physical Science Technician performs mercury and TOC analyses under Don Brown's oversight.

Willar Tyler, IPA Chemist for 7 years, performs digestions, autoanalyzer analyses, and other wet chemistry analyses.

PROFESSIONAL RESUME'

STRONG, ANN B., SUPERVISORY RESEARCH CHEMIST

EDUCATION

B.S. - Chemistry, Mississippi College
Unversity of Florida Graduate School

PROFESSIONAL EXPERIENCE

1981 - Present	Chief, Analytical Laboratory Group, GM-13 USAE Waterways Experiment Station
1980 - 1981	Supervisory Chemist, GS-12 USAE Waterways Experiment Station
1979 - 1980	Physical Scientist, GS-12 USAE Waterways Experiment Station
1976 - 1979	Chief, Monitoring and Analytical Services, GS-13. USEPA - Eastern Environmental Radiation Facility
1971 - 1976	Chief, Analytical Services, GS-12 USEPA - Eastern Environmental Radiation Facility
1970 - 1971	Supervisor, Environmental Analysis Unit, GS-11. USEPA - Eastern Environmental Research Facility
1966 - 1970	Supervisor, Milk Section, GS-09 USPHA - Southeastern Radiological Health Lab
1962 - 1966	Chemist, GS-07 USPHS - Southern Radiological Health Lab

MAJOR PROJECTS

Project Officer for EPA's Environmental Radiation Ambient Monitoring System (EPA's nationwide monitoring program for radiation.

Supervised the EPA-ORP Analytical Laboratory, including radiochemical laboratory functions and radiation counting facilities.

Consultant for radioanalytical procedures adopted for compliance with the Safe Drinking Water Act and the Resource Conservation and Recovery Act.

Provided analytical data to be used in setting radiation standards for the Clean Air Act.

Developed procedures for determining stable elements in environmental samples by flame atomization techniques.

Project Officer for determining radionuclides in human bone.

Supervised the development work for improving radiochemical procedures for plutonium, uranium, and thorium resulting in increased productivity and accuracy.

Developed procedures for determining stable strontium in milk, food and total diet.

Prepared quality assurance program for water and wastewater analysis for Environmental Laboratory, USAE/WES.

Prepared basic quality assurance program for Lower Mississippi Valley Division-Corps of Engineers (in review).

Served as Acting Chief, Analytical Laboratory Group, December 1980 through June 1981.

Chief, Analytical Laboratory Group, 1981 - present. Supervised the chemical analysis of all samples for the Environmental Laboratory.
Manages the CE audit sample program for Superfund and DERP programs.
Manages the CE interlaboratory testing program.

MEMBERSHIPS

Association of Official Analytical Chemists, Committee on Hazardous Substances
ASTM, D-19 Committee
Water Pollution Control Federation
International Radiation Protection Association
Health Physics Society
Alabama Chapter of Health Physics (secretary--two terms)
National Subcommittee on Detection of Changes in Environmental Levels
Phi Theta Kappa Honor Society
National Committee to review and revise National Interim Primary Drinking Water Regulations

AWARDS

Who's Who in the South and Southwest - 1979
Who's Who in the South and Southwest - 1980
Who's Who in Technology Today - 1980

Who's Who in Technology Today - 1984
Significant Accomplishments Award - 1982
Significant Accomplishments Award - 1983
Significant Accomplishments Award - 1984
Exceptional Performance Award - 1987

PUBLICATIONS

Strong, Ann B., Porter, Charles R., Carter, Melvin W., and Wilson, Edward F. "Localization of Fallout in United States from May 1966 Chinese Nuclear Test," Public Health Reports, Vol 82, No. 6, June 1967.

Strong, Ann B., Rehnberg, Georgia L., and Moss, Ursula R. "Determination of Strontium in Environmental Media," Talanta, Vol 15, No. 1, 1968.

Rehnberg, Georgia L., Strong, Ann B., Porter, Charles R., and Carter, Melvin W. "Levels of Stable Strontium in Milk and Total Diet", Environmental Science and Technology, Vol 3, No. 2, February, 1969.

Butler, F. E., Lieberman, R., Strong, A. B., Moss, U. R. "Sampling and Analysis of Soils for Plutonium," Proceedings of the Environmental Plutonium Symposium, Los Alamos, NM, August 4-5, 1971.

Strong, Ann B., Porter, Charles R., Kahn, B. "Stable Strontium: Calcium Ratios in U. S. Bone and Total Diet Samples, "Second International Conference on Strontium Metabolism, USAEC Series CONF 720818, Glasgow Strontian, Great Britain, August 16-19, 1972.

Strong, Ann B. and Brooks, Imogene B. "Tritium in Surface Waters Affected by Nuclear Facilities," Proceedings of the International Conference on Radiation Effects and Tritium Technology for Fusion Reactors, 3 Volumes, October 1-5, 1975, Gatlinburg, TN.

Strong, Ann B., Smith, J. Michael, and Johnson, Raymond H., Jr. "EPA Assessment of Fallout in the United States from Atmospheric Nuclear Testing on September 26 and November 17, 1976, by the People's Republic of China," EPA 520/5-77-002, August, 1977.

Smith, J. Michael, Broadway, Jon A., and Strong, Ann B. "United States Population Dose Estimates of Iodine-131 in the Thyriod After the Chinese Atmospheric Nuclear Weapons Tests," Science, Vol 200, April 7, 1978.

Blanchard, R. L. Strong, A. B., Lieberman, R., and Porter, C. R. "The Eastern Environmental Radiation Facility's Participation in Interlaboratory Comparisons of Environmental Sample Analyses," ORP/EERF-79-2, March, 1979.

Broadway, J. A. and Strong, A. B. "Population Dose and Health

Impact Estimates Calculated from Radionuclides in Human Bone," Submitted to Science for publication.

Broadway, J. A. and Strong A. B. "Radionuclides in Human Bone (1972-1975) with Estimates of Population Dose and Health Impact. Health Physics, Vol 45, September, 1983.

Strong, Ann B. "Radiological Procedures for Environmental Samples," USEPA Technical Report (to be published).

Eicholz, G. G., Desrosiers, A. E., Kahn, B., Strong, A., Wakamo, C. L., Wilkie, W. H., and Williams, E. F. "Detection of Changes in Environmental Levels Due to Nuclear Power Plants," Upgrading Environmental Radiation Data, EPA 520/1-80-012, August 1980.

Strong, Ann B. "Laboratory Analyses." Proceedings of a seminar o: Attaining Water Quality Goals Through Water Management Procedures. 17-18 February, 1982.

Lutton, R.J., Butler, D.K.; Meade, R.J.; Patrick, D.M.; Strong, A.B.; and Taylor, H. M.; "Tests for Evaluating Sites for Disposal of Low -Level Radioactive Waste", NUREG/CR 3038 Prepared for US Nuclear Regulatory Commission by Geotechnical Laboratory, US Army Engineer Waterways Experiment Station, Vicksburg, MS 39180 December 1982

Lutton, R.J.; Strohm, W.E.; and Strong, A. B.; "Subsurface Monitoring Programs at Sites for Disposal of Low-Level Radioactive Waste" NUREG/CR-3164, Prepared for US Nuclear Regulatory Commission by Geotechnical Laboratory, US Army Engineer Waterways Experiment Station, Vicksburg, MS 39180 April 1983

Strong, Ann B. "Laboratory Inspections for Quality Assurance/Quality Control." Proceedings of a seminar on Water Quality, R&D: Successful Bridging Between Theory and Applications. 25-27 February, 1986.

Strong, Ann B.; Myers, Karen; and Warren, Glennard; "Proceedings of the Fifth Corps Chemists Meeting 15-16 May 1988" MP El 89-2, Environmental Laboratory, US Army Engineer Waterways Experiment Station, Vicksburg, MS 39180

PRESENTATIONS

"Evaluation of Rapid Field Method for the Collection of Radionuclides from Milk," presented at the Annual Meeting of the Health Physics Society, Chicago, IL, June 28-July 2, 1970

"Radiological Surveillance Around a Nuclear Power Barge in a Fresh Water Lake," presented at the annual meeting of the Health Physics Society, Las Vegas, NV, June 12-16, 1972.

"Environmental Radiation Ambient Monitoring System," presented at

EPA Symposium, Las Vegas, Nevada, February, 1973.

"Off-Site Plutonium Surveillance in the Cape Kennedy Environs," presented at the annual meeting of the Health Physics Society, June 12-16, 1972.

"Tritium in Surface Waters Affected by Nuclear Facilities," presented at the International Conference on Radiation Effects and Tritium Technology for Fusion Reactors, October 1-5, 1975, Gatlinburg, TN.

"The Distribution of Fallout in the United States from the Chinese Nuclear Tests of September 26 and November 17, 1976, as Observed by the ERAMS," presented at the annual meeting of the Health Physics Society, July 3-8, 1977, Atlanta, GA.

TRAINING & CONFERENCES

Water Quality Seminars, Dallas, TX - 1982
Superfund IPR, Washington, DC - 1982
QA for Superfund Conference, Washington, DC - 1982
Division Lab Conference, Atlanta, GA - 1983
Quality Assurance for the Analytical Laboratory NBS - 1984
Interagency Workshop and QA for Marine Pollution Measurements, NBS - 1984
Corps Chemists Seminar, NED - 1985
Pittsburgh Conference, New Orleans - 1985
Superfund - DERP Conference, Omaha - 1985
CLP Conference, EPA-Las Vegas, NV - 1985
Chemical and Biological Characterization of Sludge and Sediment EPA, Cincinatti - 1986
Water Quality Seminar, New Orleans - 1986
Chemical Quality Management Seminar for Superfund & DERA, Battelle - 1986
Division Laboratory Conference, San Francisco - 1987
Water Quality Seminar, Charleston, S.C. - 1988
Corps Chemists Meeting, WES - 1988
Annual Meeting of the Association of Official Analytical Chemists, Palm Beach, FL - 1988
Interagency Workgroup on Marine and Estuarine Sampling and Analytical Protocols, Washington, D.C. - 1988
Hazardous Waste Worker Training Program April 1989
Corps Chemists Meeting, WES - 1989
Quality Assurance in Environmental Measurements Symposium Las Vegas, NV May 1989
Nulear Radiation Safety (LSU) WES - June 1989

PROFESSIONAL RESUME'

KARN, Richard A., Research Chemist

Education

B.S. Chemistry, New Mexico Institute of Mining and Technology, 1968.

M.S. Organic Chemistry, University of Colorado, 1973.

Professional Experience

1979 - Present	Research Chemist, Analytical Laboratory Group, WES. Specialty: GC/MS, Environmental samples, Organic Team Leader
1975 - 1979	Chemist, Rocky Mountain Arsenal, Specialty: GC/MS, Development of procedures for organo-phosphorus compoun
1974 - 1975	Dugway Proving Ground Analytical testing for nerve gases
1973 - 1974	Dow Corning Corp. Process development; identification and removal of trace impurities in silicon chemical production

Major Projects

GC/MS Analyses for contaminated harbor studies - Black Rock, Indiana, and New Bedford

Investigation of TCLP procedures for volatile organics

Investigation of Organic Compounds from Rocky Mountain Arsenal

GC/MS analyses for solidification stabilization projects

Training

Basic Gas Chromatography, Hewlett Packard, 1976.

Gas Chromatography trouble-shooting, Hewlett Packard, 1976.

Basic Skills in Statistics, Denver Regional Training Center, 1978.

HP 5980 Series GC/MS Operations and Maintenance, 1978.

Glass Capillary and Priority Pollutants in Water, Hewlett Packard, 1979.

HP 5985 Mass Spec Operators Course, Hewlett Packard, 1980.

Modern Mass Spectrometry, Hewlett Packard, 1982.

Analysis of Priority Pollutants by GC/MS/DS, Hewlett Packard, 1980.

Advances in Analytical Methods for Monitoring Organic Chemicals in the Environment, EPA, 1984.

Wetland and Upland Plant and Animal Bioassay, 1985.

Hazardous Waste Worker Training Program, May, 1989

Aquarius, Hewlett-Packard, Atlanta, GA June 1989

Awards

Sustained Superior Performance, 1982.

Special Service, 1982.

Sustained Superior Performance, 1984.

Special Service, 1985.

Sustained Superior Performance, 1987

Sustained Superior Performance, 1988.

Publication

"An Improved Cyclization Procedure for Chloropropylchlorosilance: Efficient Synthesis of Silacyclobutanes"

"Unexpected Low Reactivity for Spirosilanes; 4-Silaspiro (3,3) heptane"

"Electrophilic and Nucleophilic Reactivity of 4-Silaspiro (3,3) Heptane and r-Silaspiro (3,5) Nonane"

"Toxic Metal Removal Using Silyated Silica Gel"

"Effects of Cleanup Procedures on Measured PCB Concentrations In Chicago River Sediment", 1988

"QA/QC Guidance for Organic Chemical Analysis" 1988

Presentations:

Corps Chemists Meeting, Boston, MA 1986
Corps Chemists Meeting, Cincinnati, OH 1987
Corps Chemists Meeting, WES, 1988
Corps Chemsist Meeting, WES, 1989

PROFESSIONAL RESUME'

BROWN, Donald R., Chemist

Education

B.S. Chemistry, University of Kentucky, 1973.

Professional Experience

1973 to Present	Chemist, Analytical Laboratory Group, WES. Specialty: Inorganic Analysis including Nutrients, Metals, TOC, Phenols, Cyanides, etc. Skilled in operation of Auto-analyzer, Atomic Absorption Spectrometer, DC Plasma, Inductively Coupled Argon Plasma, UV-VIS and computer applications.

Training

Micro-processing Fundamentals, New Orleans, LA, 1978.

Maintenance of Zeeman AA, Berkeley, CA, 1978.

Electronics for Chemists, Philadelphia, PA, 1978.

Hazardous Chemical Safety School, 1978.

Chemical Systems Information Network, NTIS Workshop, 1983.

Statistical Methods, Northeast Louisiana University, Tallulah Branch, 1985.

Environmental Analysis - Priority Pollutants ACS Short Course, Pittsburgh Conference, 1986.

Elementary Statistics and Probability, Mississippi College, 1986.

Statistical Data Analysis, Mississippi College, 1987.

Transportation of Hazardous Materials, U.S. Dept. of Transportation, WES, 1988.

Computer Programming, PASCAL 1. Hinds Community College, 1988.

Nuclear Radiation Safety (LSU) WES, June 1989

Memberships

American Chemical Society

Awards

Sustained Superior Performance, 1986.

Outstanding Performance, 1988.

PROFESSIONAL RESUME'

STEVENSON, Linda K.

Education

Course work at Hinds Jr. College, Vicksburg Branch to be applied to an Associate of Arts Degree in Applied Science. Physical Geology, Elementary Algebra, Intermediate Algebra, Introduction to Computer Programming, Physical Science Survey I, Introduction to Microcomputers and BASIC, Advanced BASIC Programming, Data Base Management, and Computer Programming-PASCAL 1.

Experience

May, 1976 - March, 1983	Secretary, Environmental Laboratory, WES
April, 1983 - Present	Physical Science Aid/Technician, Analytical Laboratory Group, WES

Major Duties

Responsible for data management in the ALG, logs samples into laboratory, prepares computer data work sheets, keeps sample backlog current, maintains fiscal records, prepares final data reports, performs moisture, solids, and volatiles analyses and performs extractions for Organic analysis.

Awards

Outstanding Performance 1986

Outstanding Performance 1988

PROFESSIONAL RESUME'

BROWN, B. Newberry, Chemist

Education

B.S. Delta State College, 1951
M.S. ED (Chemistry) Mississippi State University, 1965

Professional Experience

January, 1984 to Present	Chemist, Analytical Laboratory Group, WES. Specialty: Gas Chromatography
January, 1980 to December, 1984	Instructor, Wood Jr. College. Taught Freshman College Chemistry, Physics and Physical Science
December, 1976 to January, 1980	Chemist, Analytical Laboratory Group, WES. Specialty: Metals Analysis
August, 1963 to December, 1976	Instructor, Wood Jr. College. Taught Freshman College Chemistry, Physics, and Algebra
August, 1962 to August, 1963	High School Science teacher.
September, 1953 to August, 1962	Instrument mechanic for Mississippi Power & Light Co.
October, 1950 to October, 1953	Science teacher.

Major Projects

PCB analyses by Gas Chromatograph for such projects as Black Rock Harbor, Indiana Harbor, and New Bedford Harbor. Developed procedures for congener analysis.

Pesticide and herbicide analyses for various water quality projects.

PAH analysis by Gas Chromatograph.

Recent Training

Statistical Methods, Northeast Louisiana University, Tallulah Branch, 1985.
Laboratory Applications of Lotus 1,2,3 and Other Software, ACS, Denver CO April 1987
Computer Programming, Pascal I Hinds Community College, Vicksburg, MS Aug-Dec 1988

Application Oriented Basic Programming for HP 5880/3388, Hewlett Packard, 1985.

Advanced BASIC Programming, Hinds Jr. College, Vicksburg Branch, 1987.

Laboratory Applications of LOTUS 1, 2, 3 and other software, American Chemical Society, Denver, Co., 1987.

Computer Programming, PASCAL 1, Hinds Community College, Vicksburg Branch, 1988.

Awards

Sustained Superior Performance, 1985.

PROFESSIONAL RESUME'

MORROW, Agnes B.

Education

B.S. Chemistry, Alcorn State University, 1985

Experience

July, 1986 - Present	Chemist, Analytical Laboratory Group, WES. Performs distillations, extractions, etc. for organic prep for GC and GC/MS analyses. Performs oil and grease analyses. Runs GC for pesticide analysis.

Committee

Alcorn State University Committee

Training

"Fundamentals of Gas Chromatography", Hewlett-Packard, New Orleans, LA -1988

"Logical Troubleshooting for Gas Chromatography", Hewlett-Packard New Orleans, LA -1988

"Computer Programming, Pascal", Hinds Community College, Vicksburg, MS -1988

PROFESSIONAL RESUME'

MYERS, Karen L., Biologist

Education

B.S. Microbiology, University of Southern Mississippi, 1973.

One semester Graduate School, University Southern Mississippi.

Professional Experience

1982 - Present	Biologist, Analytical Laboratory Group, WES. Specialty: DC argon Plasma Emission Spectroscopy, Gas chromatography

Major Projects

Black Rock Field Verification Study

Naval Weapons Station Study

New Bedford Harbor

Training

Micro-computer Methods, Mississippi State University, WES Center, 1983.

Introduction to Data Processing, Hinds Jr. College, Vicksburg Branch, 1983.

FORTRAN Programming and Applications, Hinds Jr. College, Vicksburg Branch, 1983.

Calculus II, Hinds Jr. College, Vicksburg Branch, 1984.

Calculus III, Hinds Jr. College, Vicksburg Branch, 1984.

Calculus IV, Hinds Jr. College, Vicksburg Branch, 1984.

BASIC Programming, Hinds Jr. College, Vicksburg Branch, 1984.

Differential Equations, Hinds Jr. College, Vicksburg Branch, 1985.

Advanced Momentum, Heat, and Mass Transfer, Mississippi State University Grad Center, Vicksburg, 1985.

Introduction to Partial Differential Equations, Jackson Engineers Grad Program, 1985.

Engineering Mechanics, Hinds Community College, 1986.

Organizing and Writing Professional and Technical Communications, M. D. Morris, P.E., WES, 1987.

Awards

Outstanding Performance, 1987

Member of Phi Kappa Phi

PROFESSIONAL RESUME'

WARREN, Glennard M.

Education

B.S. Chemistry, Alcorn State University, 1979

Professional Experience

August, 1979 - Present	Chemist, Analytical Laboratory Group, WES. Specialty: Graphite Furnace AA and Auto-analysis for nutrients.

Training

Environmental Biology, Alcorn State University, Vicksburg Branch, 1980.

Environmental Microbiology, Mississippi State University, WES Center, 1981.

Basic Hydraulics, Hinds Jr. College, Vicksburg Branch, 1984.

Computer Literacy, Alcorn State University, WES Center, 1984.

Water Resources I, Mississippi State University, WES Center, 1985.

Principals of Management, Hinds Jr. College, Vicksburg Branch, 1986.

Interpersonal Relations, OPM Vicksburg, 1986.

Statistics, Alcorn State University, 1986.

Advanced Geochemistry, Jackson Engineering Graduate Program, 1987

Analytical Atomic Spectrometry, Absorption, Emission, and Mass, ACS Education Division, New Orleans, 1987.

Principles of Oral Communication, Hinds Community College, 1987.

Effective Speaking and Human Relations, Dale Carnegie, 1988.

Nuclear Radiation Safety, WES, June 1989

P

PROFESSIONAL RESUME'

CHRISTIAN, Jeffretha M.

Education

Three years college, Alcorn State University

Experience

Teaching assistant, Elementary School

Physical Science Aid/Technician — 1980 - Present, on-the-job training in total solids, suspended solids, volatile solids, Mercury by Cold vapor, total organic carbon, Elutriate Preparation and acid digestions

Training

College Algebra, Hinds Jr. College, Vicksburg Branch, 1985.

Interpersonal Communications Leadership Seminars, 1986.

Introduction to Micro-computers and BASIC, Hinds Jr. College, Vicksburg Branch, 1986.

Environmental Biology, Alcorn State University, Sept-Dec 1988

Appendix G

QA PROGRAM FOR ENVIRONMENTAL CHEMICAL ANALYSES

SAFETY POLICY

The overall safety policy of the CE is given in a number of Engineering Regulations. At the WES level, specific safety policy is delineated in Station Regulations and is under the general supervision of the Safety and Occupational Health Office staffed by a safety officer, two registered nurses and a safety technician. A medical doctor is also on duty three days a week.

At the Analytical Laboratory Group level, safety meetings are held to update employees on any changes to policy. Training sessions such as those offered by J. T. Baker are held to educate personnel in good laboratory safety practices and the use of Material Safety Data Sheets. Sessions have also been held on the proper use of respirators in the laboratory. The 40 hour training course required by OSHA for workers at hazardous waste sites is offered to pertinent personnel. Some employees have also been trained in Department of Transportation requirements for shipping hazardous materials.

Laboratory safety inspections are held on a routine basis to assure that hoods, fire extinguishers, eye baths, showers, gas outlets, and electrical outlets are all in proper working order. Training sessions in the proper use of fire extinguishers are held annually by the WES fire department. Flammable solvents must be stored in proper storage cabinets and other reagents must be stored according to chemical reactivity classification.

Laboratory wastes are disposed of according to hazard classification. All hazardous materials are collected in lab packs

until they are turned over to the WES Safety Office for final disposal. WES is a RCRA permitted facility.

Laboratory personnel are trained in spill control measures and the proper use of spill control kits. Each laboratory area has appropriate spill control kits available for procedures being conducted.

Personnel are encouraged to take advantage of free First Aid and CPR Courses.

Annual physicals are provided to laboratory personnel to encourage a healthy work environment and to assure the absence of effects from laboratory chemicals.

New personnel are given on-the-job training in routine safety procedures. All training is documented in each employee's personnel file.

TRAINING

All professional personnel hired in entry level positions (GS-5, -7) are in career intern positions. At the time they are hired, a formal training program is set up for the individual based on career goals. Specific formal training courses as well as on-the-job training are included in the program. All training is documented in the employee's personnel file.

In addition, training goals are reviewed each year at performance appraisal time. An individual development plan is prepared for all employees at that time.

STANDARD OPERATING PROCEDURE FOR GLASSWARE CLEANING

Methods for cleaning glassware are selected according to the substances that are to be removed and the analysis required.

Metals Analysis

1. Rinse with tap water immediately after use

2. Place glassware in ultrasonic cleaner filled 3/4 full with reverse osmosis (R.O.) water and 15-20 ml commercial laboratory cleaning solution(i.e. Micro or Alconox). Cleaning solution should come into contact with all surfaces.

3. When tank is full, turn on ultrasonic cleaner and allow to run 2-3 hours. Turn off cleaner prior to removing glassware.

4. Rinse 3-5 times with R.O. water.

5. Rinse in 25% hydrochloric acid bath.

6. Rinse in 25% nitric acid bath.

7. Rinse 3 times with distilled water.

8. Invert and air dry on laboratory carts.

9. Store in closed cabinets until use.

Nutrient Glassware

1. Rinse with tap water immediately after use.
2. Place in ultrasonic cleaner as above.
3. Soak overnight in 10% sulfuric acid bath
4. Rinse 3-5 times with R.O. water.
5. Rinse 3 times with distilled water.

6. Invert and air dry on laboratory carts.

7. Store in closed cabinets until use.

Ultrasonic cleaner tank should be drained every 2 weeks.

Organic Analysis

1. Rinse dirty glassware with last solvent used.

2. Rinse with tap water.

3. Place in soap bath and allow to soak at least 24 hours. (Change bath weekly--Use 1 bottle of strong basic soap such as Chem-solv to 1 full 20 gallon bath).

4. Remove from soap bath and rinse with warm tap water.

5. Rinse in 10 % hydrochloric acid bath.

6. Rinse with R.O. water.

7. Rinse with methylene chloride.

8. Place in oven overnight at 275°.

9. Cool, remove from oven, and store in closed cabinet in an inverted position or covered with aluminum foil.

10. Prior to use, the glassware is again rinsed with the solvent to be used in the analysis.

SAMPLE BOTTLE PREPARATION

WES normally purchases pre-cleaned (to EPA specifications) bottles and VOA vials for sample collection. In instances where large sample volumes are required (i.e. elutriate analysis requiring one-gallon or two and one-half gallon containers)

cleaning procedures similar to those employed for laboratory glassware are used. Certificates of contamination checks are filed and lot numbers of bottles shipped to sample collectors are maintained in a log book.

SAMPLE SHIPPING PROCEDURES

The Analytical Laboratory Group (ALG) at WES is not normally involved with the actual field collection of samples. However, the ALG frequently provides sample bottles as described above. The field crew is requested to initiate chain-of-custody procedures for projects requiring chain-of custody using appropriate forms (WES form 2196). Field personnel are requested to label and identify samples to meet project specifications. They are given instructions as to the proper containers to use with the intended analyses, the proper preservation technique, and volumes needed.

For chain-of-custody samples, shipping containers are to be sealed, whether transported by field personnel or shipped by commercial carrier. Samples are to shipped with sufficent ice to maintain a 4° C temperature and with sufficient packing to prevent breakage.

SAMPLE RECEIPT AND LOG-IN PROCEDURES

Upon receipt at the laboratory, the sample management officer fills out a cooler receipt form (copy attached).

COOLER RECEIPT FORM

PROJECT:_____

Cooler received on_____ and opened on _____ by

Signature_____

1. Were custody seals on outside of cooler?----------YES NO
   If yes, how many and where?_____
   Were signature and date correct?----------YES NO

2. Were custody papers taped to lid inside cooler-------YES NO

3. Were custody papers properly filled out(ink, signed)-YES NO

4. Did you sign custody papers in the appropriate place?YES NO

5. What kind of packing material was used?_____

6. Was sufficient ice used(if appropriate)?----------YES NO

7. Were all bottles sealed in separate plastic bags?----YES NO

8. Did all bottles arrive in good condition(unbroken)?--YES NO

9. Were all bottle labels complete(#,date, signed,anal., preserv. etc.)? YES NO

10. Did bottle labels and tags agree with custody papers?YES NO

11. Were correct bottles used for tests indicated?------YES NO

12. Were VOA vials checked for absence of bubbles?------YES NO

13. Did each bottle contain sufficient sample?----------YES NO

14. Were acid/base preserved samples checked with pH paper?----------Yes NO

Explain any discrepancies------->

Receipt of chain-of-custody samples is recorded in a permanent receipt book. The sample collector or project manager is notified of any discrepancies noted on the cooler receipt form.

If samples are delivered by WES personnel, they are required to fill out WES Form 2079 with the information indicated by asterisks and to sign a sample receipt, WES Form 2118. The ALG sample management officer then verifies that the number of samples and the sample identification are as stated on Form 2097.

If samples are received by commercial carrier, Form 2097 is filled out by the sample management officer. The WES uses preprinted consecutively numbered duplicate gummed labels to identify samples. One label is placed on the sample bottle and the other label is placed on Form 2079 under the heading Lab I.D. No. next to the corresponding Project I. D. NO. This information is also entered into a permanent log book, WES Form 2108 (copy attached). The estimated cost is calculated, entered onto the Sample Receipt Form 2118, and the original copy returned to the Project manager.

All samples except volatiles are stored in the locked walk-in cooler which is maintained at 4° and equipped with an alarm system that goes off when the temperature deviates by more than $\pm$ two degrees. A separate refrigerator is maintained for volatile analysis and and temperatures are recorded in a refrigerator log. Two ALG employees have keys to the cooler and a permanent log is maintained to check chain-of-custody samples in and out of the cooler for analysis. The analyst is required to keep chain-of-custody samples behind locked

ALG SAMPLE RECEIPT	DIVISION	BRANCH	
PROJECT NAME	JOB NO.	DATE RECEIVED	NO. SAMPLES

SAMPLE NO.	PARAMETERS	ESTIMATED COST

PROJECT REP SIGNATURE	DATE	ALG SAMPLE RECIPIENT SIGNATURE	DATE

WES FORM MAR 77 2118

COPY 1

ALG SAMPLE RECEIPT	DIVISION	BRANCH	
PROJECT NAME	JOB NO.	DATE RECEIVED	NO. SAMPLES

SAMPLE NO.	PARAMETERS	ESTIMATED COST

PROJECT REP SIGNATURE	DATE	ALG SAMPLE RECIPIENT SIGNATURE	DATE

WES FORM MAR 77 2118

COPY 2

A. L. G. SAMPLE LOG

* Chain of Custody

SAMPLE NO. 02806 TO

LAB NO.	JOB NO.	SOURCE	NO. ANALYSES / NO. SAMPLES	DATE RECEIVED	INITIAL COST	SPECIAL COST	DATE DATA RETURNED	DATE BOTTLES RETURNED	ANALYSES REQUIRED
02806-2811	CQETN383A1E6100	Calcasieu (NOD)	78 / 6	26 May 89	2154.00 Dig-50.00 Sed	Poss HGA 1260.00			Sb, As, Be, cd, cr, cu, Pb, Hg, Ni, Se, Tl, Ag, ZN
2812	"	Calcasieu (NOD)	/ 1	26 May 89	1134.00	Poss HGA 210.00			Sb, As, Be, cd, cr, Cu, Pb, Hg, Ni, Se, Tl, Ag, ZN, CN, Pests, PCBs,
2813-2814	CQEWK383A1E6100	Tenneco Pump Plant (VXD)	16 / 2	1 June 89	358.00 Sed	Poss HGA 70.00			Ca, Fe, Mg, Si, Cl, Hardness, PH, Alkalinity
2815-2832	RD9G107E1170001	APG (Wade)	/ 18	2 June 89	5400.00				PCBs
2833-2846	CQERA3831AES100	APG Air P&T Study (Zappi)	48 / 16	2 June 89	624.00				Na, Ca, Mg
2849-2863	CQERA3831AES100	APG Air P&T Study (Zappi)	15 / 15	2 June 89	300.00				Cl
2864-2878	CQERA3831AES100	APG Air P&T Study (Zappi)	15 / 15	2 June 89	300.00				TOC
2879	RD8G107E1170001	APG-Edgewood (R. Wade)	/ 1	5 June 89	300.00				PCB

ENVIRONMENTAL LABORATORY

DATA REPORTING SHEET ANALYTICAL LABORATORY GROUP

SHEET ____ OF ____

PROJECT *		DATE RECEIVED *	SAMPLE LOCATION: FREEZER NO.
DATA CHECKED BY	JOB NO. *	DATE COMPLETED	SHELF NO.

CHEMICAL PRESERVATIVE *	PARAMETER	*	*										
LAB I.D. NO	PROJECT I.D. NO *												

G-10

doors if he is away from the area where analyses are in progress.

The sample management officer then enters the information into the ALG computer data management system. Job files (example attached) are created for use by the analysts in recording their data.

SAMPLE DISPOSAL PROCEDURES

Samples are normally maintained by the ALG for a period of 6 months. At that time they are returned to the project managers if feasible. If this is not practical, the ALG requests permission from the project manager to dispose of the samples or place them in a semi-permanent repository. Samples that have been determined to contain no hazardous materials are disposed of via the sewerage system or sent to a sanitary landfill. Samples classified as hazardous are submitted to the WES Safety Office for disposal.

INSTRUMENT MAINTENANCE PROCEDURES

All Perkin-Elmer instruments (atomic absorption units) at WES are maintained by service contract that provides for twice yearly preventive maintenance and emergency repair. Permanent records are maintained of service visits.

The Plasma Emission Spectrometer (Spectrametrix) is maintained by service contract that provides for twice yearly preventive maintenance and emergency service. Permanent records are maintained of service visits.

All Hewlett-Packard Instruments (GC and GC/MS systems) are maintained by service contract that provides for twice yearly preventive maintenance and emergency repair. In addition, the GC/MS systems are tied via modem to the Analytical Response Center Support which can duplicate software problems at HP's service center. Records of all service calls are maintained in

permanent files.

All analytical balances are serviced annually by a certified balance technician.

All other instrumentation is serviced on an as needed basis and records are maintained of any repair calls.

Daily or routine maintenance of instruments is performed by the individual analysts and recorded in the instrument logs (i.e. Richard Karn is responsible for the GC/MS systems, Newberry Brown maintains the GCs, Mike Warren maintains the AAs, Don Brown maintains the ICP, DCP, and autoanalyzers, Jeffretha Christian maintains the TOC analyzer and the mercury analyzer.)

CALIBRATION PROCEDURES

Calibration of the GC/MS Systems. One instrument tune is met, the GC/MS system is calibrated before any samples or blanks are analyzed. Initial calibration is accomplished through the analysis of a minimum of five concentrations of standards. Five concentrations are used to confirm the linearity of response of the compounds examined. System calibration is verified at the start of every 12 hour analytical period. Relative response factors (RRF) are calculated for each compound based on the concentrations and areas of the characteristic ions for the compound to be measured and its assigned internal standard. Percent Relative standard (RSD) deviations are then calculated for each compound based on the mean of the initial relative response factors of the five standard concentrations. The percent RSD of the calibration check compounds listed in tables 1 and 2 are used to check calibration. The maximum

acceptable percent RSD for these compounds is 30.0.

Table 1

Volatile Calibration Check Compounds

Vinyl Chloride

1,1-Dichloroethene

Chloroform

1,2-Dichloropropane

Toluene

Ethylbenzene

Table 2

Semivolatile Calibration Check Compounds

Acenaphthene	4-Chloro-3-Methylphenol
1,4-Dichlorobenzene	2,4-Dichlorophenol
Hexachlorobutadiene	2-Nitrophenol
N-Nitroso-di-n-phenylamine	Pentachlorophenol
Di-n-octylphthalate	2,4,6-Trichlorophenol
Fluoranthene	Phenol
Benzo(a)pyrene	

In addition, system performance check compounds (SPCC) are injected to ensure that minimum RRFs are achieved. The compounds used are listed in tables 3 and 4 along with their minimum acceptable RRFs.

Table 3

Volatile System Performance Check compounds

Compound	Minimum Acceptable RRF
Chloromethane	0.300
1,1-Dichloroethane	0.30
1,1,2,2-Tetrachloroethane	0.30
Bromoform	0.25
Chlorobenzene	0.30

Table 4

Semivolatile System Performance Check Compounds

Compound	Minimum Acceptable RRF
N-Nitroso-Di-n-propylamine	0.050
Hexachlorocyclopentadiene	0.050
2,4-Dinitrophenol	0.050
4-Nitrophenol	0.050

Once the percent RSD for the CCCs and the minimum RRF for the SPCCs are met, the calibration is valid and sample analysis can begin.

Gas chromatograph calibration. A calibration Curve is determined for each of the compounds to be analyzed. Determination of the calibration curve involves preparing standards (usually three to five) from EPA standards. The samples are analyzed under normal conditions and the area under the peak is determined through integration. To determine the calibration factor, the area under the curve is divided by a standard amount (sample weight):

$$\text{Calibration factor} = \frac{\text{Area under the curve}}{\text{sample weight}}$$

To determine the variation between the Calibration Factors, a Percent Relative Deviation is calculated. The percent RSD is calculated by dividing the standard deviation of the Calibration factor by the mean of the calibration factors:

$$\%RSD = \frac{\text{Standard Deviation}}{\text{Mean}}$$

If the variation is less than 10 percent, the calibration is considered linear for all the samples analyzed for that particular compound.

Metals Analyses. Standard curves derived from data consisting of one reagent blank and three or four concentrations shall be prepared for each analyte. The response for each prepared standard shall be based upon the average of three replicate readings of each standard. the standard curve shall be used with each subsequent analysis provided that the standard curve is verified by using at least one reagent blank and one standard at a level normally encountered or expected in such samples. If the results of the verification are not within ±10% of the original curve, a reference standard should be employed to determine if the discrepancy is with the standard or the instrument. New standards should be prepared quarterly at a minimum.

Balances. Balances are checked daily using weights traceable to class S weights. Once each month the balances are checked with the Class S weights. Acceptance for balances which are direct reading to 0.01 gram shall be ±0.01 g for 0 to 100 g and ±0.1 percent of the applied weight over 100 g. A certified balance technician checks the balances annually.

DETECTION LIMITS. Procedures described in the CLP documents are used to determine ability to meet contract required detection limits (i.e. for metals, standards are run at 3 to 5 times instrument detection limits taking seven consecutive measurements on three non-consecutive days; the standard deviation is determined each day and three times the average standard deviation is used as the actual instrument detection limit.) For organic analyses, the method detection limits for the method are used, keeping in mind that the analyses are highly matrix dependent and higher detection limits are reported when interferences prohibit the reporting of the specified limits.

REFERENCE STANDARDS

The WES uses a number of sources for reference standards, including NIST (NBS) and EPA. Other Commercial sources are specified as being traceable to NIST if at all possible.

Reference Standards-Metals

NIST	SPECTROMETRIC Solutions
3104	Barium
3105	Beryllium
3107	Boron
3108	Cadmium
3112	Chromium
3113	Cobalt
3114	Copper
3128	Lead
3131	Magnesium

3132 Manganese

3133 Mercury

3134 Molybdenum

3136 Nickel

3141 Potassium

3149 Selenium

3150 Silicon

3151 Silver

3152 Sodium

3153 Strontium

3158 Thallium

3161 Tin

3165 Vanadium

3168 Zinc

3171 Multielement Solution Mix A (Al, Be, Cd, Cr, Fe, Mg, Mn, Ni, K, Na)

3172 Multielement Solution Mix B (As, Ba, Ca, Co, Cu, Pb, Se, Ag, Sr. Zn)

2124-1,-2,-3,-4 Spectrometric Standard Solutions (Co, Cu, Fe, Ni)

2126-1,-2,-3,-4 Spectrometric Standard Solutions (Sb, As, Se, Sn)

2127-1,-2,-3,-4 Spectrometric Standard Solutions (Al, Be, P, Si)

SPECPURE STANDARDS

14485A Aluminum

14487A Calcium

14375A Iron

ENVIRONMENTAL PROTECTION AGENCY

ICAP-7 (Ag, Si, B, Al, Ba, K, Na)

ICAP-19 (Sb, As, Be, Cd, Ca, Cr, Cu, Co, Fe, Pb, Mg, Mo, Ni, Se, V, Zn, Ti, Mn)

LEEMAN LABS

621-4100 Solution A (Al, Ca, Mg, Fe Traceable to EPA ICAP 7 and ICAP 19)

621-4100 Solution AB (Ca, Mg, Fe, Ag, Cd, Ni, Pb, Zn, V, Ba, Be, Co, Cr, Cu, Mn Traceable to ICAP 7 and ICAP 19)

621-4100 Interference Check Sample (Mg Traceable to NBS SRM 3131)

621-4100 Interference Check Sample (Fe Traceable to NBS SRM 3126)

621-4100 Interference Check Sample (Ca Traceable to NBS SRM 3109)

621-4100 Interference Check Sample (Al Traceable to NBS SRM 3101)

OTHER INORGANIC REFERENCE STANDARDS

NIST

3181 Anion Standard Solution Sulfate

3182 Anion Standard Solution Chloride

3183 Anion Standard Solution Chloride

ORGANIC REFERENCE STANDARDS

PURCHASED ORGANICS

SUPELCO

4-8902 Supelpreme-HC Internal Standards Mix

4-8878 Acids Spiking Solution

4-8869 Base-Neutrals Spiking Solution

4-8876 Purgeable Surrogate Standard Mix-CLP

4-8875 Acids Surrogate Standard Mix-CLP

4-8925 Base Neutral Surrogate Standard Mix-CLP

4-8990 Dibutylchlorendate

4-8835 Purgeables Internal Standard Mix-CLP

4-8908 Supelpreme-HC Hazardous Substances Mix 2

4-8902 Supelpreme-HC Internal Standards Mix

The ALG Has all of these Standards + Reference Materials THAT are available from EPA

Please Print or Type

Form Approved O.M.B. 2080-0016
4-30-89

Quality Control Sample Request

Name _____ Telephone _____
Company _____
Laboratory _____
Street _____
City _____ State _____ Zip Code _____
Approval of Laboratory Director _____
Check Activity for which samples are requested:

_____ Drinking Water _____ Wastewater _____ Ambient Monitoring _____ Toxics (TSCA) _____ Superfund (CERCLA) _____ Solid Wastes/Hazardous Wastes (RCRA)

Water Quality/Water Pollution Samples

_____ Demand

EPA/API Reference Oils
- _____ Arabian Light Crude
- _____ Prudhoe Bay Crude
- _____ South Louisiana Crude
- _____ No. 2 Fuel (high arom.)
- _____ No. 6 Fuel (high visc.) Bunker C

_____ LAS
_____ Mineral
_____ Nonionic Surfactant Std.
_____ Nutrients
_____ Oil & Grease
_____ Pesticides in Fish
_____ Phenols (4AAP Method)
_____ Suspended Solids
_____ Other _____

PCBs in Oils
- _____ Aro. 1016 in Capac.
- _____ Aro. 1016 in Hydraul.
- _____ Aro. 1016 in Trans.
- _____ Aro. 1242 in Capac.
- _____ Aro. 1242 in Hydraul.
- _____ Aro. 1242 in Trans.
- _____ Aro. 1254 in Capac.
- _____ Aro. 1254 in Hydraul.
- _____ Aro. 1254 in Trans.
- _____ Aro. 1260 in Capac.
- _____ Aro. 1260 in Hydraul.
- _____ Aro. 1260 in Trans.
- _____ Trace Metals WP - I
- _____ Trace Metals WP - II
- _____ Trace Metals WP - III
- _____ Other _____
- _____ Other _____

Water Supply Samples
- _____ WS Corrosivity/Sodium
- _____ WS Herbicides
- _____ WS Nitrate/Fluoride
- _____ WS Chl. Hyd. Pest. I
- _____ WS Chl. Hyd. Pest. II
- _____ WS Res. Free Chlorine
- _____ WS Trace Metals
- _____ WS Trihalomethanes
- _____ WS Turbidity
- _____ WS Vol. Org. Cont. - I
- _____ WS Vol. Org. Cont. - II
- _____ WS Vol. Org. Cont. - III
- _____ WS Vol. Org. Cont. - IV
- _____ WS Vol. Org. Cont. - V
- _____ WS Vol. Org. Cont. - VI
- _____ WS Vol. Org. Cont. - VII

Priority Pollutants/Hazardous Wastes/Toxic Chemicals
- _____ n-Alkanes
- _____ Chlorinated Hydrocarbons
- _____ Chl. Hyd. Pest. WP - I
- _____ Chl. Hyd. Pest. WP - II
- _____ Chl. Hyd. Pest. WP - III
- _____ Cyanide
- _____ EP Pest. & Herb.
- _____ EP Metals
- _____ GC/MS Acids
- _____ GC/MS Base Neutrals - I
- _____ GC/MS Base Neutrals - II
- _____ GC/MS Base Neutrals - III
- _____ GC/MS Pesticides - I
- _____ GC/MS Pesticides - II
- _____ Other _____

- _____ Haloethers
- _____ ICAP
- _____ Nitroaro. & Isophorone

PCBs (specific Aroclors)
- _____ Aroclor 1016
- _____ Aroclor 1221
- _____ Aroclor 1232
- _____ Aroclor 1242
- _____ Aroclor 1248
- _____ Aroclor 1254
- _____ Aroclor 1260
- _____ Phthalate Esters
- _____ Polynuclear Aromatics I
- _____ Polynuclear Aromatics II
- _____ Other _____

Biological Samples
- _____ Algae for Ident. #1
- _____ Algae for Ident. #2
- _____ Algae for Ident. #3
- _____ Algae for Ident. #4

Bacteria Indicator Strains
- _____ Enter. aerogenes
- _____ E. coli
- _____ Klebsiella pneumoniae
- _____ Pseudomonas aeruginos
- _____ Streptococcus faecalis
- _____ Sterile Lyophil. Blank
- _____ Chlorophyll Fluoro.
- _____ Chlorophyll Spectro.

Reference Toxicants
- _____ Sod. Lauryl Sulfate
- _____ Cadmium Chloride
- _____ Simulated Plankton
- _____ Other _____
- _____ Other _____

Date Requested: _____ Date Shipped: _____

EPA-360 (Cin) (Rev. 6/83, Pt. 1)

G-16-A
15

PLEASE COMPLETE THE FORM AND MAIL TO:
QUALITY ASSURANCE RESEARCH DIVISION, Room 525
EMSL-CINCINNATI
U.S. ENVIRONMENTAL PROTECTION AGENCY
CINCINNATI, OH 45268

Form Approved O.M.B. 2080-0016
4-30-89

Date Request Received_____
Laboratory Code Number_____
Request Number_____
Verified_____

The USEPA Repository for Toxic and Hazardous Materials
Request for Materials

Please Print or Type

Name_____ Telephone_____
Company_____
Laboratory_____
Street_____
City_____ State_____ Zip Code_____
Approval of Laboratory Director_____

Check Activity for which samples are requested: _____Ambient Monitoring _____Superfund (CERCLA)
_____Drinking Water _____Wastewater _____Toxics (TSCA) _____Solid Wastes/Hazardous Wastes (RCRA)

Concentrations are 5000 µg of QAS-pure compound per mL of methanol solvent unless otherwise noted.

_____ *E001 Acenaphthene*
_____ *E002 Acrolein***
_____ *E003 Acrylonitrile (10,000 µg/mL)*
_____ *E004 Benzene*
_____ *E005 Benzidine*
_____ *E006 Chlorobenzene*
_____ *E007 1,2,4-Trichlorobenzene*
_____ *E008 Hexachlorobenzene (1,000 µg/mL)**
_____ *E009 1,2-Dichloroethane*
_____ *E010 1,1,1-Trichloroethane (10,000 µg/mL) (QAR)*
_____ *E011 Hexachloroethane*
_____ *E012 1,1-Dichloroethane*
_____ *E013 1,1,2-Trichloroethane (QAR)*
_____ *E014 1,1,2,2-Tetrachloroethane (10,000 µg/mL) (QAR)*
_____ *E015 Chloroethane (11,000 µg/mL)****
_____ *E016 bis(2-Chloroethyl) ether*
_____ *E017 2-Chloroethyl vinyl ether (QAR)*
_____ *E018 2-Chloronaphthalene*
_____ *E019 2,4,6-Trichlorophenol*
_____ *E020 p-Chloro-m-cresol*
_____ *E021 Chloroform*
_____ *E022 2-Chlorophenol*
_____ *E023 1,2-Dichlorobenzene*
_____ *E025 1,4-Dichlorobenzene*
_____ *E026 3,3'-Dichlorobenzidine (QAR)*
_____ *E027 1,1-Dichloroethylene (1,000 µg/mL)*
_____ *E028 trans-1,2-Dichloroethylene (11,500 µg/mL)*
_____ *E029 2,4-Dichlorophenol*
_____ *E030 1,2-Dichloropropane (10,000 µg/mL)*
_____ *E033 2,4-Dinitrotoluene*
_____ *E034 2,6-Dinitrotoluene*
_____ *E036 Ethylbenzene (10,000 µg/mL)*
_____ *E037 Fluoranthene*
_____ *E038 4-Chlorophenyl phenyl ether*
_____ *E039 4-Bromophenyl phenyl ether*
_____ *E040 bis(2-Chloroisopropyl) ether (QAR)*
_____ *E041 bis(2-Chloroethoxy) methane (QAR)*
_____ *E042 Methylene chloride (10,000 µg/mL)*
_____ *E043 Methyl chloride****
_____ *E044 Methyl bromide (9,940 µg/mL) (QAR)****
_____ *E046 Dichlorobromomethane*
_____ *E047 Fluorotrichloromethane*
_____ *E050 Hexachlorobutadiene (QAR)*
_____ *E051 Hexachlorocyclopentadiene*
_____ *E052 Isophorone*
_____ *E053 Naphthalene*
_____ *E054 Nitrobenzene*
_____ *E055 2-Nitrophenol*
_____ *E056 4-Nitrophenol*
_____ *E057 2,4-Dinitrophenol (QAR)*
_____ *E058 4,6-Dinitro-o-cresol*
_____ *E059 N-Nitrosodimethylamine*
_____ *E060 N-Nitrosodiphenylamine*
_____ *E061 N-Nitrosodi-n-propylamine*
_____ *E062 Pentachlorophenol*
_____ *E063 Phenol*
_____ *E064 bis(2-Ethyl hexyl) phthalate*
_____ *E065 Butyl benzyl phthalate*
_____ *E066 Di-n-butyl phthalate*
_____ *E067 Di-n-octyl phthalate*

(compounds continued on reverse)

**In Acetone* ***In para-Dioxane* ****In 2-Propanol* *****In Acetonitrile* **In Methylene chloride* ***In Isooctane* ****In Cyclohexanone*

Date Requested:_____ Date Shipped:_____

PA-360 (Cin) (Rev. 6/83, Pt. 3)

16 B
G 17

_____ E068 Diethyl phthalate
_____ E069 Dimethyl phthalate
_____ E070 Benzo(a)anthracene (1,000 µg/mL)
_____ E071 Benzo(a)pyrene (1,000 µg/mL) (QAR)*
_____ E072 Benzo(b)fluoranthene (2,500 µg/mL)*
_____ E073 Benzo(k)fluoranthene (1,000 µg/mL)*
_____ E074 Chrysene (1,000 µg/mL)*
_____ E075 Acenaphthylene (QAR)
_____ E076 Anthracene (1,000 µg/mL)*
_____ E077 Benzo(g,h,i)perylene (1,000 µg/mL)**
_____ E078 Fluorene (QAR)
_____ E079 Phenanthrene
_____ E081 Indeno(1,2,3-c,d)pyrene (500 µg/mL)*
_____ E082 Pyrene (1,000 µg/mL)
_____ E083 Tetrachloroethylene
_____ E084 Toluene (10,000 µg/mL)
_____ E085 Trichloroethylene (10,000 µg/mL)
_____ E088 Dieldrin (1,000 µg/mL)
_____ E089 Chlordane (QAT)
_____ E091 4,4'-DDE
_____ E092 4,4'-DDD
_____ E093 alpha-Endosulfan (1,000 µg/mL)**
_____ E094 beta-Endosulfan (1,000 µg/mL)**
_____ E095 Endosulfan sulfate (1,000 µg/mL) (QAR)**
_____ E096 Endrin (QAR)
_____ E097 Endrin aldehyde (2,500 µg/mL)
_____ E098 Heptachlor
_____ E099 Heptachlor epoxide (2,500 µg/mL)
_____ E100 alpha-BHC (2,500 µg/mL)
_____ E101 beta-BHC (2,500 µg/mL)*
_____ E102 gamma-BHC (Lindane)
_____ E103 delta-BHC (1,000 µg/mL)
_____ E104 PCB-Aroclor 1242 (QAT)
_____ E107 PCB-Aroclor 1232 (QAT)
_____ E108 PCB-Aroclor 1248 (QAT)
_____ E110 PCB-Aroclor 1016 (QAT)
_____ E111 Toxaphene (QAT)
_____ E124 4,4'-DDT
_____ E125 PCB-Aroclor 1016 (QAT)++
_____ E126 PCB-Aroclor 1221 (QAT)
_____ E129 PCB-Aroclor 1260 (500 µg/mL) (QAT)++
_____ E129 PCB-Aroclor 1260 (1,000 µg/mL) (QAT)++
_____ E129 PCB-Aroclor 1260 (3,000 µg/mL) (QAT)++
_____ E130 PCB-Aroclor 1262 (QAT)++
_____ E131 PCB-Aroclor 1268 (2,500 µg/mL) (QAT)++
_____ E132 PCB-Aroclor 1242 (500 µg/mL) (QAT)++
_____ E132 PCB-Aroclor 1242 (1,000 µg/mL) (QAT)++
_____ E132 PCB-Aroclor 1242 (3,000 µg/mL) (QAT)++
_____ E135 PCB-Aroclor 1254 (500 µg/mL) (QAT)++
_____ E135 PCB-Aroclor 1254 (1,000 µg/mL) (QAT)++
_____ E135 PCB-Aroclor 1254 (3,000 µg/mL) (QAT)++
_____ E136 Bromochloromethane (10,000 µg/mL)
_____ E149 2,4-Dichlorotoluene
_____ E150 2-Chlorotoluene
_____ E151 3-Chlorotoluene
_____ E152 4-Chlorotoluene (QAR)
_____ E153 4-Chlorobenzotrifluoride
_____ E156 Pentachloronitrobenzene
_____ E168 alpha, alpha,2,6-Tetrachlorotoluene
_____ E169 Benzyl chloride (QAR)****
_____ E170 2,3-Dichloro-1-propylene (10,000 µg/mL)
_____ E171 1,2-Dibromoethane (EDB) (10,000 µg/mL)
_____ E173 cis-1,2-Dichloroethylene (10,000 µg/mL) (QAR)
_____ E175 1,2,3-Trichlorobenzene
_____ E176 1,3,5-Trichlorobenzene
_____ E177 1,2,4,5-Tetrachlorobenzene**** (2,500 µg/mL) (QAR)*
_____ E179 2,4,5-Trichlorophenol (QAR)
_____ E180 2,4,6-Trichloroaniline
_____ E182 3-Chlorophenol
_____ E183 4-Chlorophenol
_____ E200 Chlorodibromomethane (10,000 µg/mL) (QAR)
_____ E201 ortho-Xylene
_____ E202 meta-Xylene
_____ E203 para-Xylene
_____ E212 Bromoform (10,000 µg/mL) (QAR)
_____ E214 1,3-Dichlorobenzene
_____ E218 cis- and trans-1,3-Dichloropropylene (QAR)
_____ E219 Mirex (1,000 µg/mL)*
_____ E220 Aldrin
_____ E222 2,3,5-Trichlorophenol (QAR)
_____ E224 2,4-Dimethylphenol (QAR)
_____ E225 1,2,3,4-Tetrachlorobenzene (2,500 µg/mL)
_____ E231 Dibenzo(a,h)anthracene (1,000 µg/mL)**
_____ E236 n-Decane
_____ E237 n-Undecane
_____ E238 n-Dodecane
_____ E239 n-Tridecane
_____ E240 n-Tetradecane
_____ E241 n-Pentadecane
_____ E242 n-Heptadecane (2,500 µg/mL)
_____ E244 n-Nonadecane (1,000 µg/mL)
_____ E250 ortho-Cresol (QAR)
_____ E251 meta-Cresol (QAR)
_____ E252 para-Cresol
_____ E255 Dibutyl ether
_____ E257 Styrene
_____ E258 Epichlorohydrin****
_____ E260 Pentachlorobenzene (2,500 µg/mL)
_____ E261 Dibenzofuran
_____ E262 Diphenyl ether

(compounds continued on reverse)

*In Acetone **In para-Dioxan ***In 2-Propanol ****In Acetonitrile +++In Cyclohexanone

Date Requested:_____ Date Shipped:_____

PA-360 (Cin) (Rev. 6.83, Pt. 4)

16c
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PLEASE COMPLETE THE FORM AND MAIL TO:
QUALITY ASSURANCE RESEARCH DIVISION, Room 525
EMSL-CINCINNATI
U.S. ENVIRONMENTAL PROTECTION AGENCY
CINCINNATI, OH 45268

Form Approved O.M.B. 2080-0016
4-30-89

Date Request Received _____
Laboratory Code Number _____
Request Number _____
Verified _____

The USEPA Repository for Toxic and Hazardous Materials
Request for Materials

Please Print or Type

Name _____ Telephone _____
Company _____
Laboratory _____
Street _____
City _____ State _____ Zip Code _____
Approval of Laboratory Director _____
Check Activity for which samples are requested: _____ Ambient Monitoring _____ Superfund (CERCLA)
_____ Drinking Water _____ Wastewater _____ Toxics (TSCA) _____ Solid Wastes/Hazardous Wastes (RCRA)

Concentrations are 5000 µg of QAS-pure compound per mL of methanol solvent unless otherwise noted.

_____ *E263 Diphenylamine*
_____ *E270 Acrylamide (10,000 µg/mL)*
_____ *E271 Pyridine (10,000 µg/mL)*
_____ *E282 Diisodecyl phthalate*
_____ *E284 Acetone*
_____ *E285 Diethyl ether*
_____ *E286 1,2-Epoxybutane*****
_____ *E295 Phenacetin*
_____ *E298 N-Nitrosopyrrolidine*
_____ *E299 2-Fluoroacetamide*
_____ *E300 Pentachloroethane*
_____ *E305 4-Chloroaniline*
_____ *E311 Methyl ethyl ketone (10,000 µg/mL)*
_____ *E322 Methylene bis(o-chloroaniline)*
_____ *E324 o-Nitroaniline*
_____ *E325 m-Nitroaniline*
_____ *E329 Ethylenethiourea*
_____ *E330 2,4-Dichlorophenoxyacetic acid (2,4-D)*****
_____ *E334 N-Nitrosodiethylamine*
_____ *E335 1,1,1,2-Tetrachloroethane (QAR)*
_____ *E337 Malononitrile*
_____ *E338 Propionitrile*
_____ *E342 p-Nitroaniline*
_____ *E349 4-Methyl-2-pentanone*
_____ *E360 Carbon tetrachloride*
_____ *E363 Carbon disulfide*
_____ *E364 Hexachloropropylene (1000 µg/mL)*
_____ *E366 Safrole*
_____ *E368 1,2,3-Trichloropropane*
_____ *E369 Saccharin (2000 µg/mL)*
_____ *E375 3-Chloropropionitrile (1000 µg/mL)*
_____ *E406 Bromobenzene*
_____ *E411 Acetophenone*
_____ *E439 Methylmethacrylate (1000 µg/mL)*
_____ *E455 Dinoseb*****
_____ *E458 1-Nitrosopiperidine*
_____ *E470 PCN Halowax 1099 (QAT)*
_____ *E471 PCN Halowax 1001 (QAT)*
_____ *E472 PCN Halowax 1000 (QAT)*
_____ *E473 Acetonitrile****
_____ *E475 Allyl alcohol (1000 µg/mL)*
_____ *E480 para-Dioxane (10,000 µg/mL)*
_____ *E536 Vinyl chloride ****
_____ *E541 Benzoic acid*****
_____ *E542 Aniline*
_____ *E543 Propargyl alcohol (1000 µg/mL)****
_____ *E548 N,N-Dimethylformamide*
_____ *E552 2,4,5-TP (Silvex) (QAR)*****
_____ *E560 Ethylparathion (1000 µg/mL)*****
_____ *E565 2-Naphthylamine (1000 µg/mL)*
_____ *E567 7,12-Dimethylbenz(a)anthracene (1000 µg/mL) (QAR)*
_____ *E572 Methylparathion (1000 µg/mL)*****
_____ *E573 Kepone (1000 µg/mL) (QAR)*
_____ *E662 3-Nitrophenol*
_____ *E669 1-Methyl ethyl benzene (Cumene)*
_____ *E686 Methacrylonitrile (1000 µg/mL)*
_____ *E687 Ethylmethacrylate (1000 µg/mL)*
_____ *E688 2-Picoline*
_____ *E700 Resorcinol*
_____ *E713 Picloram (1000 µg/mL)*****
_____ *E715 Carbofuran*
_____ *E952 p,p'-Methoxychlor*
_____ *E954 Aldicarb (1000 µg/mL)*****
_____ *E993 1,2-Dibromo-3-chloropropane*
_____ *E995 Aldicarb sulfone (1000 µg/mL)*****
_____ *E996 Aldicarb sulfoxide (1000 µg/mL)*****
_____ *E1089 Alachlor (1000 µg/mL)*
_____ *E1090 Atrazine (1000 µg/mL)*
_____ *E1097 Dibromomethane*
_____ *E1103 1,3,5-Trimethylbenzene (Mesitylene)*
_____ *E1104 sec-Butylbenzene*

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_____ E1105 n-Butylbenzene
_____ E1106 tert-Butylbenzene
_____ E1107 1,2,4-Trimethylbenzene (QAR)
_____ E1108 4-Isopropyltoluene (p-Cymene) (QAR)
_____ E1109 1,3-Dichloropropane
_____ E1112 n-Propylbenzene (1-Phenylpropane)
_____ E1166 1,1-Dichloro-1-propylene (QAR)
_____ E1167 2,2-Dichloropropane

Surrogates and Internal Standard for USEPA GC/MS Methods 624 and 625

_____ E188 Phenanthrene - d_{10} (150 µg/mL)
_____ E189 Phenol - d_5 (100 g/mL)*
_____ E190 2,4-Dimethylphenol-3,5,6-d3 (100 µg/mL) (QAR)*
_____ E191 Pentachlorophenol $^{13}C_6$ (100 µg/mL)*
_____ E192 Dimethyl phthalate - d_6 (150 µg/mL)*
_____ E193 2-Fluorophenol (QAR) (100 µg/mL)*
_____ E194 2-Fluorobiphenyl (100 µg/mL)*
_____ E195 1-Fluoronaphthalene (100 µ g/mL)*
_____ E196 1,4-Dichlorobutane-d_8 (150 µg/mL)
_____ E197 2-Bromo-1-chloropropane-d_6 (150 µg/mL) (QAT)
_____ E198 Bromochloromethane-d_2 (150 µg/mL)
_____ E199 Benzo(g,h,i)perylene-$^{13}C_{12}$ (100 µg/mL)*
_____ E232 Fluorobenzene (150 µg/mL)
_____ E233 4-Bromofluorobenzene (150 µg/mL)
_____ E234 4,4-Dibromooctafluorobiphenyl (100 µg/mL)*
_____ E776 1,2-Dichlorobenzene-d_4 (150 µg/mL)

*In Acetone **In para-Dioxane ***In 2-Propanol ****In Acetonitrile + Methylene chloride + + In Isooctane

Date Requested:_____ Date Shipped:_____

EPA-360 (Cin) (Rev. 6/83, Pt. 4)

6 16 E

REQUEST FOR REFERENCE STANDARDS

MAIL TO: Robert E. Thompson, NSI
U.S. Environmental Protection Agency
Pesticides & Industrial Chemicals Repository (MD-8)
Research Triangle Park, NC 27709 USA

16 Jun 89
Date

elephone: Technical Assistance: (919) 541-3951
(FTS) 629-3951
Emergencies: (702) 798-2690
(FTS) 798-2690
Off-hour EMERGENCIES: (919) 836-0639

Date Request Received
Date of Shipment
Laboratory Code Number 30530
Request Number
Verified

THIS BLOCK FOR AGENCY USE ONLY

The following reference standards are required for our program:

Index Code Number	Compound (Index Name)
1080	Methyl Carbophenothion
4260	Malothion
4580	Methyl parathion
5245	Ethyl parathion
3340	Ethion
2080	Diazinon
2940	2,4-D acid (2,4-D) X2
2309	2,4-DP (Dichlorprop)
6840	2,4,5-T
6120	2,4,5-TP (Silvex) X2
2112	Dibutyl Chlorendate X2

Index Code Number	Compound (Index Name)

Please complete this form in full, PRINTING OR TYPING name and address. Use black ink if possible. Use back of sheet to complete list if necessa
No cover letter is needed.

Name and address of laboratory: USACE Waterways Experim
Station
3909 Halls Ferry Rd.
Vicksburg, MS 39180-
Attn: Richard Karn, EE-A

Richard A. Karn
Requestor's Name (printed)

IMPORTANT:

1. Please do not request more than two (2) samples of any one compound. Many high purity, analytical-grade compounds are scarce and exper to refine. Note that the supply of many reference standard compounds is quite limited and therefore samples of these materials will be sc what less than the former 100 mg size. All standards of pesticidal materials will be supplied in masses between 50 and 100 mg, dependin supply and demand. All standards of industrial chemicals will be in 200 mg amounts.
2. Please return *AT ONCE* the acknowledgment card(s) enclosed with your shipment. One card is included with each parcel. These cards provide sole evidence of shipment delivery. A tracer must be mailed after 30 days, so please help us reduce costly paperwork.
   Occasionally a bottle of liquid material may appear empty upon receipt. This occurs when high-viscosity compounds collect in the vessel ca when volatile compounds disperse. Before concluding you were sent an empty vessel, allow the vial to sit upright for 2 hours; then remove and examine.
3. Suggestions for other compounds to be added to the Repository inventory are always welcomed. Please use the space indicated on the revers this form.

G-16 F

SUGGESTIONS FOR FUTURE REFERENCE STANDARD ADDITIONS

Industrial Chemical Compounds (Systematic chemical names are preferred)		Pesticides and Pesticide Derivatives (Trade, generic, and/or common names are preferred)
Ethyl Carbophenothion		

4-8907 Supelpreme-HC Hazardous Substances Mix 1

4-8851 Purgeables A

4-8852 Purgeables B

4-8853 Purgeables C

A List of EPA Standards ordered from the repository at Research Trian Park is attached.

Chlorinated Biphenyl Standards were purchased from the National Research Council of Canada for congener analysis.

STANDARD REFERENCE MATERIALS AND CHECK SAMPLES FOR QUALITY CONTROL

The following SRMS from NIST are in the laboratory:

1642b Mercury in water

1643b Trace elements in water

1645 Trace elements in river sediment

1646 Trace elements in estuarine sediment

1566 Oyster tissue

1567 Bovine Liver

1573 Tomato leaves

Quality Control samples from Environmental Resource Associates are received on a quarterly basis for waste water, potable water, priority pollutants, and pesticides.

Additional quality control samples from EPA and NRC of Canada are ordered as needed to supplement samples listed above.

DATA EVALUATION

All data is checked by the analyst, the inorganic team leader or the organic team leader, and the Chief, ALG before it

is submitted to the customer. The following items are checked:

1. Completeness
2. Duplicate values for precision
3. Recovery of Spikes for accuracy
4. Method blanks for contamination
5. Surrogate recoveries for organic analysis
6. Data for QA check samples (EPA, ERA or NBS SRMs)
7. Reasonableness and trends

If data falls outside acceptable limits as described in the procedures, samples are rerun if sample is available. If data falls outside acceptable limits on the reruns and QA check sample data is good, then data may be reported with qualifying explanations. Acceptable data is usually defined by the procedures (i.e. SW-846, CLP, 600 methods, 500 methods).

CONTROL CHARTS

All inorganic parameters routinely analyzed are charted. A minimum of 20 points is required for limit calculations. Precision control charts are prepared from the duplicate analysis data and accuracy charts are prepared from matrix spikes. Future charts will also be prepared utilizing blank spike data. Charts are also prepared using SRMs and external QA samples. Control charts for organics are limited to the CLP spiked compounds and surrogates. PCB-1248 is the spiking compound for PCB analysis.

CORRECTIVE ACTIONS

During the performance of work under any laboratory task, out of control conditions may occur which call for corrective action.

Typically QC parameters given in the various methods are employed to determine the need for action. The type of action required shall be documented, although documentation may only require appropriate instrument and/or laboratory logbook entries, as opposed to a formal corrective action memo.

Corrective actions may also include procedural matters related to internal custody of samples, sample log-in, sample data processing and so forth. These are usually provided via inhouse memo from the Chief, ALG.

As necessary modifications to inhouse procedures, equipment, standards, policy and so forth may be needed to rectify recurring or otherwise significant laboratory problems. These are usually resolved via group meetings where a course of action is decided upon and a time for correction is defined.

When significant changes are made the QA manual will be updated to reflect the changes.

Use of a form to document corrections will be implemented October 1989.

GC/MS TUNING

GC/MS tuning is performed using Hewlett-Packard recommended procedures for the quadrapole systems and is included in the instrument manuals (copy attached).

DATA REDUCTION.

Data reduction is shown in the following flow chart. It is the responsibility of the analyst to conduct initial data reduction. The readings from the instruments or analytical system are calculated into

DATA REDUCTION
ANALYSIS FLOW CHART

LABORATORY DATA MANAGEMENT SYSTEM
(Bench Sheets)

↓

ANALYSIS OF SAMPLES

↓

ANALYST'S NOTEBOOK

↓

RAW DATA TO BENCH SHEETS

↓

TEAM LEADER

↓

LABORATORY DATA MANAGEMENT SYSTEM ⇄ QC CHECK

↓

CHIEF, ANALYTICAL LABORATORY GROUP

↓

LABORATORY DATA MANAGEMENT SYSTEM

↓

FINAL REPORT
(Initialed by C/ALG)

DFTPP TUNING

REFERENCES:

E-16	
D-98	regulations detailed
E-11	spectrum
D-102	2.6.1.2 - Demonstrates consistant spectra from MS to MS. Spells out requirements for qualitative verification that must be met.

NOTES:

A) DFTPP must be verified once per shift or every 12 hrs.

B) Note that all MS give different looking DFTPP spectra, which implies a wide range of PFTBA spectra.

```
 69 = 100 --|
219 =  55   |-- fairly normal
502 = 1.2 --|

 69 = 100 --|
219 =  20   |-- perfectly ok, often
502 = 0.2 --|   seen on benchtops
```

C) There are many different ways to tune DFTPP. The methods given below (some specific, some general) are ways that many users have found successful. In addition, there are many "markers" that different users look for during the tune that seem to indicate that the tune will be successful. Some of the more popular of these goals are:

1) Tune until 219 and 131 are approximately equal and range between 20 and 60% of 69.
2) After tuning, check spectrum scan and see if m/e 50 and m/e 502 are equal. The closer to equal, the better the chance that the tune will be successful.

Look for guidelines such as these on your MS to help guide you during tuning.

D) DO NOT TUNE UNLESS IT IS NECESSARY! It will cost you only 5 - 10 min to attempt to verify your last tune. Experience has shown that the more often you tune, the more often you have to recalibrate your ID file. This is because you are adjusting your mass response ratios when you tune.

PROCEDURE A: 5987/5988

MANUAL TUNE - PFTBA OPEN

1) REPEAT PROFILE - set plunger to max 219
2) Set GC temp to 225-275 (ie. elution temp of DFTPP) so GC flow = flow at DFTPP elution.
3) Run AUTOTUNE - set source to 250
4) MANUAL TUNE (source @ 250, GC ~ 250)
5) set A/D =1 (0.5 sec/scan or faster)
6) run REPEAT PROFILE about 1 min to warm up and stabilize the source and EM.
7) PARAMETER RAMP (make sure plunger is adjusted) Begin to max 219, starting with REP
   A) REP
   B) IF
   C) DO
   D) ENT LENS

   leave the X-RAY alone at this point.
8) in REPEAT PROFILE use the X-RAY to set 69:219:502 ratio. 502/219 should be >3% and <5%. (do not be confused and set 502/69 ratio).
9) use DO to achieve final ratios, if needed.
10) do PEAK WIDTH and MASS AXIS CALIBRATION
11) check spectrum to make sure calibrations did not shift peak ratios.
12) store tune as MTDFTP

NOTES:

A) If 502 has a precursor, you will probably lose m/e 441 in DFTPP spectra. This is more of a problem on bench-tops than big systems. Cleaning the ENT LENS will often eliminate this precursor.

PROCEDURE B: 5995

A) Set oven to 250 with XFR,OVN1,250 (ie. elution temp of DFTPP, so GC flow = flow at DFTPP elution)
B) Set source to 250 with XFR,SORS,250
C) Run AUTOTUNE
D) MANUAL TUNE - PFTBA OPEN
Note: If you have previously created a manual tune file, use NEW TUNE FILE to load it.

1) Edit temp zones
   Source 250
   Analyser 180
   Transfer 280
2) Edit scan parameters to set A/D =1 (0.5 sec/scan or faster)
3) run REPEAT PROFILE about 1 min to warm up and stabalize source and EM.
4) PARAMETER RAMP - begin to max 219
   A) REP leave alone
   B) IF most useful lens
   C) ENT LENS can be useful
   leave the X-RAY alone at this point.
   NOTE: on benchtops, if we set IF = 100, this is often all that is necessary. As a first attempt just adjust the IF and leave other lenses alone.
5) in REPEAT PROFILE, use X-RAY to set 69:219:502 ratio. 502/219 ratio should be >3% and <5% (do not be confused and set 502/69 ratio). Adjust ratio using X-RAY, being careful not to adjust too far and deteriorate peak shape.
6) do PEAK WIDTH and MASS AXIS CALIBRATION
7) SPECTRUM SCAN - check spectrum to make sure calibrations did not shift peak ratios. Remember, 502/219 should be >3% and <5%. Also, look at 131/219 ratio. They should be approximately equal.
8) Store tune as MTDFTP

NOTES:

A) If 502 has a precursor, you will probably lose m/e 441 in DFTPP spectra. This is more of a problem on bench-tops than big systems. Cleaning the ENT LENS will often eliminate this precursor.

appropriate concentration units for the results. Rounding occurs only after calculations are complete. The calculations are normally performed on the raw data sheets. The results are then transcribed on to the computer generated data files.

The next step in data reduction is data review by the team leader. Data is checked for validity and completeness. Questionable data is brought to the attention of the analyst and corrective action is taken if necessary.

Data is then forwarded to the sample management officer for entry into the Laboratory Data Management system.. Data entry is checked by the team leader, corrections are made, and the report is then sent to the quality assurance officer and the Chief, Analytical Lab Group for review. Any corrections are made and final report is generated that has been initialed by the Chief, ALG. All raw data is filed.

DATA OUTLIERS

Sample data that falls outside acceptable limits are reanalyzed if possible. If sample is not available to rerun the sample, data are reported with qualifying statements and with external QA data to support the results, assuring that the analytical system was in control. Method blanks are analyzed with each set of samples and reported with the sample data. Organic method blanks are spiked with surrogates.

AGENCY APPROVALS

As the primary research and development facility for Corps environmental research, the ALG has not solicited formal approval from other agencies or states, although the ALG has conducted numerous research projects for the Environmental Protection Agency, the Navy,

the Coast Guard, the Air Force, and other Army branches. The ALG is a validated Quality Assurance Laboratory for the Corps Superfund and DERP projects.

INTERNAL AUDITS

Several methods of internal audits are used including worksheet review, on-site analyst review, intra- and interlaboratory sample examination. Presently the ALG participates in the Corps of Engineers Interlaboratory Testing Program and subscribes quarterly to the Environmental Research Associates Quality Control Sample program (beginning October 1989, subscription will be bimonthly). In addition the QA officer submits EPA, NRC or NIST quality assurance samples to the laboratory for internal review of the measurement process. System audits are conducted semi-annually to evaluate sample handling, sample analysis,record control, proficiency testing, personal practices, training, workload, and manpower needs.

DOCUMENT FLOW

The sample management officer is also the in charge of document control. She maintains the master logbook, the computer logbook, the computer-generated bench sheets, the completed computer generated bench sheets and the final computer generated reports. Each analyst maintains the raw data files from his/her instruments. The document flow is outlined in the Data Reduction Analysis Flow Chart. Final Data reports are approved by the team leader, the quality assurance officer and the Chief, ALG.

QA REPORTS

QA reports are tailored to customer request. QA reports for

DERP projects where WES ALG serves as the QA lab utilize the following outline.

A. Cover letter

B. Report Narrative

1. Summary

2. QC data discussion

3. QA/QC data comparison

4. Other Problems

5. Corrective Actions

C. Data comparison tables