NUS CORPORATION

Gannett Fleming
ENVIRONMENTAL ENGINEERS, INC.

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FINAL
PROJECT OPERATIONS PLAN

FOCUSED REMEDIAL INVESTIGATION
FEASIBILITY STUDY

ARMY CREEK LANDFILL SITE
NEW CASTLE COUNTY, DELAWARE

EPA WORK ASSIGNMENT
NUMBER 37-10-3L34
CONTRACT NUMBER 68-W8-0037

NUS PROJECT NUMBER 1017

JULY 1989

Revision Number: _____ Copy Number: 6

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ARMY CREEK LANDFILL SITE
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REPORT TITLE: PROJECT OPERATIONS PLAN
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DATE: JULY 7, 1989

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1.0 PROJECT DESCRIPTION

This Project Operations Plan (POP) provides guidance for the remedial investigation (RI) of contaminated stream sediments, surface water, and groundwater adjacent to the Army Creek Landfill in New Castle County, Delaware.

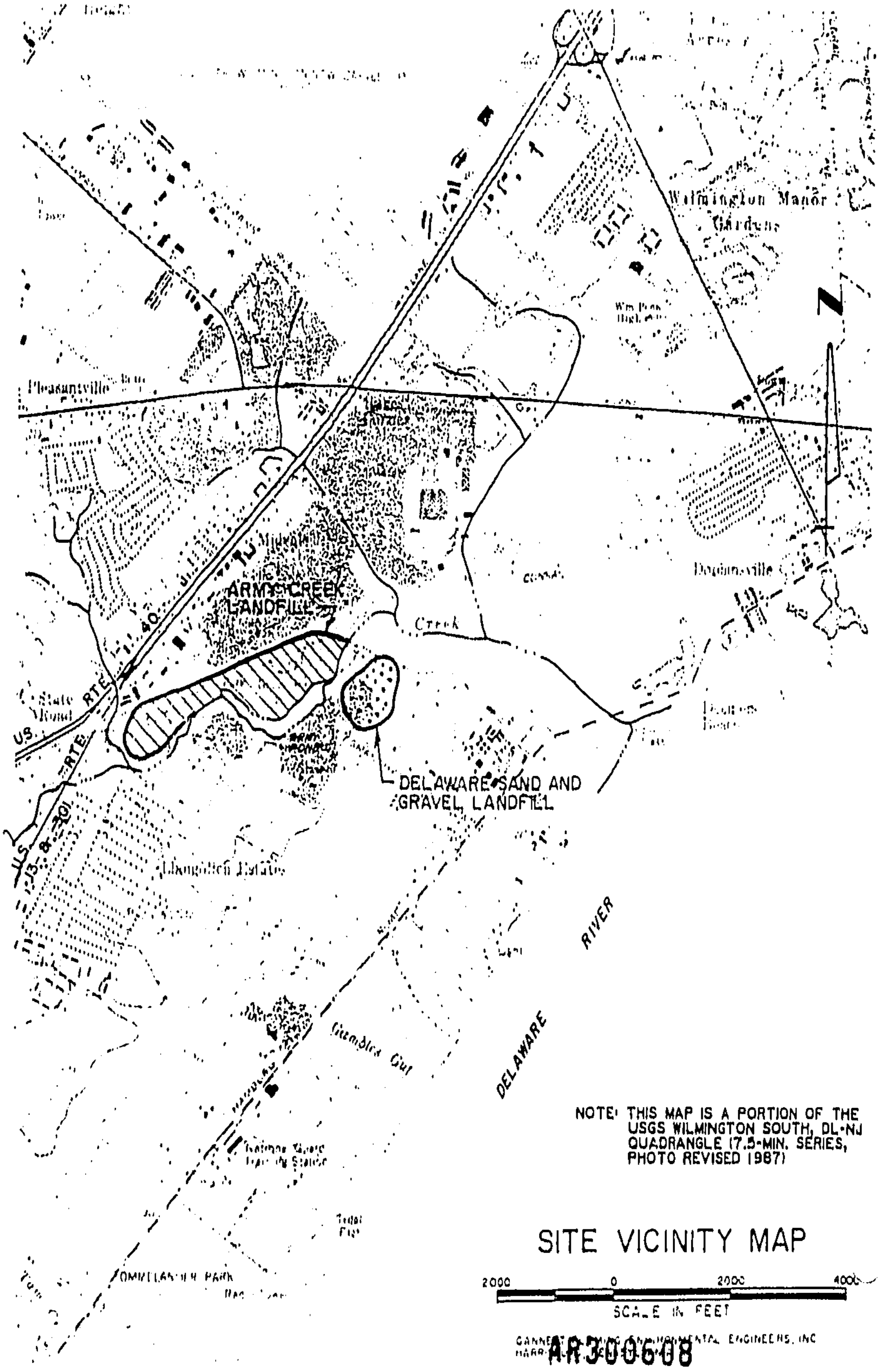
This section provides a brief review of the site description and site history as well as the scope of work for the RI.

1.1 SITE LOCATION AND DESCRIPTION

The Army Creek Landfill, formerly known as the Llangollen Landfill, is located approximately 7 miles southwest of the City of Wilmington, Delaware (Figure 1-1). The landfill is bordered to the north and west by Conrail tracks, and on the south and east by Army Creek. The highways adjacent to the landfill are U.S. Routes 13 and 301 to the west, and Delaware Route 9 to the east. Map coordinates for the site are approximately 39 degrees, 39 minutes north latitude, and 75 degrees, 37 minutes west longitude. The site was placed on the Superfund National Priorities List (NPL) due primarily to contamination of local groundwater which is withdrawn by the Artesian Water Company from water supply wells near Llangollen Estates, a residential development located southwest of the site (Figure 1-2). The former Amoco Chemical Plant, closed in 1980 due to fire, is located 1/4 mile east of the site. Delaware Sand and Gravel (DS&G), another landfill which also has been placed on the Superfund NPL, is adjacent to Army Creek Landfill and separated from it only by Army Creek, a tributary of the Delaware River.

1.2 SITE HISTORY

The Army Creek Landfill is a 60-acre site that was operated by New Castle County from 1960 through 1968 for disposal of municipal and industrial waste. The site is adjacent to DS&G, an industrial waste disposal site closed by the Delaware Department of Natural Resources Environmental Conservation (DNREC) in 1976. Approximately 1.9 million cubic yards of refuse were landfilled at Army Creek.



NOTE: THIS MAP IS A PORTION OF THE USGS WILMINGTON SOUTH, DL-NJ QUADRANGLE (7.5-MIN. SERIES, PHOTO REVISED 1987)

SITE VICINITY MAP

GANNE[illegible] ENTAL ENGINEERS, INC
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FIGURE 1-1

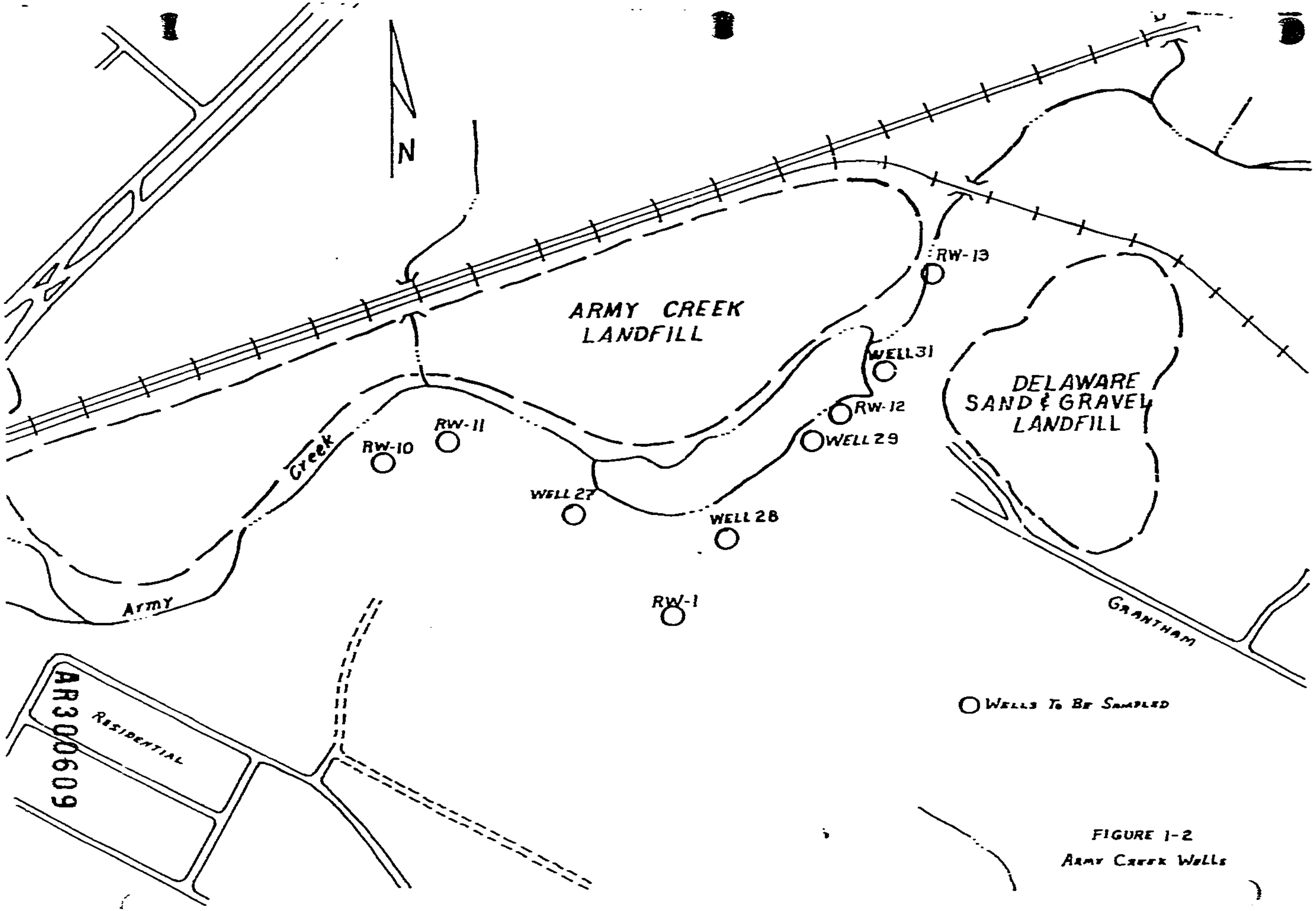


FIGURE 1-2
ARMY CREEK WELLS

After groundwater contamination was discovered in 1972, New Castle County installed a series of groundwater recovery wells downgradient of the landfill to prevent the contaminant plume from reaching wells belonging to the Artesian Water Company. Approximately 5,000 residential customers are serviced in that area by Artesian. According to the U.S. Environmental Protection Agency (EPA), it appears that pumping of the recovery wells has created a groundwater divide between the Army Creek Landfill and the Artesian Water Company's Llangollen Wellfield. The recovery wells appear to be effectively capturing, and thereby preventing further migration of the plume of organic and inorganic contaminants from both Army Creek and DS&G landfills that have been detected in the recovery wells and monitoring wells.

Army Creek was proposed for inclusion on the NPL in October of 1981, and was included in September of 1983 (original NPL). In 1984, EPA entered into a Consent Agreement and Order with New Castle County to perform a Feasibility Study (FS) which was completed in July 1986.

The Army Creek Record of Decision (ROD) was issued on September 30, 1986. The ROD required installation of a RCRA-type landfill cap and continued operation of the recovery well system at a currently estimated cost of $25 million. The September 30, 1986 ROD deferred the decision on groundwater treatment until after the National Pollutant Discharge Elimination System (NPDES) permit was issued, and until the DS&G RI/FS was completed. The Army Creek FS addressed groundwater treatment focusing on iron and solids removal. The DS&G FS addressed isolated (near drum disposal area) groundwater contamination within the DS&G site and not the total site area. Neither study addressed all of the specific contaminants, both inorganic and organic, found in groundwater leaving the sites. A second ROD is needed to select a groundwater treatment remedy for the discharge of recovery well water to Army Creek, and to determine the appropriate remedial measure to clean up contaminated sediments in Army Pond, if necessary.

According to the field investigation data developed in August 1988 under EPA Work Assignment No. 0-123, Field Investigation, Army Creek Site, the groundwater, surface water, and soils contain various concentrations of metals and organic compounds. Surface water and sediment in Army Creek is contaminated with iron, zinc, and chromium, and the water quality standard for these are exceeded. To comply with a Consent Order with the State, signed in 1987, New Castle County is designing a groundwater discharge treatment plant for iron removal that is expected to be completed in July 1990.

The groundwater has been sampled and analyzed in the past and has shown varying degrees of contamination with time. A representation of those data is presented in Tables 1-1 through 1-3.

1.3 SCOPE OF WORK

The field investigation will be conducted to collect data by sampling those recovery wells that are actively pumping during the field activity period (see Figure 1-2 for approximate locations). The schedule found in Table 1-4 is based on the assumption that no unforeseen site restrictions or data quality problems will arise, and that reviews of written submittals by EPA Region III and any other agencies will be accomplished in two weeks.

Table 1-1 Volatile Organic Compound Concentrations

Contaminant Concentrations (ug/l)

Recovery Well	Sample Date	Benzene	1, 1 Di chloro ethene	Chloro benzene	Chlorodi bromo methane	1, 1 Di chloro ethane	1,2 Di chloro ethane	1,2 Di chloro propane	Ethyl benzene	Methylene Chloride	Toluene	Total Xylenes
RW1	8/77	4		0			2		0		0	0
	3/83	0.72			0			40.6	205	32	0	
	10/84	12			0			0	0	0	0	
	3/85	2.5	2.5	2.5		2.5	2.5		2.5		2.5	
	Average	4.8	2.5	1.3	0.0	2.5	2.3	20.3	51.9	16.0	0.6	0.0
RW9	3/83	0			0			0	0	0	0	
	84	2.1		0					0			
	Average	1.05		0	0			0	0	0	0	
RW10	3/83	0			0			0	0	0	0	
	12/84	0			0			0	0	0	5	
	3/85	2.5		2.5		2.5	51		2.5		2.5	
	4/86	2.5		2.5		2.5	33		2.5	2.5	2.5	2
	4/86	2.5		2.5		2.5	30		2.5	1	2.5	2.5
	Average	1.5		2.5	0	2.5	38	0	1.5	0.875	2.5	2.25
RW12	3/83	10.3			0			25.1	158	21.2	0	
	12/84	0			0			0	0	0	0	
	3/85	2.5	2.5	0	0	0	0		2.5			
	4/86	7		5		2.5	2.5		2.5	2.5	5	2.5
	Average	4.95	2.5	2.5	0	1.25	1.25	12.6	40.8	7.9	1.7	2.5
RW13	3/83	0			0			26.8	186	21.3	0	
	1/84	0			0			0	0	0	0	
	84	37					1.4		4.4		110	
	3/85	30	2.5	2.5		2.5	2.5		2.5		83	
	4/86	2.5		2.5		1	5		1	2.5	2.5	30
	Average	13.9	2.5	2.5	0	1.75	3.0	13.4	38.8	7.9	39.1	30.0
RW14	3/83	0		0	0			0	142	0	0	
	12/84	0		0	0			0	0	0	0	
	3/85	2.5	2.5	2.5		2.5	2.5		2.5		2.5	
	3/85	2.5		2.5		2.5	2.5		2.5		2.5	2.5
	4/86	2		5		2.5	2.5		2.5	2.5	2.5	2.5
	Average	1.4	2.5	2	0	2.5	2.5	0	29.9	0.83	1.5	2.5
27	8/77	13		0			6		11		0	0
	3/83	0			0			0	0	0	0	
	1/84	0			0			0	0	0	0	
	3/85	2.5	5	2.5		2.5	2.5		2.5		2.5	
	4/86	1		2		2	2.5		2.5	2.5	2.5	2.5
	Average	3.3	5	1.5	0	2.25	3.7	0	3.2	0.83	1	1.25
28	8/77	6		0.3			180		0		0	0
	3/83	0			0			0	0	0	0	
	10/84	40			0			0	0	0	0	
	3/85	2.5	2.5	2.5		2.5	2.5		2.5		2.5	
	4/86	3		5		2.5	6		2.5	2.5	2.5	2.5
	Average	10.3	2.5	2.6	0	2.5	62.8	0	1	0.83	1	1.25
29	8/77	2.7		1			0		0		0	0
	11/81			5						0		
	10/83	0			0			0	0	0	0	
	10/84	45			0			0	0	0	0	
	84	31		18		2.5	3.8		7.8			
	3/85	2.5	2.5	24		2.5	2.5		59		16	
	4/86	9		22		2	7		4	2.5	2.5	2.5
	Average	15.0	2.5	14	0	2.3	3.3	0	11.8	0.625	3.7	1.25
31	8/77	0.8		0.2			0		0		0	0
	11/81			5						0		
	3/83	41.3			19.2			0	228	16.3	63	
	10/84	150			0			0	0	0	0	
	3/85	130	2.5	2.5		2.5	2.5		24		360	
	4/86	6		2.5		2.5	2.5		10	2.5	8	49
	4/86	14		1		1	1		8	10	16	43
	Average	57.0	2.5	2.2	9.6	2	1.5	0	45	5.8	74.5	30.7
AVERAGE FOR GROUNDWATER		11.33	2.81	3.11	0.96	2.18	13.14	4.63	22.38	4.16	12.56	7.96
	MCLG	0	7	60P			0	6P	680P		2000P	440P
	MCL	5	7	100P	100*		5	5P	700P		2000P	10000P
TOXICITY CRITERIA		5,300	238,000	19,500			20,000	8,100	32,000	193,000	550,000	

MCLG = MAXIMUM CONTAMINANT LEVEL GOAL

P = PROPOSED

* = THM TRIHALOMETHANE

Source: Technical Direction Memorandum Review of Army Creek & Delaware Sand and Gravel RI/FS Study and ROD Documents, EPA Work Assignment No. 3?-07-3134 GFEE/NUS, February 1989.

Table 1-2 Base Neutral and Acid Extractable Compound Concentrations

		Contaminant Concentrations (ug/l)												
Recovery Well	Sample Date	Anthracene	Benzo(k) fluoranthene	Bis(2-chloro ethoxyl) methane	Bis(2-ethylhexl) phthalate	4-Bromo phenol ether	Butyl benzl phthalate	Di-n-butyl phthalate	P-chloro-m-cresol	2-Chloro naphthalene	1,2-Di chloro benzene	1,3-Di chloro benzene	1,4-Di chloro benzene	Bis(2-chloroethyl ether)
RW1	3/83	0.7	0	0	37.3	0	0	8.4	0	0	0	0	0	0
	10/84	0	0	14	5	0	5		0	5	5	5	5	0
	Average	0.35	0	7	21.15	0	2.5	8.4	0	2.5	2.5	2.5	2.5	0
RW9	3/83	0	0	0	8.2	0	0	686	0	0	0	0	0	0
	10/84													
	Average	0	0	0	8.2	0	0	686	0	0	0	0	0	0
RW10	3/83	0	0	0	15.5	0	0	14.2	0	0	0	0	0	0
	12/84	0	0	5	5	0	5		0	0	5	5	5	0
	4/86				2			5			5	5	5	5
	4/86				5			5			5	5	5	5
	Average	0	0	2.5	6.88	0	2.5	8.07	0	0	3.75	3.75	3.75	2.5
RW12	3/83	0	0	0	15.5	0	0	6.9	0	0	0	0	0	0
	12/84	0	0	0	40	5	0		0	0	5	5	5	0
	4/86				5			5			5		3	3
	Average	0	0	0	20.17	2.5	0	5.95	0	0	3.33	2.5	2.67	1.00
RW13	3/83	0	0	0	39.3	0	0	6.8	0	0	0	0	0	0
	1/85	0	0	5	0	0	0		0	0	0	5	5	0
	4/86				2			5			5		5	4
	Average	0	0	2.5	13.77	0	0	5.90	0	0	1.67	2.5	3.33	1.33
RW14	3/83	0	92.8	0	36.6	0	0	13.2	0	0.2	0	0	0	0
	12/84	0	0	5	5	0	5		0	5	5	5	5	0
	4/86			0	2	0	0	5	0	0	5	0	5	5
	Average	0	46.4	1.67	14.53	0	1.67	9.10	0	1.73	3.33	1.67	3.33	1.67
27	3/83	0	0	0	23.6	0	0	10.6	0	0	0	0	0	0
	10/84	0	0	12	5	0	5		0	5	0	5	5	0
	4/86				2			5			5		5	4
	Average	0	0	6	10.20	0	2.5	7.8	0	2.5	1.67	2.5	3.33	1.33
28	10/83	0	0	10	5	0	0	64	0	0	0	0	0	0
	10/84	0	0	0	10	10	5		0	0	13	5	5	0
	4/86				5			5			5		5	
	Average	0	0	5	6.67	5	2.5	34.50	0	0	6	2.5	3.33	0.00
29	11/81				5		0	0					5	0
	10/83	0	0	0	0	0	0	83	0	0	0	0	0	0
	10/84	0	0	5	5	0	5		0	0	5	35	15	
	4/86				5			5			5		9	32
	Average	0	0	2.5	3.75	0	1.67	29.33	0	0	3.33	17.5	7.25	10.67
31	11/81				5		5	5				0		
	3/83	0	0	0	38.8	0	0	13.4	3.9	0	0	0	0	0
	10/84	0	0	5	0	0	5		0	5	5	5	5	0
	4/86				3			2			5		5	3
	4/86				10			1			1		1	20
	Average	0	0	2.5	11.36	0	3.33	5.35	1.95	2.5	2.75	1.67	2.75	5.75
AVERAGE FOR GROUNDWATER		0.035	4.64	2.97	11.67	0.75	2	80.04	0.20	0.92	2.83	3.71	3.23	2.43
	MCLG										620P		75	
	MCL										600P		75	
WATER INGESTION					4,200			770			300P	400	94	
TOXICITY CRITERIA					11,100	122	220	940	30		2000P	1,510	763	238,000

Source: Technical Direction Memorandum Review of Army Creek & Delaware Sand and Gravel RI/FS Study and ROD Documents, EPA Work Assignment No. 37-07-3L34 GFEE/NUS, February 1989.

Table 1-2 Base Neutral and Acid Extractable Compound Concentrations (con't)

Recovery Well	Sample Date	2,3-Di nitro toluene	2,6-Di nitro toluene	4,6-Di nitro-o-cresol	Diethyl phthalate	Fluor-anthene	Nitro-benzene	4-Nitro-phenol	Penta-chloro-phenol	Phen-anthene	Phenol	2,4,6-Trichloro Phenol
		Contaminant Concentrations (ug/l)										
RW1	3/83	0	0	0	20.9	0	0	0	0	0	0	0
	10/84	0	0	0	5	0	5	0	0	0	0	0
	Average	0	0	0	12.95	0	2.5	0	0	0	0	0
RW9	3/83	0	0	0	0	0	0	0	0	0	0	0
	10/84										16	
	Average	0	0	0	0	0	0	0	0	0	8	0
RW10	3/83	0	0	0	67.5	0	0	0	0	0	0	0
	12/84	44	22	0	0	0	0	0	0	0	0	0
	4/86				5						5	
	4/86				5						5	
	Average	22	11	0	19.4	0	0	0	0	0	2.5	0
RW12	3/83	0	0	0	23.1	0	0	0	0	0	0	3
	12/84	0	28	0	5	0	34	0	0	0	0	0
	4/86				5						5	
	Average	0	14	0	11.03	0	17	0	0	0	1.67	1.5
RW13	3/83	0	0	0	17.8	0	0	0	0	0	0	0
	1/85	0	0	0	10	0	0	0	0	0	0	0
	4/86				5						5	
	Average	0	0	0	10.9	0	0	0	0	0	1.67	0
RW14	3/83	0	0	0	45.8	2.8	0	0	0	0.7	0	0
	12/84	38	32	0	5	0	0	0	0	0	0	0
	4/86				5						5	
	Average	19.0	16.0	0.0	18.6	1.4	0.0	0.0	0.0	0.4	1.7	0.0
27	3/83	0	0	0	38.2	0	0	5.6	4.3	0	0	0
	10/84	0	0	0	0	0	.5	0	0	0	0	0
	4/86				5						5	
	Average	0	0	0	14.4	0	3	2.8	2.2	0	1.7	0
28	10/83	0	0	0	0	0	0	0	0	0	0	0
	10/84	0	0	5	0	0	11	0	0	0	0	0
	4/86				5						5	
	Average	0	0	2.5	1.67	0	5.5	0	0	0	1.67	0
29	11/81										5	
	10/83	0	0	0	0	0	0	0	0	0	0	0
	10/84	0	0	0	5	0	11	0	0	0	12.5	0
	4/86				5						5	
	Average	0	0	0	3.33	0	5.5	0	0	0	5.6	0
31	11/81										0	
	3/83	0	0	0	27.2	0	0	33.2	17.6	0	0	0
	10/84	116	116	0	5	0	11	0	0	0	0	0
	4/86				5						5	
	4/86				1							
	Average	58	58	0	9.55	0	5.5	16.6	8.8	0	1.25	0
AVERAGE FOR GROUNDWATER		9.9	9.9	0	10.18	0.14	3.85	1.94	1.10	0.035	2.57	0.15
	MCLG								220P			
	MCL								200P			
WATER INGESTION				13.4	350,000	42	35		21		3,500	17,500
TOXICITY CRITERIA				230	52,100	3,980	27,000	8,280	3	600	2,560	970

Source: Technical Direction Memorandum [illegible]view of Army Creek & Delaware Sand and Gravel RI/FS Study and ROD Docu[illegible] EPA [illegible]

Table 1-3 Metals and Alkalinity Detected in Recovery Wells

Recovery Well	Sample Date	Cr (ug/L)	Cu (ug/L)	Fe (ug/L)	Pb (ug/L)	Mn (ug/L)	Hg (ug/L)	Ni (ug/L)	Se (ug/L)	Zn (ug/L)	Ca (ug/L)	Mg (ug/L)	Alkalinit (mg/L as CaCO3)
RW1	1983	2.5	8.9		5.3	950	1.80	310.0	9.1	20.0			
	1985			1,770		405							
	1986												40.5
	Average	2.5	8.9	1,770	5.3	678	1.80	310.0	9.1	20.0			40.5
RW9	1983	37.3	9.5		3.4	1,300		16.0	100.0	5.0			
	1984	50.0	25.0		1.0		0.10	25.0		25.0			
	Average	43.7	17.3		2.2	1,300	0.10	20.5	100.0	15.0			
RW10	1983	12.3	10.5		5.5	480	0.70	60.0	7.9	30.0			
	1985			295		345							
	1986	8.3	4.4	416	2.5	270	0.05	9.6	2.5	9.5	8,220	4,350	
	1986	9.5	5.6	490	2.5	270	1.00	11.0	2.5	9.4	8,360	4,690	24.0
	Average	10.0	6.8	400	3.5	341	0.58	26.9	4.3	16.3	8,290	4,520	24.0
RW12	1983	18.5	14.2		1.7	1,000	2.80	90.0	1.0	10.0			
	1985			130		105							
	1986	9.0	4.7	9,850	16.0	698	0.10	6.0	2.5	16.0	11,300	6,240	110.0
	Average	13.8	9.5	4,990	8.9	601	1.45	48.0	1.8	13.0	11,300	6,240	110.0
RW13	1983	2.5	14.4		3.3	700	2.30	250.0	1.0	10.0			
	1984	50.0	25.0		10.0		0.10	25.0		25.0			
	1985			4,150		370							
	1986	2.0	4.5	3,600	2.5	302	0.05	3.0	2.5	14.0	7,640	2,950	21.0
	Average	18.2	14.6	3,875	5.3	457	0.82	92.7	1.8	16.3	7,640	2,950	21.0
RW14	1983	2.5	9.0		3.4	790	3.00	50.0	1.0	20.0			
	1985			6,400		690							
	1985			5,960		640							
	1986	8.0	5.5		2.5	431	0.05	6.0	2.5	30.0	11,700	7,390	104.5
	Average	5.3	7.3	6,180	3.0	638	1.53	28.0	1.8	25.0	11,700	7,390	104.5
27	1983	2.5	19.7		5.1	670	4.00	190.0	26.7	20.0			
	1985			4,880		390							
	1986	2.5	3.5	11,200	2.5	618	0.05	3.0	2.5	10.0	8,440	6,600	102.0
	Average	2.5	11.6	8,040	3.8	559	2.03	96.5	14.6	15.0	8,440	6,600	102.0
28	1983	73.3	34.4		3.0	1,590		2.9	100.0	20.0			
	1985			22,620		1,210							
	1986	7.3	5.4	11,670	2.5	1,120	0.05	6.2	2.5	7.3	11,700	7,200	116.5
	Average	40.3	19.9	17,145	2.8	1,307	0.05	4.6	51.3	13.7	11,700	7,200	116.5
29	1983	84.7	8.0		5.0	1,560		2.3	100.0	10.0			
	1984	50.0	25.0		1.0		1.00	25.0	1.0	25.0			
	1985			32,040		2,280							
	1986	6.0	4.9	23,000	2.5	2,550	0.05	3.0	2.5	7.3	17,900	12,400	241.0
	Average	46.9	12.6	27,520	2.8	2,130	0.53	10.1	34.5	14.1	17,900	12,400	241.0
31	1983	2.5	24.6		13.1	1,210	1.10	240.0	1.0	10.0			
	1985			12,880		1,190							
	1986	1.0	2.5	11,000	5.0	910	0.10	4.0	1.0	25.0		4,340	
	1986	8.3	3.3	10,500	2.5	833	0.05	3.0	2.5	9.0	8,610	4,350	58.8
	Average	3.9	10.1	11,460	6.9	1,036	0.42	82.3	1.5	14.7	8,610	4,345	58.8
AVERAGE FOR GROUNDWATER		18.7	11.9		4.4	905	0.93	72.0	22.1	16.3	10,698	6,456	81.8
	RMCL	120	1,300		20		3		45				
	MCL	50			50		2		10				
	TOXICITY CRITERIA	11	6.5		1.3		0.012	56	35	47			
	DE EFFLUENT LIMIT	150	500	2,000	150		5	1000	20	1000			

Source: Technical Direction Memorandum Review of Army Creek & Delaware Sand and Gravel RI/FS Study and ROD Documents, EPA Work Assignment No. 37-07-31.34

Table 1-4

Key Milestones

Milestone	Elapsed Time (weeks)	Date
Work Assignment Receipt	0	4/26/89
SAS Request Completed/Submitted	1	5/05/89
Draft Work Plan/Project Operations Plan	6	6/09/89
Field Water Sampling	10	7/07/89
Final Work Plan/Project Operations Plan	10	7/07/89
Submit Draft RI Report	20	9/08/89
Submit Draft FS Report	23	9/29/89
Submit Final RI/FS Report	28	10/31/89

2

2.0 PROJECT ORGANIZATION AND MANAGEMENT

2.1 SITE CONTROL

This section provides a brief discussion of site access, site security control, and field command for the one- to two-day field task to be performed during the groundwater investigation.

2.1.1 Site Access

The Army Creek Landfill is accessible by public roads. The site entrance is adjacent to Routes 13 and 301 along the west, and Delaware Route 9 along the east. The site owner, New Castle County, Delaware, will be contacted in writing by the Regional Project Manager (RPM) in order to secure written permission to access the site. Anticipated dates for groundwater sampling will be provided to the County.

2.1.2 Site Security/Control

The Army Creek Landfill has very limited security along its perimeters. At the entrance, there exists a chain link fence and gate, and mounds of soil. The gate can be easily bypassed to walk on to the site. The field effort and equipment will be controlled from the Gannett Fleming Environmental Engineers, Inc., (GFEE) vehicles parked in front of the entrance gate.

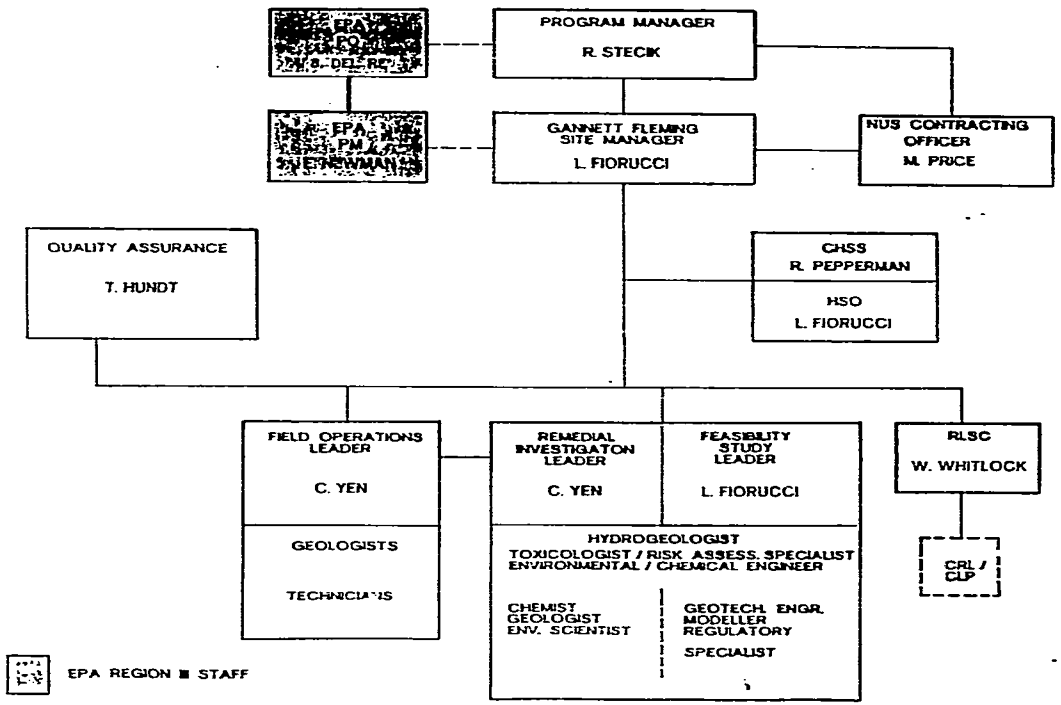
2.1.3 Field Office/Command

For this limited one- to two-day field sampling task, the GFEE vehicles will serve as Field Office/Command and equipment stores.

2.2 PROJECT ORGANIZATION

The overall project organization and responsibilities of key management personnel are discussed in Section 5.0 of the RI/FS Work Plan. The organizational chart presented in Section 5.0 of the Work Plan is reproduced here as Figure 2-1 for easy reference.

FIGURE 2-1
PROJECT ORGANIZATION



Field work will be performed by a single team under the direction of the Field Operations Leader (FOL). The FOL reports directly to the Project/Site Manager. The FOL will be responsible for coordinating and overseeing all field activities and will interface with the Health and Safety Site Officer (HSSO) in planning and performing the RI field tasks. In accordance with the Health and Safety Plan (HASP--Appendix C), the HSSO or his designee will interact with the field members during the performance of their tasks.

2.3 RESPONSIBILITIES OF KEY PERSONNEL

Key personnel for field operations are identified in Figure 2-1. Their organizations, addresses, and phone number(s) are listed in Table 2-1; and their specific responsibilities are discussed below.

- FOL--The FOL is responsible for all day-to-day aspects of the Army Creek Landfill Site RI field work. The responsibilities of the FOL include:

    - Ensuring that all field team members are familiar with the POP and HASP.
    - Ensuring that all field team members have completed health and safety training.
    - Coordinating the activities of all field teams.
    - Reporting to the Project Manager on a regular basis regarding the status of all field work and any problems encountered.
    - Completing Task Modification Requests (TMRs) as necessary, for approval by the Project Manager.

- Field Team Leader (FTL)--The FTL reports directly to the FOL and has the following responsibilities:

    - Providing field team members with daily assignments.
    - Ensuring that field team members comply with the procedures outlined in the POP.

Table 2-1

Army Creek Landfill Project Key Personnel

Organization	Name	Address	Phone Number(s)
EPA	S. Del Re E. Newman	Region III 841 Chestnut Street Philadelphia, PA 19107	(215) 597-1265
NUS	R. Stecik	One Devon Square Suite 222 724 W. Lancaster Avenue Wayne, PA 19087	(215) 971-0901
GFEE	L. Fiorucci T. Hundt C. Yen W. Whitlock	Suite 417 W. Quad Village of Cross Keys Baltimore, MD 21210	(301) 433-8832
	R. Pepperman	207 Senate Avenue Camp Hill, PA 17011	(717) 763-7211

  - Coordinating with the FOL to ensure sample shipping schedules are met.
  - Reporting on a daily basis to the FOL on the progress of the team.

- HSSO--The HSSO reports to the ARCS III Health and Safety Officer (HSO) and indirectly to the FOL and Project Manager. Details of the HSSO's responsibilities are presented in the HASP and include:

  - Controlling specific health and safety-related field operations such as personnel decontamination, monitoring of worker heat or cold stress, distribution of safety equipment, etc.
  - Ensuring that field team personnel comply with all procedures established by the HASP.
  - Identifying assistant HSSO designees.
  - Terminating work if an imminent safety hazard, emergency situation, or other potentially dangerous situation is encountered.

- Regional Laboratory Sample Coordinator (RLSC)--The RLSC is responsible for the following tasks:

  - Scheduling laboratory service through the EPA Sample Management Officer (SMO) and other vendors.
  - Tracking samples and coordinating with the EPA Central Regional Laboratory (CRL).
  - Scheduling data validation.
  - Identifying laboratory analytical methods and laboratory quality control (QC).

- Quality Assurance Representative (QAR)--The QAR is responsible for project quality assurance (QA) audits (see 10.1).

2.4 SCHEDULE

The projected schedule for the Army Creek Landfill Site RI/FS activities is presented in Section 5.3 of the RI/FS Work Plan (GFEE, 1989). Anticipated target dates for field tasks are as follows:

Activity	Anticipated Start	Anticipated Finish
Mobilization	6/21/89	6/23/89
Groundwater Sampling*	6/23/89	7/07/89
Demobilization	7/07/89	7/12/89

* The time required to sample the groundwater is estimated to be two days in the field and one day for documentation of sampling activities. The estimated time window for the sampling task is about two weeks.

3

3.0 QUALITY ASSURANCE OBJECTIVES

The Work Plan for the Army Creek Landfill Site contains a summary of available site information, defines the data gaps and the RI/FS objectives, and identifies the types and amount of data necessary to complete the RI/FS. The objectives of this RI/FS are to identify risks to human health and the environment from past and potential future contamination to creek sediments, surface water and groundwater, and to develop remedial options for contaminated groundwater treatment and assess their effectiveness in reducing the risk from exposure to groundwater. An additional objective is to develop a summary of Remedial Alternatives for use by EPA in the development of the second ROD.

To meet these objectives, additional site data are required. The data collection and QA requirements described in this document are intended to provide data that are adequate in both number and quality to support completion of this Focused RI/FS.

3.1 DATA QUALITY OBJECTIVES (DQOs)

DQOs are qualitative and/or quantitative statements regarding the quality of data needed to support the RI/FS activities. To develop site-specific DQOs, the intended use of the data must be defined. This use must be balanced between data quality needs and time as well as cost constraints.

Specific analytical protocols are selected to meet the DQOs in the following ways:

- Compare Applicable or Relevant and Appropriate Requirements (ARARs), risk-based criteria, soil cleanup levels, and data needs for risk assessment or engineering purposes to the detection limits for available analytical methods.
- Select analytical methods to allow quantification of the analytes at levels sufficiently below soil cleanup levels to minimize the number of critical data points.

- Evaluate the maximum allowable variability in the data based on the ARARs comparison.

Table 3-1 presents a summary of the proposed sampling and analysis program for the Army Creek Landfill Site. The information in this table was developed to meet the RI/FS objectives.

3.2 PRECISION, ACCURACY, REPRESENTATIVENESS, COMPLETENESS, AND COMPARABILITY (PARCC) GOALS

The quality of a data set is measured by certain characteristics of the data, namely the PARCC parameters. These parameters are suitable for assessing the quality of CLP results. The PARCC parameters are described below.

3.2.1 Precision and Accuracy

Precision and accuracy characterize the amount of variability and bias inherent in a data set. Precision describes the reproducibility of measurements of the same parameter for a sample under the same or similar conditions. Precision is expressed as a range (the difference between two measurements of the same parameter) or as a relative percent difference (the range relative to the mean, expressed as a percent). Range and Relative Percent Difference (RPD) values are calculated as follows:

$$\text{Range} = OR - DR$$

and

$$RPD = \frac{OR - DR}{1/2(OR+DR)} \times 100\%$$

where:

OR = original sample result

DR = duplicate sample result

TABLE 3-1

SUMMARY OF GROUNDWATER SAMPLING AND ANALYSIS PROGRAM
ARMY CREEK LANDFILL SITE

Analysis Required	Data Use (A)	Selected Analytical Option	Target Detection Limit	Proposed Analytical Method	Source of Analysis	Number of Samples	Field Duplicate (B)	Field Blank (C)	Matrix Spike (D)	Trip Blank (E)
TCL Organics, 15-day turnaround	1,2,3	IV	CRDL	CLP Protocol	CLP-RAS/SAS	10	1	1	1	1
TAL Metals and Cyanide 15-day turnaround	1,2,3	IV	CRDL	CLP Protocol	CLP-RAS/SAS	10	1	1	1	0
TAL Dissolved Metals 15-day turnaround	1,2,3	IV	CRDL	CLP Protocol	CLP-RAS/SAS	10	1	1	1	0
Sulfide	1,3	III	1 mg/L	STD. METH. 427D	CLP-SAS	10	1	1	1	0
Ammonia	1,3	III	0.03 mg/L	STD. METH. 417A, 417E	CLP-SAS	10	1	1	1	0
TKN	1,3	III	0.03 mg/L	STD. METH. 420A, 417E	CLP-SAS	10	1	1	1	0
Phosphorous	1,3	III	0.01 mg/L	STD. METH. 424B,C-III,F	CLP-SAS	10	1	1	1	0
Nitrate & Nitrite	1,3	III	0.05 mg/L	EPA 353.2	CLP-SAS	10	1	1	1	0
Sulfate	1,3	III	5 mg/L	EPA 375.4	CLP-SAS	10	1	1	1	0
Chloride	1,3	III	5 mg/L	EPA 325.3	CLP-SAS	10	1	1	1	0
Alkalinity	1,3	III	4 mg/L	EPA 310.1	CLP-SAS	10	1	1	0	0
Acidity	1,3	III	10 mg/L	EPA 305.1	CLP-SAS	10	1	1	0	0

TABLE 3-1

SUMMARY OF GROUNDWATER SAMPLING AND ANALYSIS PROGRAM
ARMY CREEK LANDFILL SITE
PAGE TWO

Analysis Required	Data Use (A)	Selected Analytical Option	Target Detection Limit	Proposed Analytical Method	Source of Analysis	Number of Samples	Field Duplicate (B)	Field Blank (C)	Matrix Spike (D)	Trip Blank (E)
TSS	1,3	III	4 mg/L	EPA 160.2	CLP-SAS	10	1	1	0	0
TDS	1,3	III	10 mg/L	EPA 160.1	CLP-SAS	10	1	1	0	0
TOC	1,3	III	20 mg/L	EPA 415.1	CLP-SAS	10	1	1	0	0
pH	1	I	NA	NA	Field Analysis	10	1	0	0	0
Temperature	1	I	NA	NA	Field Analysis	10	1	0	0	0
D.O.	1	I	NA	NA	Field Analysis	10	1	0	0	0
Conductivity	1	I	NA	NA	Field Analysis	10	1	0	0	0

NOTES:

(A) 1 - Site Characterization
    2 - Risk Assessment
    3 - Engineering Evaluation of Alternatives

(B) Field Duplicate - A single sample split into two portions, each of which is submitted blindly to the laboratory. Assesses the overall precision of sampling and analysis program (also known as a Replicate Sample).

(C) Field Blank - These blanks are prepared prior to the sampling trip from analyte-free material. They are transported to the field and exposed to the same conditions as field samples. The caps are removed, preservatives added and other steps are taken to provide exposure equivalent to field samples.

(D) Matrix Spike - A sample collected with three times the sample volume. Sample is split in the lab and two portions spiked with known amounts of analytes before analysis.

(E) Trip Blank - These blanks are prepared prior to the sampling trip from analyte free materials. Preservatives are added and the containers sealed prior to the sampling trip. Without ever being unsealed, they travel with similar items used for sample collection.

The internal laboratory control limits for precision are three times the standard deviation of a series of RPD or range values. RPD values may be calculated for both laboratory and field duplicates, and can be compared to the control limits as a QA check.

Accuracy is the comparison between experimental and known calculated values expressed as a percent recovery (%R). Percent recoveries are derived from the analysis of a standard spiked into deionized water (standard recovery) or into actual samples (matrix spike or surrogate spike recovery). Percent recovery is calculated as follows:

$$\%R = \frac{E}{T} \times 100\%$$

where:

E = experimental result

T = true value or theoretical result

with

$$\text{Theoretical result} = \frac{(\text{Sample aliq.})(\text{Sample conc.}) + (\text{Spike aliq.})(\text{Spike conc.})}{\text{Sample aliquot}}$$

Control limits for accuracy are set at the mean plus or minus three times the standard deviation of a series of %R values. Organic %R values are set at the mean plus or minus two times the standard deviation.

Field and laboratory precision and accuracy performance can affect the attainment of project objectives, particularly when compliance with established criteria is based on laboratory analysis of environmental samples. Such criteria are used in risk assessment and screening of remedial alternatives. Given the uncertainties associated with field work and laboratory activity, the following overall minimum acceptable precision and accuracy goals are identified to meet the project objectives:

- Precision: ± 50 percent RPD
- Accuracy: ± 50 percent recovery

Analytical precision and accuracy will be evaluated upon receipt of the laboratory data. Analytical precision will be measured as the relative standard deviation of the data from the laboratory (internal) duplicates. Analytical accuracy measures the bias as the percent recovery from the matrix spike and surrogate spike samples. Contract Laboratory Program (CLP) requirements will be used.

Field sampling precision and accuracy are not easily measured. Field contamination, sample preservation, and sample handling will affect precision and accuracy. By following the appropriate ARCS III Program Standard Operating Procedures (SOPs, see Appendix A), precision and accuracy errors associated with field activities can be minimized. Field duplicates and field (equipment rinsate) blanks will be used to estimate the field sampling and accuracy based on the existing guidance that specifies the type and proportion of samples submitted for QA/QC (EPA, March 1987).

Validity of data with respect to its intended use will be assessed based on laboratory-supplied QA/QC data and protocols routinely employed for validation of CLP-Routine Analytical Services (RAS)/Special Analytical Services (SAS) results. In general, results that are rejected by the validation process will be disqualified from application to the intended use. Qualified data will be used to the greatest extent practicable.

3.2.2 Representativeness

Representativeness describes the degree to which analytical data accurately and precisely define the material being measured. Several elements of the sampling and sample-handling process must be controlled to maximize the representativeness of the analytical data. Sample collection, preservation, and storage are discussed in Sections 4.1 and 5.0 of this document. Section 4.3 of the Work Plan contains details on the site sampling program and the rationale for sampling locations.

Representativeness of data is also affected by sampling techniques. Sampling techniques are described in Section 4.3 of the POP. To ensure that the data are representative, ARCS III Program Standard Operating Procedures (SOPs) will be used.

3.2.3 Completeness

Completeness describes the amount of data generated that meets the objectives for precision, accuracy, and representativeness versus the amount of data expected to be obtained. Where analysis is precluded or where DQOs are compromised, effects on the overall investigation will be considered. Whether or not any particular sample is critical to the investigation will be evaluated in terms of the sample location, the parameter in question, the intended data use, and the risk associated with error.

Due to the focused nature of this RI/FS, each parameter is critical to the site assessment. Consequently, there exists some critical data requirements below which the objectives of the study will be compromised. If in the evaluation of laboratory results it becomes apparent that the data for a specific parameter are of insufficient quality, either with respect to the number of samples or an individual analysis, resampling of the deficient data points may be necessary.

3.2.4 Comparability

One of the objectives of the POP is to provide analytical data of comparable quality between sample locations. Data from previous groundwater sampling and analyses tasks has been questionable due to variations in proper handling and analyses protocols. Both analytical procedures (i.e., CLP) and sample collection techniques (as defined in ARCS III's SOPs) will maximize the comparability of the RI data within this investigation.

4

4.0 FIELD INVESTIGATION ACTIVITIES

The field investigation at the Army Creek Landfill Site will consist of field screening and sampling of water from approximately nine recovery wells. The wells are identified as Numbers 1, 10, 11, 12, 13, 27, 28, 29, and 31.

4.1 GENERAL FIELD GUIDELINES

4.1.1 Sample Identification System

Each sample taken at the Army Creek Landfill Site for analysis will be assigned a unique sample tracking number. The sample tracking number will consist of:

- The site
- The well sampled
- The QC designation code

The site code for the Army Creek Landfill Site will be AC. Each recovery well will be numbered according to Weston's designation. The QC codes are designated as follows:

- A = Duplicate
- B = Field Blank
- C = Rinsate Blank
- D = Trip Blank

Additional codes may be added, as necessary.

All samples will be labeled with the preprinted numbers in accordance with the standard method of the EPA and the Contract Laboratory Program (CLP).

All pertinent data concerning a sample and its identification will be recorded in the field logbook and the sample log sheet.

4.1.2 Sample Handling

Sample handling includes the field-related considerations connected with the selection of sample containers, preservatives, allowable holding times, and the analyses requested. The EPA User's Guide to the Contract Laboratory Program (EPA, December 1986) and the Federal Register (EPA, October 26, 1984) address the topics of containers and sample preservation. Table 4-1 provides a site-specific summary of all sample handling considerations.

4.1.3 Sample Packing and Shipping

Samples will be packaged and shipped in compliance with the EPA User's Guide to the Contract Laboratory Program (EPA, December 1986), any applicable Department of Transportation regulations, and ARCS III Program SOPs. All samples will be shipped within 48 hours or preserved and kept cool for shipment at a later time depending on holding time requirements. The material will be kept in a locked, secure area. Sample storage and custody is further discussed in Section 5.0. The FOL or his designee will be responsible for contacting the EPA Sample Management Office (SMO) for each shipment by 10 a.m. the day after samples are shipped and will report the following:

- Sampler name and telephone number.
- Case number and/or SAS number of the project.
- Site name/code.
- Number(s), matrix(ces), and concentration(s) of samples shipped.
- Laboratory(ies) to which the samples were shipped.
- Carrier name and air bill number(s) for the shipment.
- Method of shipment (e.g., overnight, 2-day).
- Date of shipment.
- Suspected hazards associated with the samples or site.

4.1.4 Documentation

Custody of samples must be maintained and documented at all times. Chain-of-custody begins with receipt of the sample bottles followed by the collection of the samples in the field. Procedures used for chain-of-custody

TABLE 4-1

SUMMARY OF ANALYSES, BOTTLE REQUIREMENTS, PRESERVATION REQUIREMENTS, AND HOLDING TIMES ARMY CREEK LANDFILL SITE

Analysis	Number of Samples	Number of Containers per Sample	Type of Container	Preservation Requirements	Holding Time(1)
TCL Organics, VOA 15-day turnaround	14	3	40-ml glass vial	4 degree C	7 days from collection date
TCL Organics, B/N/A & Pesticide, 15-d t.a.	13	2	80-oz amber glass bottle	4 degree C	7 days to extraction from collection 40 days afterwards
TAL Metals 15-day turnaround	13	1	1-liter PE bottle	4 deg. C, Nitric Acid to pH < 2	28 days for Hg, 6 months others
Cyanide 15-day turnaround	13	1	1-liter PE bottle	4 deg. C, NaOH to pH > 12	14 days
TAL Dissolved Metals 15-day turnaround	13	1	1-liter PE bottle	4 deg. C, Nitric Acid to pH < 2	28 days for Hg, 6 months others
Sulfide	13	2	300 ml BOD bottle	4°C, 12 drops of 2N Zinc Acetate, fill sample, NaOH to pH>9, stopper w/o headspace	7 days
Ammonia & TKN	13	2	1-liter PE bottle	4 deg. C, Sulfuric Acid to pH < 2	28 days
Phosphorous	13	1	80-oz glass bottle	4 deg. C, Mercuric Chloride at 40 mg/l	48 hours
Nitrate & Nitrite	13	1	1-liter PE bottle	4 deg. C, Sulfuric Acid to pH < 2	48 hours
Sulfate & Chloride	13	1	1-liter PE bottle	4 degree C	28 days
Alkalinity & Acidity	12	1	1-liter PE bottle	4 degree C	14 days
TSS & TDS	12	1	1-liter PE bottle	4 degree C	7 days
TOC	12	2	40-ml glass vial	4 deg. C, Sulfuric Acid to pH < 2	28 days
pH, Temperature, D.O., Conductivity	11	NA	NA	NA	Analyzed immediately

NOTES:
(1) From Validated Time of Sample Receipt except where noted.

will be in compliance with RCRA Inspection Manual (EPA, 1980). Appendix A provides a description of the chain-of-custody procedures to be followed. An example of the chain-of-custody record is included in Appendix B.

In addition to the EPA-required CLP documentation (e.g., traffic reports) and QA of samples, certain standard forms will be completed for sample description and documentation. These shall include the sample log sheet and project sample summaries. An example of these forms can be found in Appendix B.

A bound/weatherproof field notebook shall be maintained by each sampling event leader and the HSSO. The FOL, or designee, shall record all information related to sampling or field activities. This information may include sampling time, weather conditions, unusual events, field measurements, description of photographs, etc.

The site logbook(s) shall be maintained by the FOL. The logbook(s) will contain a summary of the day's activities and will reference the field notebooks when applicable.

At the completion of field activities, the FOL shall submit to the Project Manager all field records, data, field notebooks, logbooks, chain-of-custody receipts, sample log sheets, daily logs, etc. The Project Manager shall ensure that these materials are entered into the ARCS Program document control system in accordance with appropriate administrative guidelines.

Changes in project operating procedures may be necessary as a result of changed field conditions or unanticipated events. A summary of the sequence of events associated with field changes is as follows:

- The FOL notifies the Project Manager of the need for change.
- If necessary, the Project Manager will discuss the change with the pertinent individuals (e.g., EPA Region III RPM) and will provide a verbal approval or denial to the FOL for the proposed change.
- The FOL will document the change on a TMR form (See Appendix B) and forward the form to the Project Manager at the earliest convenient time (e.g., end of the work week).

- The Project Manager, will sign the form and distribute copies to the RPM, QA Manager, FOL, and the project file.
- A copy of the completed TMR form will also be attached to the field copy of the affected document (i.e., Work Plan and the POP).

4.2 GENERAL FIELD OPERATIONS

4.2.1 Mobilization/Demobilization

Following approval of the Work Plan and this POP, GFEE will begin mobilization activities. All field team members will review the Work Plan, the POP, and the HASP which is contained in Appendix C of this document. In addition, a field team orientation meeting will be held to familiarize personnel with the scope of the RI field activities.

Equipment mobilization may include, but will not be limited to, the mobilization and setup of the following equipment:

- Field screening equipment
- Sampling equipment
- Health, safety, and decontamination equipment

The FOL will coordinate the mobilization of the equipment prior to arriving on site. The FOL will also make any necessary equipment purchases in order to conduct the field investigation. The equipment for the health, safety, and decontamination tasks will be loaded in Harrisburg and driven to the site under the guidance of the team FOL, health and safety specialist, or geologist.

Equipment for groundwater sampling will be mobilized separately and transported by field technicians from Harrisburg to the site. After field activities are completed, the field technicians will demobilize the equipment under the supervision of the FOL.

4.3 GENERAL SAMPLING OPERATIONS

Groundwater samples will be taken from the nine recovery wells. Since these wells are being continuously pumped, samples will be collected using the sampling bottles as the groundwater comes out of the well.

4.4 SAMPLE ANALYSIS

Samples collected at the Army Creek Landfill Site will be field-screened and submitted for laboratory analysis as presented in Table 4-1. This table also summarizes bottle requirements, preservation requirements, and holding time for each sample.

Samples of groundwater will be collected in beakers that have been properly decontaminated as described in Section 4.5. Field screening analyses that include pH, dissolved oxygen (DO), temperature, and conductivity will be performed with the samples collected in the beakers.

4.5 DECONTAMINATION

Prior to initiation of operations at the Army Creek Landfill Site, areas will be classified as the Support Zone, Contamination Reduction Zone, and Exclusion Zone, as appropriate. In addition, entrance and exit corridors for personnel and equipment will be established. Once established, these corridors will be the only pathways used to enter or exit the work area during non-emergency conditions. Emergency evacuation corridors, rally points, first-aid stations, and command posts will be identified and marked so as to be easily identified from anywhere in the work area.

Equipment involved in field sampling activities will be decontaminated prior to and during field activities. The standard decontamination steps of equipment will include:

- Potable water rinse
- Alconox or liquinox detergent wash
- Potable water rinse

- Acetone rinse
- Potable water rinse
- Distilled/deionized water rinse
- Air dry
- Wrap small equipment in aluminum foil, and place large equipment in clean plastic sheeting, if not used immediately.

All personnel wishing to exit the work area must pass through the established Contamination Reduction Corridor. Within the corridor, a systematic decontamination line will be set up according to the level of protection required. Specific details pertaining to decontamination stations, decontamination solutions, and disposal of protective suits and decontamination solutions can be found in the HASP of this report (Appendix C).

5

5.0 LABORATORY SAMPLE CUSTODY

To ensure the integrity of a sample from collection through analysis, it is necessary to have an accurate, written record that traces the possession and handling of the sample. This documentation is referred to as the sample chain-of-custody.

A sample is under custody if:

- The sample is in the physical possession of an authorized person.
- The sample is in view of an authorized person after being in his/her possession.
- The sample is placed in a secure area by an authorized person after being in his/her possession.
- The sample is in a secure area, restricted to authorized personnel only.

Sample custody during collection and shipping is discussed in Section 4.5.1 of ARCS III Program SOP SA-6.1. Laboratory sample custody is discussed in the following sections. All laboratories used in this study must follow these procedures.

5.1 SAMPLE RECEIPT

When samples are received, the shipping manifest is signed and dated to acknowledge sample receipt. The sample custodian must examine the shipping containers and verify that the correct number of containers were received. The shipping containers are then opened and the enclosed sample paperwork is removed.

Samples are removed from the shipping containers and the bottle condition must be noted. The information on the chain-of-custody form, the airbill, the packing list, the containers and sample tags, and the laboratory request is reviewed to note any discrepancies. Discrepancies must be resolved through the SMO.

5.2 SAMPLE STORAGE

All samples received by the laboratory must be stored at 4°C until analysis. Laboratory holding times are specified by the CLP contract and are presented in Table 4-1.

5.3 LABORATORY SAMPLE TRACKING

Laboratory sample tracking procedures are discussed in Section F of the CLP Statement of Work (EPA, December 1987). All laboratories used must conform to these requirements.

6

6.0 CALIBRATION PROCEDURES AND FREQUENCY

Instruments used in the field and in the laboratory will be calibrated according to the procedures described below.

6.1 FIELD INSTRUMENTS

Several monitoring instruments may be used during field activities, including:

- pH meter
- DO meter
- Conductivity meter
- Thermometer
- Field air monitoring instruments

The pH, DO, and conductivity meters will be calibrated prior to mobilization and periodically at the discretion of the FOL. The calibration procedures and frequencies are described in the ARCS SOP SF-1.1 Section 5: Onsite Water Quality Testing (Appendix A). Air monitoring instruments QA is described in Appendix D.

Calibration of sample monitoring equipment will be documented in an Equipment Calibration Log (ARCS III Program SOP SA-6.4, Appendix B). During calibration, an appropriate maintenance check will be performed on each piece of equipment. If damaged or defective parts are identified during the maintenance check, and it is determined that the damage could have an impact on the instrument's performance, the instrument will be removed from service until the defective parts are repaired or replaced. Usage, maintenance, and repair will be documented in the Equipment Calibration Log.

6.2 LABORATORY INSTRUMENTS

Calibration frequency to be performed at off-site laboratories for each of the environmental measurement parameters is presented below.

- Target Compound List Organics--Presented in CLP Statement of Work. (February, 1988).
- Total Organic Carbon (TOC) Content--Calibration verification as per manufacturer's instructions or weekly at a minimum.

The QC procedure routinely employed in organic chemistry analyses are presented below.

Standardization

Precision and accuracy are an integral part of QC, but they are only effective when instrument, solutions, and procedures have been standardized.

Solutions are standardized by preparing standards of known purity and concentrations, and using these standards to evaluate other solutions. Standards are either traceable to the National Bureau of Standards, or they are certified by the manufacturer to contain a known concentration of analyte.

Standardization of instruments and methods are accomplished by preparing series of standard solutions and analyzing the standards according to a written procedure. From the results of the standard analyses, standard curves are constructed and used to determine the concentration of the species in each sample.

Verification Standards

In general, methods that do not require a complete daily standard curve require the analysis of at least one standard each day to verify instrument and method performance. The results of the daily standard analyses must be within the QC limits, which are set at the number of available values ± three times the standard deviation. Appropriate corrective measures must be taken if the acceptance criteria are not met.

Preparation Blanks

As a check for glassware and reagent contamination, an aliquot of deionized water is taken through each preparation procedure each day that samples are prepared.

Duplicates

One in 20 samples that are analyzed for a specific parameter is run in duplicate. Different aliquots are often used in many instances to conserve samples and to test for matrix interferences. RPD is calculated and compared to the internally established control limits as described in the QC section of the QA/QC program summary.

Matrix Spikes

One in 20 samples that are analyzed for a specific parameter is spiked with the analyte for those parameters for which a stable standard is available. An aliquot of standard solution is added to the sample. Percent recovery is calculated and compared to the internally established control limits as described in the QC section of the QA/QC program summary.

7

7.0 ANALYTICAL PROCEDURES

Groundwater samples will be collected for chemical analyses. The analytical procedures to be used have been summarized in Table 3-1. Method references are included as footnotes. Details of the analytical procedures for the SAS requested for the Army Creek Landfill RI/FS are described in Appendix E. The operational procedures for field screening using pH, DO, and conductivity meters are presented in ARCS SOP SF-1.1 (Appendix A).

8

8.0 CHEMICAL DATA REDUCTION, VALIDATION, AND REPORTING

Data reduction, validation, and reporting will be conducted as described below.

8.1 DATA REDUCTION

The calculation of final results from raw data varies from parameter to parameter with the calibration approach. The ratio of analyte concentration to instrument response is determined for one or more standards. In general, if the concentration/instrument response ratio is linear, the average of the ratios is used to calculate sample results. If the response is not linear, response is plotted against concentration and sample results are quantified from the resultant curve.

Results are generally expressed to two significant figures. Results for aqueous samples are expressed in ug/l.

8.2 DATA VALIDATION AND REPORTING

The results of QC checks are the primary tools used for data validation. QC checks are described in Section 9.0. Acceptance criteria (control limits) are also discussed in Section 3.0. Raw data and final results are reviewed by the laboratory group leader on a daily basis. The group leader confirms that documentation is complete and legible; that qualitative identifications are accurate; that results are expressed in the appropriate units and number of significant figures; and that the required QC checks have been run and meet acceptance criteria. Review and approval of the data is documented by the group leader.

The chemical-analytical data generated by the CLP laboratory will be reviewed by GFEE and the NUS Chemistry and Toxicology Group. Review of the chemical-analytical data will include a quality assurance assessment to determine whether specified protocols were followed by laboratory personnel.

Results for field blanks and duplicates will be reviewed for consistency (i.e., RPD values) and to identify laboratory artifacts. The CLP laboratory will provide reagent blank, surrogate spike, matrix spike, and matrix spike duplicate results. This information will also be reviewed through comparison with the specified control limits. All data validation will be performed in accordance with the latest National EPA Functional Guidelines and Region III CRL Guidelines. Documentation of the validity of laboratory results will be provided to the EPA in the form of letter reports. Only validated data accepted by EPA will be put to the designated use.

Chemical-analytical data generated during the study will be reduced to a concise form for presentation in the RI Report. The analytical results will be managed using a computer program. This program is capable of handling all Target Compound List (TCL) organic and Target Analyte List (TAL) inorganic chemicals, and will be customized for the Army Creek Landfill Site to accommodate all indicator parameters. QA procedures will be implemented to detect errors occurring during data entry.

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9.0 INTERNAL QUALITY CONTROL CHECKS

QC checks to be implemented in the field and in the laboratory are described below.

9.1 FIELD QC CHECKS

In addition to periodic calibration of field equipment and appropriate documentation, QC samples will be collected or generated during environmental sampling activities. Types of field QC samples are defined as follows:

Field Blanks--These blanks are prepared prior to the sampling trip from clean, analyte-free materials closely resembling the sample matrices to be encountered in the actual samples. They consist of containers and chemicals/reagents transported to the field and exposed to the same conditions as field samples. Caps are removed from containers, preservatives are added and other related steps are taken to provide the blank with exposure to contamination equivalent to that of the field samples. Although similar to a trip blank, the field blank differs in that it is unsealed during the sample period.

Trip Blanks--These blanks are similar to field blanks with the exception that they are not exposed to field conditions. Preservatives are added and containers sealed prior to the sampling trip. Without ever being unsealed, they travel with similar items used for sample collection.

Rinsate Blanks--A sample of laboratory pure water used as the final rinsate after sampling equipment is cleaned in the field for reuse.

Field Duplicates--Field duplicates are samples that are divided into two portions at the time of sampling. Field duplication provides precision information regarding homogeneity, handling, shipping, storing, preparation, and analysis. Field duplicates will be submitted at a frequency of one per every 20 samples, or one per sampling trip if less than 20 samples are collected during a trip.

The proposed field QC samples are listed in Table 3-1.

9.2 LABORATORY QC CHECKS

Laboratory analysis will be conducted in accordance with the appropriate analytical methods (Table 3-1). Internal laboratory QC checks include surrogate and matrix spike addition, and analysis and reagent blank generation and analysis. Laboratory QC procedures for organic and inorganic analyses are summarized in the CLP Statement of Work (EPA 1986, 1987).

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10.0 PERFORMANCE AND SYSTEM AUDITS

10.1 FIELD AUDITS

Due to the limited time period for field activities (two days) a field audit is not considered feasible.

10.2 LABORATORY AUDITS

Blind or double-blind Performance Evaluation (PE) samples are sent to CLP laboratories on a quarterly basis. The CLP Statement of Work contains specific requirements for performance and systems audits.

If non-CLP laboratories are used for SAS analyses, they must be fully certified and approved. QA/QC procedures must be in use. An internal audit schedule must be available, as well as a record of audits by certification agencies. The results of all audits and the corrective action process must be available.

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11.0 PREVENTIVE MAINTENANCE

Measuring equipment used in environmental monitoring or analysis, and test equipment used for calibration and maintenance shall be controlled by established procedures. Measuring and test equipment shall have an initial calibration and shall be calibrated at scheduled intervals against certified standards, according to the CLP Statement of Work, Attachment E, or the procedures for other methods. Equipment will be calibrated periodically. Test equipment used for calibration of sensors shall also be calibrated at least once a year or when maintenance or damage indicates a need for recalibration. The GFEE equipment center will provide spare equipment as needed and it will have rapid access to sources of spare parts as necessary.

The air monitoring devices used for screening will be cleaned and maintained in the manner specified in Appendix D, Quality Assurance Program for Air Monitoring Instrumentation.

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12.0 DATA ASSESSMENT FOR PRECISION, ACCURACY, AND COMPLETENESS

The procedures used to assess precision, accuracy, and completeness (PAC) of laboratory data are described in Section 3.0. Upon completion of the analytical phases of the project, data will be reviewed and validated as outlined in Section 8.0. In conjunction with the data review and validation, the specific PAC results will be compared with the laboratory QC criteria and the completeness objective. Discrepancies may affect the usefulness of the data.

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13.0 CORRECTIVE ACTION

The need for corrective actions may become apparent during surveillance of activities, procurement of services and supplies, or other operations that may affect the quality of work. Deficiencies and nonconformances will be promptly identified by the QAR's QA checks outlined in this POP. Corrective action will be taken immediately.

The identification of significant conditions adverse to quality, the cause of the conditions, and the corrective actions shall be documented and reported to the EPA Project Officer at a frequency specified by the RPM. Critical problems encountered are to be reported within 24 hours to the Project Officer. The GFEE Project Manager will have overall responsibility for corrective actions.

The corrective action program covers the analysis of the cause of any negative audit findings and the corrective actions required. This program includes the investigation of the cause of significant or repetitious unsatisfactory conditions relating to the quality of sampling, service, or the failure to implement or adhere to required QA practices.

In accordance with CLP Protocols and the data requirements of the project, the RPM and GFEE Project Manager will determine the appropriate action(s), i.e., additional sampling, and implement them as required.

1

14.0 QUALITY ASSURANCE REPORTS TO MANAGEMENT

The QAR shall forward to the QA Officer a report summarizing the QA and QC status for the project and any conditions adverse to quality. Topics to be included in the report are as follows:

- Status of project (time table).
- Assessment of measurement data accuracy, precision, and completeness.
- Results of any audits.
- Results of surveillances.
- Any nonconformances initiated.
- Training provided to project personnel.
- Any significant QA problems, together with recommended solutions.
- Any significant changes in the POP.

The QA Officer will compile the reports from the QAR into a composite report for the Project Manager.

REFERENCES

REFERENCES

ARMY CREEK LANDFILL SITE

U.S. Environmental Protection Agency (1980), RCRA Inspection Manual.

U.S. Environmental Protection Agency/U.S. Army Corps of Engineers (May, 1981), Procedures for Handling and Chemical Analysis of Sediment and Water Samples.

U.S. Environmental Protection Agency (October 26, 1984), Federal Register.

U.S. Environmental Protection Agency (December, 1986), User's Guide to the Contract Laboratory Program. Office of Emergency and Remedial Response.

U.S. Environmental Protection Agency (March, 1987), Data Quality Objectives for Remedial Response Activities. EPA 540/G-87/003A.

U.S. Environmental Protection Agency (July, 1987), Contract Laboratory Program.

U.S. Environmental Protection Agency (February, 1989); Technical Direction Memorandum, Review of Army Creek and Delaware Sand and Gravel RI/FS Study and ROD Documents; EPA Work Assignment No. 37-07-3L34, GFEE/NUS.

APPENDICES

APPENDICES

A

APPENDIX A

STANDARD OPERATING PROCEDURES FOR RI ACTIVITIES

ARMY CREEK LANDFILL SITE

APPENDIX A

STANDARD OPERATING PROCEDURES FOR RI ACTIVITIES

NUS CORPORATION

WASTE MANAGEMENT SERVICES GROUP

STANDARD OPERATING PROCEDURES

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Effective Date 01/01/88	Revision 0
Applicability WMSG	
Prepared Earth Sciences	
Approved A. K. Bomberger, P.E.	

Subject
SAMPLE IDENTIFICATION AND CHAIN-OF-CUSTODY

TABLE OF CONTENTS

1.0 PURPOSE

This purpose of this procedure is to provide information on chain-of-custody procedures to be used under the NUS Program.

2.0 SCOPE

This procedure describes the steps necessary for transferring samples through the use of Chain-of-Custody Records. A Chain-of-Custody Record is required, without exception, for the tracking and recording of all samples collected for on-site or off-site analysis (chemical or geotechnical) during program activities. Use of the Chain-of-Custody Record Form creates an accurate written record that can be used to trace the possession and handling of the sample from the moment of its collection through analysis and its introduction as evidence. This guideline identifies the necessary custody records and describes their completion.

This guideline does not take precedence over region-specific or site-specific requirements for chain-of-custody.

3.0 GLOSSARY

Chain-of-Custody Record Form - A Chain-of-Custody Record Form is a printed two-part form that accompanies a sample or group of samples as custody of the sample(s) is transferred from the custodian to the subsequent custodian. Attachment C shows the Chain-of-Custody Records used by EPA Region III. A Chain-of-Custody Record Form is a controlled document, provided by the regional office of EPA. One copy of the form must be retained in the project file.

Controlled Document - A consecutively-numbered form released by EPA or Program Management Office (PMO) for use on a particular work assignment. All unused forms must be returned or accounted for at the conclusion of the assignment.

- Custodian - The person responsible for the custody of samples at a particular time, until custody is transferred to another person (and so documented), who then becomes custodian. A sample is under your custody if:
  - It is in your actual possession.
  - It is in your view, after being in your physical possession.
  - It was in your physical possession and then you locked it up to prevent tampering.
  - It is in a designated and identified secure area.
- Sample - A sample is physical evidence collected from a facility or the environment, which is representative of conditions at the point and time that it was collected.

4.0 RESPONSIBILITIES

- Field Operations Leader - Responsible for determining that chain-of-custody procedures are implemented up to and including release to the shipper
- Field Samplers - Responsible for initiating the Chain-of-Custody Record and maintaining custody of samples until they are relinquished to another custodian, to the shipper, or to the common carrier

- Remedial Investigation Leader - Responsible for determining that chain-of-custody procedures have been met by the sample shipper and analytical laboratory.

5.0 PROCEDURES

5.1 OVERVIEW

The term "chain-of-custody" refers to procedures which ensure that evidence presented in a court of law is what it is represented to be. The chain-of-custody procedures track the evidence from the time and place it is first obtained to the courtroom and, secondly, provide security for the evidence as it is moved and/or passes from the custody of one individual to another. In addition, procedures for consistent and detailed records facilitate the admission of evidence under Rule 803(b) of the Federal rules of Evidence (P.L. 93-575).

Chain-of-custody procedures, recordkeeping, and documentation are an important part of the management control of samples. Regulatory agencies must be able to provide the chain of possession and custody of any samples that are offered for evidence, or that form the basis of analytical test results introduced as evidence. Written procedures must be available and followed whenever evidence samples are collected, transferred, stored, analyzed, or destroyed.

5.2 SAMPLE IDENTIFICATION

The method of identification of a sample depends on the type of measurement or analysis performed. When in-situ measurements are made, the data are recorded directly in bound logbooks or other field data records, with identifying information (project code, station numbers, station location, date, time, samplers), filed observations, and remarks, and signed.

5.2.1 Sample Label

Samples, other than in-situ measurements, are removed and transported from the sample location to a laboratory or other location for analysis. Before removal, however, a sample is often divided into portions, depending upon the analyses to be performed. Each portion is preserved in accordance with the Sampling Plan. Each sample container is identified by a sample label (see Attachment A) Sample labels are provided by PMO. The information recorded on the sample label includes:

Project	EPA Work Assignment Number
Sample No.	The unique sample number identifying this sample
Date	A six-digit number indicating the day, month, and year of sample collection; e.g., 12/21/85.
Time	A four-digit number indicating the 24-hour time of collection (for example: 0954 is 9:54 a.m., and 1629 is 4:29 p.m.)
Medium	Water, Soil, Sediment, Sludge, Leachate, etc.
Sample Type	Grab or Composite
Preservation	Type, quantity, and concentration of preservative added.
Analysis	Same as Analyses on Sample Identification Tag
Sampled By	Name of the sampler
Lab #	Sample number assigned by the receiving laboratory (not used for onsite analyses)
Remarks	If for CLP analysis, include the CLP case or SAS number, and CLP sample number from the traffic report, SAS Packing List, or Dioxin Shipment Record. Also, pertinent observations of the sampler (e.g., sequence number for sequential samples)

Using just the EPA work assignment number of the sample label maintain the anonymity of sites. This may be necessary, even to the extent of preventing the laboratory performing analysis from knowing the identity of the site (e.g., if the laboratory is part of an organization that has performed previous work on the site).

5.2.2 Sample Identification Tag

A Sample Identification Tag (Attachment B) must also be used for samples collected for CLP (Contract Laboratory Program) analysis. The Sample Identification Tag is a white, waterproof paper label, approximately 3-by-6 inches, with a reinforced eyelet, and string or wire for attachment to the neck of the sample bottle. The Sample Tag is a controlled document, and is provided by the regional EPA office. Following sample analysis, the Sample Tag is retained by the laboratory as evidence of sample receipt and analysis.

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The following information is recorded on the tag:

Project Code	EPA Work Assignment Number
Station Number	Same as Sample Number on Sample Label
Month/Day/Year	Same as Date on Sample Label
Time	Same as Time on Sample Label
Designate: Comp/Grab	Same as Sample Type on Sample Label
Station Location	Site-specific station location designation defined in Site Operations Plan
Samplers	Same as Sampled By on Sample Label
Preservative	Yes or No
Analyses	Check appropriate box(es)
Remarks	Same as Remarks on Sample Label (make sure CLP Case No./SAS No. and CLP sample numbers are recorded)
Lab Sample No.	Same as Laboratory number on sample bottle

The tag is then tied round the neck of the sample bottle.

If the sample is to be split, it is aliquoted into similar sample containers. Identical information is completed on the label attached to each split and one of these is marked "Split" on the "Remarks" line.

Blank, duplicate, or field spike samples should not be identified as such on the label, as they may compromise the quality control function. Sample blanks, duplicates, spikes, and splits are defined in Procedure SA-6.6.

5.3 CHAIN-OF-CUSTODY PROCEDURES

After collection, separation, identification, and preservation, the sample is maintained under chain-of-custody procedures until it is in the custody of the analytical laboratory and has been stored or disposed of.

5.3.1 Field Custody Procedures

- Samples are collected as described in the site-specific Sampling Plan. Care must be taken to record precisely the sample location and to ensure that the sample number on the label matches the sample log sheet and Chain-of-Custody Record exactly.
- The person undertaking the actual sampling in the field is responsible for the care and custody of the samples collected until they are properly transferred or dispatched

- When photographs are taken of the sampling as part of the documentation procedure, the name of the photographer, date, time, site location, and site description are entered sequentially in the site logbook as photos are taken. Once developed, the photographic prints shall be serially numbered, corresponding to the logbook descriptions.

- Sample labels shall be completed for each sample, using waterproof ink unless prohibited by weather conditions, e.g., a logbook notation would explain that a pencil was used to fill out the sample label because a ballpoint pen would not function in freezing weather.

5.3.2 Transfer of Custody and Shipment

Samples are accompanied by a Chain-of-Custody Record Form. The Chain-of-Custody Records used in EPA Region III is shown in Attachment C. The appropriate form should be obtained from the EPA Regional Office. When transferring the possession of samples, the individuals relinquishing and receiving will sign, date, and note the time on the Record. This Record documents sample custody transfer from the sampler, often through another person, to the analyst in the laboratory. The Chain-of-Custody Record is filled out as follows:

- Enter header information (project number and name, CLP case No. or SAS No.). For each station number, enter date, time, composite/grab, station location, number of containers, analytical parameters, Traffic Report/SAS Packing List/Dioxin Shipment Record, and Sample Identification Tag Number (in Remarks column).

- Sign, date, and enter the time under "Relinquished by" entry.

- Make sure that the person receiving the sample signs the "Received by" entry, or enter the name of the carrier (e g., UPS, Federal Express) under "Received by " Receiving laboratory will sign "Received for Laboratory by" on the lower line and enter the date and time.

- Enter the bill-of-lading or Federal Express airbill number under "Remarks," if appropriate

- Place the original (top, signed copy) of the Chain-of-Custody Record Form in the appropriate sample shipping package. Retain a copy with field records

- Sign and date the custody seal, a 1- by 3-inch white paper label with black lettering and an adhesive backing. Attachment D is an example of a custody seal. The custody seal is part of the chain-of-custody process and is used to prevent tampering with samples after they have been collected in the field. Custody seals are provided by ZPMO on an as-needed basis.

- Place the seal across the shipping container opening so that it would be broken if the container is opened.

- Complete other carrier-required shipping papers.

The custody record is completed using black waterproof ink. Any corrections are made by drawing a line through and initialing and dating the change, then entering the correct information. Erasures are not permitted

Common carriers will usually not accept responsibility for handling Chain-of-Custody Record Forms this necessitates packing the record in the sample container (enclosed with other documentation in a plastic zip-lock bag). As long as custody forms are sealed inside the sample container and the custody seals are intact, commercial carriers are not required to sign off on the custody form.

If sent by mail, the package will be registered with return receipt requested. If sent by common carrier or air freight, proper documentation must be maintained.

The laboratory representative who accepts the incoming sample shipment signs and dates the Chain-of-Custody Record, completing the sample transfer process. It is then the laboratory's responsibility to maintain internal logbooks and custody records throughout sample preparation and analysis.

5.3.3 *Receipt for Samples Form*

Whenever samples are split with a private party or government agency, a separate Receipt for Samples Record Form is prepared for those samples and marked to indicate with whom the samples are being split. The person relinquishing the samples to the party or agency shall require the signature of a representative of the appropriate party acknowledging receipt of the samples. If a representative is unavailable or refuses to sign, this is noted in the "Received-by" space. When appropriate, as in the case where the representative is unavailable, the custody record should contain a statement that the samples were delivered to the designated location at the designated time. *This form must be completed and a copy given to the owner, operator, or agent-in-charge* even if the offer for split samples is declined. The original is retained by the Field Operations Leader

6.0 REFERENCES

USEPA, 1984. User's Guide to the Contract Laboratory Program, Office of Emergency and Remedial Response, Washington, D.C.

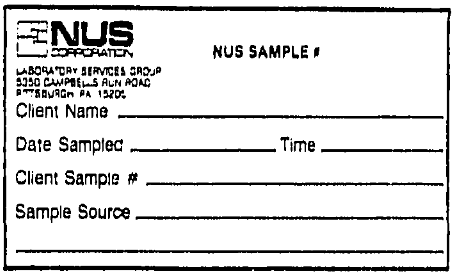
Ebasco Services Incorporated; REM III Field Technical Guideline No. FT-7.04, October 30, 1987.

Ebasco Services Incorporated; REM III Field Technical Guideline No. 7.05, October 30, 1987

7.0 RECORDS

Attachment A	-	Sample Label
Attachment B	-	Sample Identification Tag
Attachment C	-	Chain-of-Custody Record Form for use in Region III
Attachment D	-	Chain-of-Custody Seal

ATTACHMENT A
SAMPLE LABEL



NUS CORPORATION
LABORATORY SERVICES GROUP
5350 CAMPBELLS RUN ROAD
PITTSBURGH, PA 15205

NUS SAMPLE #

Client Name ______________________

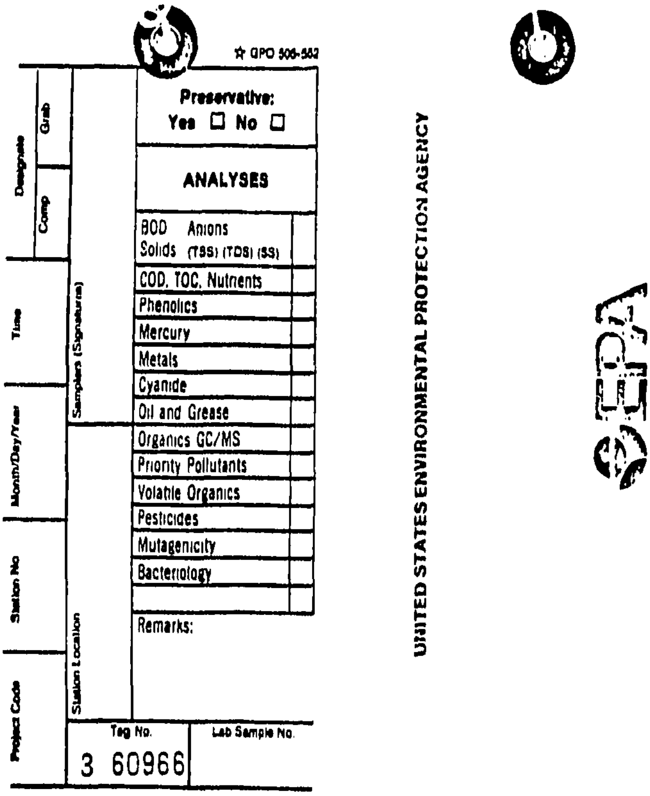
Date Sampled ____________ Time ________

Client Sample # ____________________

Sample Source _____________________

____

ATTACHMENT B
SAMPLE IDENTIFICATION TAG



☆ GPO 506-552

Preservative:
Yes ☐ **No** ☐

ANALYSES

- BOD Anions Solids (TSS) (TDS) (SS) ☐
- COD, TOC, Nutrients ☐
- Phenolics ☐
- Mercury ☐
- Metals ☐
- Cyanide ☐
- Oil and Grease ☐
- Organics GC/MS ☐
- Priority Pollutants ☐
- Volatile Organics ☐
- Pesticides ☐
- Mutagenicity ☐
- Bacteriology ☐

Remarks:

Designate: Grab / Comp

Time

Month/Day/Year

Station No

Project Code

Samplers (Signature)

Station Location

Tag No.: 3 60966

Lab Sample No.

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

EPA

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ATTACHMENT C
CHAIN-OF-CUSTODY RECORD FORM FOR USE IN REGION III
(Original is 8-1/2 x 11-3/4")

ENVIRONMENTAL PROTECTION AGENCY
Office of Enforcement

REGION 3
Curtis Bldg., 6th & Walnut Sts
Philadelphia, Pennsylvania 19106

CHAIN OF CUSTODY RECORD

PROJ NO.	PROJECT NAME					NO OF CONTAINERS								REMARKS
SAMPLERS *(Signature)*														
STA NO	DATE	TIME	COMP	GRAB	STATION LOCATION									

Relinquished by: *(Signature)*	Date / Time	Received by *(Signature)*	Relinquished by *(Signature)*	Date / Time	Received by: *(Signature)*
Relinquished by *(Signature)*	Date / Time	Received by *(Signature)*	Relinquished by *(Signature)*	Date / Time	Received by: *(Signature)*
Relinquished by *(Signature)*	Date / Time	Received for Laboratory by *(Signature)*	Date / Time	Remarks	

Distribution: Original Accompanies Shipment; Copy to Coordinator Field Files

3-15966

ATTACHMENT D
CHAIN-OF-CUSTODY SEAL

NUS CORPORATION

WASTE MANAGEMENT SERVICES GROUP

STANDARD OPERATING PROCEDURES

Number: SA-6.2	Page: 1 of 12
Effective Date: 01/01/88	Revision: 0
Applicability: WMSG	
Prepared: Earth Sciences	
Approved: A. K. Bomberger, P.E.	

Subject: SAMPLE PACKAGING AND SHIPPING

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SAMPLE PACKAGING AND SHIPPING	SA-6.2	2 of 12
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1.0 PURPOSE

This procedure provides instruction for sample packaging and shipping in accordance with U.S. Department of Transportation (DOT) regulations.

2.0 SCOPE

Samples collected at hazardous waste sites usually have to be transported elsewhere for analysis. This requires that the samples be appropriately preserved to prevent or minimize chemical alteration prior to analysis, and be transported to protect their integrity, as well as to protect against any detrimental effects from leakage or breakage. Regulations for packaging, marking, labeling, and shipping hazardous materials and wastes are promulgated by the U.S. Department of Transportation and described in the Code of Federal Regulations (49 CFR 171 through 177, in particular 172.402h, Packages Containing Samples). In general, these regulations were not intended to cover shipment of samples collected at controlled or uncontrolled hazardous waste sites or samples collected during emergency responses. However, the EPA has agreed through a memorandum of agreement to package, mark, label, and ship samples observing DOT procedures. The information presented here is for general guidance.

This procedure is applicable to all samples taken from uncontrolled hazardous substance sites for analysis at laboratories away from the site.

3.0 GLOSSARY

Carrier - A person or firm engaged in the transportation of passengers or property.

Hazardous Material - A substance or material in a quantity and form which may pose an unreasonable risk to health and safety or property when transported in commerce ("commerce" here to include any traffic or transportation). Defined and regulated by DOT (49 CFR 173.2) and listed in Attachment A of this guideline.

Hazardous Waste - Any substance listed in 40 CFR Subpart D (§261.30 et seq) or otherwise characterized as ignitable, corrosive, reactive, or EP toxic as specified under 40 CFR Subpart C (§261.20 et seq) that would be subject to manifest requirements specified in 40 CFR 262. Defined and regulated by EPA.

Marking - Applying the descriptive name, instruction, cautions, weight, or specification marks or combination thereof required to be placed outside containers of hazardous materials.

n.o.i. - Not otherwise indicated

n.o.s. - Not otherwise specified

ORM - Other regulated material

Packaging - The assembly of one or more containers and any other components necessary to assure compliance with the minimum packaging requirements of 49 CFR 174, including containers (other than freight containers or overpacks), portable tanks, cargo tanks, tank cars, multi-unit tank car tanks.

Placard - *Color-coded, pictorial sign depicting the hazard class symbol and name to be placed on all* four sides of a vehicle transporting certain hazardous materials

Reportable Quantity (RQ) - A parenthetical note of the form "(RQ-1000/454)" following an entry in the DOT Hazardous Materials table (49 CFR 172.101) indicates the reportable quantity of the substance in pounds and kilograms. If a spill of that amount or more of the substance occurs during transit or storage, a report must be filed with DOT according to §171 15-15 concerning hazardous materials incidents reports *If the material spilled is a hazardous waste, a report must always be* filed, regardless of the amount, and must include a copy of the manifest. If the RQ notation appears, it must be shown either immediately before or after the proper shipping name on the shipping paper (or manifest). Most shipping papers and manifests will have a column designated "HM" which may be used for this purpose.

4.0 RESPONSIBILITIES

Field Operations Leader or Team Sampling Leader - responsible for determining that samples are properly packaged and shipped.

Sampling Personnel - responsible for implementing the packaging and shipping requirements

5.0 PROCEDURES

5.1 INTRODUCTION

Samples collected for shipment from a site should be classified as either environmental or hazardous material (or waste) samples. In general, environmental samples are collected off-site (for example *from streams, ponds, or wells) and are not expected to be grossly contaminated with high levels of* hazardous materials. On-site samples (for example, soil, water, and materials from drums or bulk storage tanks, obviously contaminated ponds, lagoons, pools, and leachates from hazardous waste sites) are considered hazardous. A distinction must be made between the two types of samples in order to:

- Determine appropriate procedures for transportation of samples. If there is any doubt, a *sample should be considered hazardous and shipped accordingly.*
- Protect the health and safety of laboratory personnel receiving the samples. Special precautions are used at laboratories when samples other than environmental samples are received.

5.2 ENVIRONMENTAL SAMPLES

5.2.1 Packaging

Environmental samples may be packaged following the procedures outlined in Section 5.4 for samples classified as "flammable liquids" or "flammable solids." Requirements for marking, labeling and shipping papers do not apply

Environmental samples may also be packed without being placed inside metal cans as required for flammable liquids or solids

- Place sample container, properly identified and with a sealed lid, in a polyethylene bag, and seal the bag
- Place sample in a fiberboard container or metal picnic cooler which has been lined with a *large polyethylene bag*
- Pack with enough noncombustible, absorbent, cushioning materials to minimize the possibility of the container breaking
- Seal large bag
- Seal or close outside container

5.2.2 Marking Labeling

Sample containers must have a completed sample identification tag and the outside container must be marked "Environmental Sample". The appropriate side of the container must be marked "This End up" and arrows placed appropriately. No DOT marking or labeling are required.

5.2.3 Shipping Papers

No DOT shipping papers are required. However, the appropriate chain-of-custody forms must be included with the shipment.

5.2.4 Transportation

There are no DOT restrictions on mode of transportation.

5.3 DETERMINATION OF SHIPPING CLASSIFICATION FOR HAZARDOUS MATERIAL SAMPLES

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

5.3.1 Known Substances

If the substance in the sample is known or can be identified, package, mark, label and ship according to the specific instructions for that material (if it is listed) in the DOT Hazardous Materials Table, 49 CFR 172 101. A copy of this table is available from Regional Program Offices or ZPMO.

Unz and Company have published the following steps to help in locating a proper shipping name from the Hazardous Materials Table, 49 CFR 172.101

1. Look first for the chemical or technical name of the material, for example, ethyl alcohol. Note that many chemicals have more than one technical name, for example, perchloroethylene (not listed in 172 101) is also called tetrachloroethylene (listed 172 101). It may be useful to consult a chemist for all possible technical names a material can have. If your material is not listed by its technical name then

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

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

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

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

5.3.2 Unknown Substances

For samples of hazardous substances of unknown content, select the appropriate transportation category according to the DOT Hazardous Materials Classification (Attachment A), a priority system of transportation categories.

The correct shipping classification for an unknown sample is selected through a process of elimination, utilizing Attachment A. Unless known or demonstrated otherwise (through the use of radiation survey instruments), the sample is considered radioactive and appropriate shipping regulations for "radioactive material" followed.

If radioactive material is eliminated, the sample is considered to contain "Poison A" materials (Attachment B), the next classification on the list. DOT defines "Poison A" as extremely dangerous poisonous gases or liquids of such a nature that a very small amount of gas, or vapor of the liquids, mixed with air is dangerous to life. Most Poison A materials are gases or compressed gases and would not be found in drum-type containers. Liquid Poison A would be found only in closed *containers; however,all samples taken from closed drums do not have to be shipped as Poison A,* which provides for a "worst case" situation. Based upon information available, a judgment must be made whether a sample from a closed container is a Poison A.

If Poison A is eliminated as a shipment category, the next two classifications are "flammable" or "nonflammable" gases. Since few gas samples are collected, "flammable liquid" would be the next applicable category. With the elimination of radioactive material, Poison A, flammable gas, and nonflammable gas, the sample can be classified as flammable liquid (or solid) and shipped accordingly. These procedures would also suffice for shipping any other samples classified below flammable liquids in the DOT classification table (Attachment A). For samples containing unknown materials, categories listed below flammable liquids/solids on Attachment A are generally not used because *showing that these materials are not flammable* liquids (or solids) requires flashpoint testing, which may be impractical and possibly dangerous at a site. Thus, unless the sample is known to consist of materials listed as less hazardous than flammable liquid (or solid) on Attachment A, it is considered a flammable liquid (or solid) and shipped as such.

For any hazardous material shipment, utilize the shipping checklist (Attachment C) as a guideline to ensure that all sample-handling requirements are satisfied.

5.4 PACKAGING AND SHIPPING OF SAMPLES CLASSIFIED AS FLAMMABLE LIQUID (OR SOLID)

5.4.1 Packaging

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

1. Collect sample in the prescribed container with nonmetallic, Teflon-lined screw cap. To prevent leakage, fill container no more than 90 percent full.
2. Complete sample label and identification tag and attach securely to sample container.
3. Seal container and place in 2-mil thick (or thicker) polyethylene bag, one sample per bag. Position identification tag so that it can be read through bag. Seal bag.
4. Place sealed bag inside metal can and cushion it with enough noncombustible, absorbent material (for example, vermiculite or diatomaceous earth) between the bottom and sides of the can and bag to prevent breakage and absorb leakage. Pack one bag per can. Use clips, tape, or other positive means to hold can lid securely, tightly and permanently. Mark can as indicated in Paragraph 1 of Section 5.4.2, below.
5. Place one or more metal cans (or single 1-gallon bottle) into a strong outside container, such as a metal picnic cooler or a DOT-approved fiberboard box. Surround cans with noncombustible, absorbent cushioning materials for stability during transport. Mark container as indicated in Paragraph 2 of Section 5.4.2.

5.4.2 Marking/Labeling

1. Use abbreviations only where specified. Place the following information, either hand-printed or in label form, on the metal can (or 1-gallon bottle):
   - Laboratory name and address
   - "Flammable Liquid, n.o.s. UN1993" or "Flammable Solid, n.o.s. UN1325."

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

2. Place all information on outside shipping container as on can (or bottle), specifically:
   - *Proper shipping name*
   - UN or NA number
   - Proper label(s)
   - Addressee and sender

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

5.4.3 Shipping Papers

1. Use abbreviations only where specified. Complete the carrier-provided bill of lading and sign certification statement (if carrier does not provide, use standard industry form, see Attachment D). *Provide the following information in the order listed (one form may be used for more than one exterior container)*

   - "Flammable Liquid, n.o.s. UN1993" or "Flammable Solid, n.o.s. UN1325."
   - "Limited Quantity" (or "Ltd. Qty.")
   - "Cargo Aircraft Only."
   - Net weight (wt) or net volume (vol), just before or just after "Flammable Liquid, n.o.s." or "Flammable Solid, n.o.s.," by item, if more than one metal can is inside an exterior container
   - "Laboratory Samples" (if applicable)

2. Include Chain-of-Custody Record, properly executed in outside container

3. "Limited Quantity" of "Flammable Liquid, n.o.s." is limited to one pint per inner container. For "Flammable Solid, n.o.s.," net weight of inner container plus sample should not exceed one pound; total package weight should not exceed 25 pounds

5.4.4 Transportation

1. Transport unknown hazardous substance samples classified as flammable liquids by rented or common carrier truck, railroad, or express overnight package services. Do not transport *by any passenger-carrying air transport system, even if they have cargo-only aircraft.* DOT regulations permit regular airline cargo-only aircraft, but difficulties with most suggest avoiding them. *Instead, ship by airline carriers that only carry cargo*

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

6.0 REFERENCES

U.S. Department of Transportation, 1983. Hazardous Materials Regulations, 49 CFR 171-177

NUS Standard Operating Procedure SA-6.1 - Sample Identification and Chain-of-Custody

NUS Standard Operating Procedure SA-1.2 - Sample Preservation

NUS Standard Operating Procedure SF-1.5 - Compatibility Testing

EBASCO Services Incorporated. REM III Field Technical Guideline No. FT-7.07, January 8, 1986

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7.0 RECORDS

Attachment A - DOT Hazardous Material Classification (49 CFR 173.2)
Attachment B - DOT List of Class "A" Poisons (49 CFR 172.101)
Attachment C - Hazardous Materials Shipping Checklist
Attachment D - Standard Industry Certification Form

ATTACHMENT A

DOT HAZARDOUS MATERIAL CLASSIFICATION (49 CFR 173.2)

1. Radioactive material (except a limited quantity)
2. Poison A
3. Flammable gas
4. Nonflammable gas
5. Flammable liquid
6. Oxidizer
7. Flammable Solid
8. Corrosive material (liquid)
9. Poison B
10. Corrosive material (solid)
11. Irritating material
12. Combustible liquid (in containers having capacities exceeding 110 gallons (416 liters))
13. ORM-B
14. ORM-A
15. Combustible liquid (in containers having capacities of 110 gallons (416 liters) or less)
16. ORM-E

ATTACHMENT B

DOT LIST OF CLASS "A" POISON (49 CFR 172.101)

Material	Physical State at Standard Temperature
Arsine	Gas
Bromoacetone	Liquid
Chloropicrin and methyl chloride mixture	Gas
Chloropicrin and nonflammable, nonliquefied compressed gas mixture	Gas
Cyanogen chloride	Gas (> 13.1°C)
Cyanogen gas	Gas
Gas identification set	Gas
Gelatin dynamite (H. E. Germaine)	....
Grenade (with Poison "A" gas charge)	....
Hexaethyl tetraphosphate/compressed gas mixture	Gas
Hydrocyanic (prussic) acid solution	Liquid
Hydrocyanic acid, liquefied	Gas
Insecticide (liquefied) gas containing Poison "A" or Poison "B" material	Gas
Methyldichloroarsine	Liquid
Nitric oxide	Gas
Nitrogen peroxide	Gas
Nitrogen tetroxide	Gas
Nitrogen dioxide, liquid	Gas
Parathion/compressed gas mixture	Gas
Phosgene (diphosgene)	Liquid

ATTACHMENT C
HAZARDOUS MATERIALS SHIPPING CHECKLIST

PACKAGING

1. Check DOT 172.500 table for appropriate type of package for hazardous substance
2. Check for container integrity, especially the closure.
3. Check for sufficient absorbent material in package
4. Check for sample tags and log sheets for each sample, and chain-of-custody record

SHIPPING PAPERS

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

RCRA MANIFEST

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

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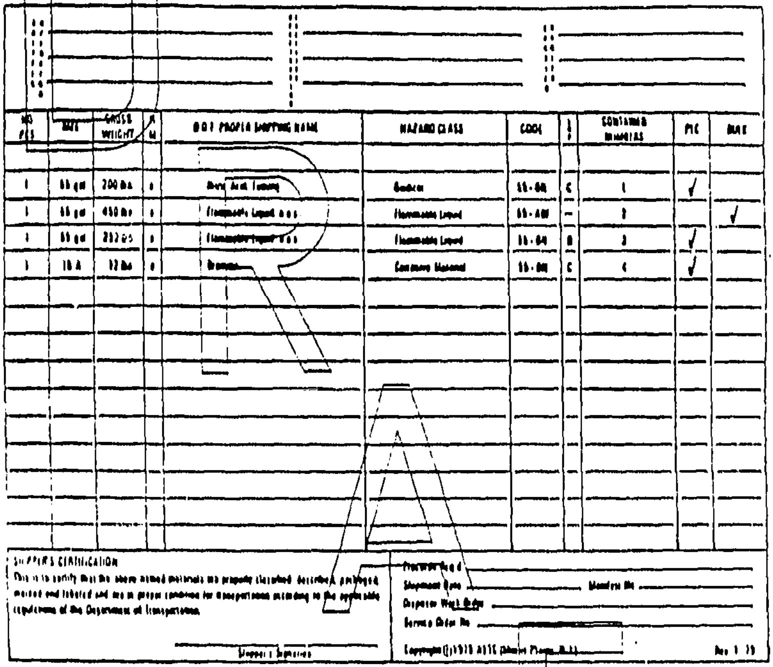
ATTACHMENT D

STANDARD INDUSTRY CERTIFICATION FORM



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		Applicability WMSG	
		Prepared Earth Sciences	
Subject SITE LOGBOOK		Approved A. K. Bomberger, P.E.	

TABLE OF CONTENTS

SECTION

1.0 PURPOSE

This procedure describes the process for keeping a site logbook.

2.0 SCOPE

The site logbook is a controlled document which records all major on-site activities during a Remedial Investigation/Feasibility Study. At a minimum, the following activities/events should be recorded in the site logbook:

- Arrival/departure of site visitors
- Arrival/departure of equipment
- Sample pickup (chain-of-custody form numbers, carrier, time)
- Sampling activities/sample logsheet numbers
- Start or completion of borehole/trench/monitoring well installation or sampling activities
- Health and Safety issues

The site logbook is initiated at the start of the first on-site activity (e.g., initial reconnaissance survey). Entries are made for every day that on-site activities take place which involve RI/FS contractor personnel. One current site logbook is maintained per site.

The site logbook becomes part of the permanent site file maintained in the RI contractor's office. Because information contained in the site logbook may be admitted as evidence in cost recovery or other legal proceedings, it is critical that this document be properly maintained.

3.0 DEFINITIONS

Site Logbook - The logbook is a bound notebook with consecutively numbered pages that cannot be removed. Upon entry of data, the logbook requires signature by the responsible site leader (see Section 5.1)

4.0 RESPONSIBILITIES

The site logbook is issued by the Regional Manager (or his designee) to the Site Manager for the duration of the project. The Site Manager releases the site logbook to the Field Operations Leader or other person responsible for the direction of on-site activities (e.g., Reconnaissance Survey Team Leader, Sampling Team Leader). It is the responsibility of this person (or his designee) to keep the site logbook current while in his possession, and return it to the Site Manager or turn it over to another field team. Following the completion of all fieldwork, the site logbook is returned to the Site Manager for inclusion in the permanent site files.

5.0 PROCEDURES

5.1 GENERAL

The cover of each site logbook contains the following information:

- Project Name and EPA Work Assignment Number
- NUS Project Number
- RI/FS Contractor and Site Manager's Name
- Sequential Book Number

- Start Date
- End Date

Daily entries into the logbook may contain a variety of information. At the beginning of each day the following information must be recorded:

- Date
- Start time
- Weather
- All field personnel present
- Any visitors present

During the day, a summary of all site activities and level of personal protection should be recorded in the logbook. The information need not duplicate that recorded in other field notebooks (e.g., sample logbook, Site Geologist's notebook, Health and Safety Officer's notebook, etc.), but should summarize the contents of these other notebooks and refer to the page locations in these notebooks for detailed information. An example of a site logbook page is shown in Attachment A.

The sample logsheet for each sample collected (see Procedure SA-6.6) must be referenced. If measurements are made at any location, the measurements and equipment used must either be recorded in the site logbook or reference must be made to the notebook and page number(s) on which they are recorded (see Attachment A).

All entries should be made in black pen. No erasures are permitted. If an incorrect entry is made, the data should be crossed out with a single strike mark, and initialed and dated. At the completion of entries by any individual, the logbook must be signed. It must also be signed by the Field Operations Leader or responsible site leader at the end of each day.

5.2 PHOTOGRAPHS

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

6.0 REFERENCES

Ebasco Services Incorporated; REM III Field Technical Guideline No. 13.03. October 30, 1987

7.0 RECORDS

Attachment A - Typical Site Logbook Entry

ATTACHMENT A
TYPICAL SITE LOGBOOK ENTRY

START TIME: ____________ DATE: ______________

SITE LEADER: ________________________
PERSONNEL:

NUS	DRILLER	EPA

WEATHER: Clear, 68°F, 2-5 mph wind from SE

ACTIVITIES:

1. Steam jenney and fire hoses were set up.

2. *Drilling activities at well ______ resumes. Rig geologist was ________________.* See Geologist's Notebook, No. 1, page 29-30, for details of drilling activity. Sample No. 123-21-S4 collected; see sample logbook, page 42. Drilling activities completed at 11:50 and a 4 inch stainless steel well installed. See Geologist's Notebook, No. 1, page 31, and well construction details for well ________.

3. Drilling rig No. 2 steam-cleaned at decontamination pit. Then set up at location of well __________.

4. Well _________ drilled. Rig geologist was __________________________. See Geologist's Notebook, No. 2, page ______ for details of drilling activities. Sample numbers 123-22-S1, 123-22-S2, and 123-22-S3 collected; see sample logbook, pages 43, 44, and 45.

5. Well ________ was developed. Seven 55-gallon drums were filled in the flushing stage. The well was then pumped using the pitcher pump for 1 hour. At the end of the hour, water pumped from well was "sand free."

6. EPA remedial project manger arrives on-site at 14:25 hours.

7. Large dump truck arrives at 14:45 and is steam-cleaned. Backhoe and dump truck set up over test pit ____________.

8. Test pit __________ dug with cuttings placed in dump truck. Rig geologist was ____________________. See Geologist's Notebook, No. 1, page 32, for details of test pit activities. Test pit subsequently filled. No samples taken for chemical analysis. Due to shallow groundwater table, filling in of test pit _____ resulted in a very soft and wet area. A *mound was developed and the area roped off*

9. Express carrier picked up samples (see Sample Logbook, pages 42 through 45) at 17:50 hours. Site activities terminated at 18:22 hours. All personnel offsite, gate locked.

____
Field Operations Leader

NUS CORPORATION

WASTE MANAGEMENT SERVICES GROUP

STANDARD OPERATING PROCEDURES

Number: SA-6.5
Page: 1 of 7
Effective Date: 01/01/88
Revision: 0
Applicability: WMSG
Prepared: Earth Sciences
Subject: FIELD REPORTS
Approved: A. K. Bomberger, P.E.

TABLE OF CONTENTS

SECTION

1.0 PURPOSE

This procedure describes the periodic field reports which are required to be filled out during the conduct of Remedial Investigation (RI) field studies. These reports on the progress of field assignments are not to be confused with the forms associated with boring and well installation, sampling, sample custody and equipment maintenance described in Procedure SA-6.4.

These reports serve several purposes:

- To maintain a written record of major events/accomplishments/problems related to the field work.
- To allow ongoing monitoring of the actual progress of field tasks in comparison to the planned schedule, and to allow timely corrective action (if required)
- To inform Site Managers of progress/accomplishments for inclusion in The Monthly Project Tracking System.

2.0 SCOPE

The reports described herein are to be used during field investigations, but do not replace or take precedence over project-specific or subcontractor-specific required reports. Additional reporting may particularly be required at enforcement-lead sites.

3.0 GLOSSARY

None

4.0 RESPONSIBILITIES

Field Operations Leader - responsible for assuring that the appropriate reports are completed in the required time-frame. Responsibilities for filling out individual reports are identified within the description of the reports (see below).

5.0 PROCEDURES

5.1 PROGRAM DESIGN

The primary means of recording onsite activities is the site logbook (see Procedure SA-6.3) and other field logbooks (e.g. geologists notebook, health and safety officer's logbook, sample logbooks). However, these logbooks and notebooks usually contain extremely detailed information which is required for data interpretation or documentation, but not for tracking and reporting of progress. Furthermore, the field logbooks remain onsite for extended periods of time and are thus not accessible for review by project management. The reports described in this guideline are, in essence, simplified summaries of the logbooks, which are designed to provide only the information needed by project management to keep informed of the progress of field activities.

5.2 DAILY ACTIVITIES REPORT

5.2.1 Description

The Daily Activities Report documents the activities and progress for each day's field work. This report is filled out on a daily basis whenever there are drilling, test pitting, well construction, or other related activities occurring which involve subcontractor personnel. These sheets summarize the work performed and form the basis of payment to subcontractors. (see Attachment A).

5.2.2 Responsibilities

It is the responsibility of the rig geologist to complete the report and obtain the driller's signature acknowledging that the times and quantities of material entered are correct.

5.2.3 Submittal and Approval

At the end of the shift, the rig geologist submits the Daily Activities Report to the Field Operations Leader (FOL) for review and filing. The Daily Activities Report is not a formal report and thus requires no further approval. The reports are retained by the FOL for use in preparing the site logbook and weekly Field Summaries, and are submitted to the Site Manager Weekly along with the Weekly Field Summary.

5.3 WEEKLY FIELD SUMMARY

5.3.1 Description

The Weekly Field Summary is an abstract of the Site Logbook, summarizing the major activities onsite for a particular week (Sunday through Saturday). It should be organized on a day-by-day basis, and contain the following information at a minimum (see Attachment B).

- Date (week ending)
- Personnel onsite (contractor, subcontractors, visitors)
- Weather conditions encountered during the week
- Site activities
- Number and type of samples collected (including C.O.C. form numbers)
- Issues impacting progress of the project.

5.3.2 Responsibilities

The Field Operations Leader or responsible individual onsite if not the FOL (e.g., geophysics team leader, sampling team leader) is responsible for completing the Weekly Field Summary at the end of each week of ongoing site activity, or at the completion of an activity (if no further activity will take place during that week)

5.3.3 Submittal and Approval

The summary, along with Daily Activities Reports, Health & Safety Officer's Reports, and any other documentation, must be delivered or sent to the Site Manager at the end of each week.

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ATTACHMENT A

DAILY ACTIVITIES RECORD - FIELD INVESTIGATION **NUS CORPORATION**

PROJECT NAME. ______________________ PROJECT NO ______
CLIENT ______________ LOCATION. ______________________
DATE ______________ ARRIVAL TIME. ________ DEPARTURE TIME. ________
CONTRACTOR. ______________ DRILLER: ______________________
BORING NO : ________ NUS REPRESENTATIVE: ______________________

ITEM (1)	ORIGINAL QUANTITY (2) ESTIMATE	QUANTITY (2) TODAY	PREVIOUS TOTAL (2) QUANTITY	CUMULATIVE QUANTITY (2) TO DATE
1. Mobilization/Demobilization	Job			
2. Overburden Drilling/Sampling, minimum 6-inch	100 ft.			
3. Overburden Drilling, 10-inch	250 ft.			
4. Overburden Drilling 14-inch	450 ft.			
5. Bedrock Drilling 6-inch	630 ft.			
6. Bedrock Drilling 10-inch	650 ft.			
7. Bedrock Drilling 14-inch	150 ft.			
8. Temporary 6-inch Steel Casing	250 ft.			
9. Temporary 10-inch Steel Casing	200 ft.			
10. Temporary 14-inch Steel Casing	250 ft.			
11. Permanent 6-inch Steel Casing	1,250 ft.			
12. Permanent 10-inch Steel Casing	400 ft.			
13. PVC Well Construction/Installation	1,120 ft.			
14. Mine Void Sealing	5			
15. Boring Backfilling	NA			
16. Well Development	24 hrs.			
17. Test Borings	200 ft.			
18. Test Pit Excavation	50 hrs.			
19. Standby	20 hrs.			

COMMENTS: ____

(1) AS LISTED IN SPECS
(2) INCLUDE QUANTITY AND UNITS
(Ex. 20 ft., 6 hrs.)

APPROVED BY

____
NUS FIELD REPRESENTATIVE

____
DRILLER OR REPRESENTATIVE

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ATTACHMENT B
PAGE 1 OF 2

WEEKLY FIELD SUMMARY REPORT

SUNDAY

Date :
Weather:
Personnel Onsite

Site Activities:

MONDAY

Date :
Weather:
Personnel Onsite

Site Activities:

TUESDAY

Date :
Weather:
Personnel Onsite

Site Activities:

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ATTACHMENT B
PAGE 2 OF 2

WEDNESDAY

Date :______________ Personnel ______________ ______________
Weather:______________ Onsite ______________ ______________
______________ ______________

Site Activities: ______________________________

THURSDAY

Date :______________ Personnel ______________ ______________
Weather:______________ Onsite ______________ ______________
______________ ______________

Site Activities: ______________________________

FRIDAY

Date :______________ Personnel ______________ ______________
Weather:______________ Onsite ______________ ______________
______________ ______________

Site Activities: ______________________________

SATURDAY

Date :______________ Personnel ______________ ______________
Weather:______________ Onsite ______________ ______________
______________ ______________

Site Activities: ______________________________

The Weekly Field Summary is an internal informational document and is not subject to project management review or approval.

6.0 REFERENCES

Ebasco Services Incorporated; REM III Field Technical Guideline No. 13.02. October 30, 1987.

Ebasco Services Incorporated; REM III Field Technical Guideline No. 2.06. June 2, 1986.

Ebasco Services Incorporated; REM III Field Technical Guideline No. 13.03. October 30, 1987.

Ebasco Services Incorporated; REM III Field Technical Guideline No. 13.01. October 29, 1987.

7.0 RECORDS

Attachment A - Rig Shift Report
Attachment B - Weekly Field Summary Report (2 pages)

NUS CORPORATION WASTE MANAGEMENT SERVICES GROUP	**STANDARD OPERATING PROCEDURES**	Number SA-6.6	Page 1 of 28
		Effective Date 01/01/88	Revision 0
		Applicability WMSG	
		Prepared Earth Sciences	
Subject	MANAGEMENT OF SAMPLING AND PREPARATION OF REQUIRED FORM	Approved A. K. Bomberger, P.E.	

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1.0 PURPOSE

The purpose of this procedure is to describe the method used in planning and managing sample shipments to the EPA Contract Laboratory Program (CLP).

2.0 SCOPE

This procedure applies to all NUS staff involved in preparation of the Field Sampling and Analysis Plan (FSAP), and personnel involved in RI field work, involving collection of samples for off-site chemical analysis.

3.0 GLOSSARY

Authorized Requestor (AR) - The EPA contact person(s) in the Regional Sample Control Center through which CLP's analytical services must be accessed.

Contract Laboratory Program (CLP) - A system of contractor-run laboratories providing analytical services and support for EPA's Superfund program. Data produced from this program is subject to rigorous QA/QC and documentation procedures to ensure its admissibility as evidence in any EPA enforcement proceedings.

Deputy Project Officer (DPO) - Appointed by the EPA Regional Administrator for each region, the EPA's DPO has partial responsibility for monitoring the laboratory contractors actually located in the region. Additional duties currently include resolution of problems in laboratory operations and laboratory site evaluations.

Environmental Monitoring and Support Laboratory/Las Vegas (EMSL/LV) and National Enforcement Investigations Center (NEIC) - Although all high-hazard samples were sent to this EPA Laboratory for preparation before entering the CLP, this is no longer done. High-hazard samples now enter CLP directly through Special Analytical Services (SAS). Current responsibilities of EMSL/LV and NEIC include methods development, QA, and automated data transfer.

Laboratory Services Manager (LSM) - The ARCS III PMO Manager responsible for all ARCS III laboratory analytical services, including ARCS III subcontractor laboratories and submission of samples to CLP.

National Enforcement Investigations Center (NEIC) - The EPA unit responsible for developing guidance and providing technical assistance to EPA enforcement efforts.

RAS Sample - A quantity of soil, water, sediment, vapor, or other media taken from the field at a single point at a single time and submitted for a set of Routine analytical Service (RAS) analyses. One sample collected and submitted for both organic and inorganic analysis would be counted as two RAS samples.

Regional Laboratory Services Coordinator (RLSC) - The REM III person responsible for coordination of ARCS III Laboratory analytical services in a single region. The RLSC is in general the single point of contact for the EPA Regional Sample Control Center (RSCC). The RLSC will usually be an employee of the lead firm for that region, but will coordinate laboratory services for all sites in the region, regardless of which ARCS III team member firm is performing site work.

Regional Sample Control Center (RSCC) - The EPA regional offices which serve as the central contact with the CLP for each region. The RSCC coordinates the level of regional sampling activities to

SECTION III CONTINUED

correspond with monthly allocations of CLP capacity, places all requests for CLP analyses, coordinates sampling and sample shipment, and resolves any problems that may arise concerning the samples

Repository Authorized Requestor (RAR) - The ARCS III personnel (one for each region) recognized by the Sample Management Office (SMO), through whom all requests for sample containers must be forwarded to the CLP Sample Bottle Repository. The RAR is usually the Regional Laboratory Services Coordinator (RLSC),

Routine Analytical Services (RAS) - Offered through CLP, for the determination of common organic and inorganic parameters and dioxin. The nature of these services is specified in contracts with each laboratory. For a detailed description of these services, see the "User's Guide to the Contract Laboratory Program" (Reference 1 of this guideline).

Sample Bottle Repository - A contractor-operated, centralized source for the most commonly-used sizes of pre-cleaned and QC-tested sampling containers for CLP samples.

Sample Management Office (SMO) - The contractor-operated office through which CLP receives analytical requests from the regions. Duties of SMO include sample scheduling and tracking, Special Analytical Services (SAS) subcontracting, laboratory invoice processing, maintenance of CLP records and management reporting, and NPO (National Program Office at EPA Headquarters) management and administrative support.

Special Analytical Services (SAS) - Analyses requiring special protocols or handling (e.g., high-hazard, non-routine parameters, enhanced detection limits) are available through this option. Individual contracts for these services are solicited, awarded and administered by SMO. For a description of these services, see Reference 1 of this guideline.

Traffic Report (TR) - Documentation used to track CLP samples from the field to the laboratory. Three separate versions exists for inorganic, organic and high hazard samples (see Attachment A). One traffic report is used per sample.

4.0 RESPONSIBILITIES

Site Manager (SM) - responsible for thorough understanding of the CLP (or non-CLP) requirements and incorporation of these requirements into the FSAP and project schedule. The SM retains overall responsibility for the success of the sampling and analysis and serves as the prime interface with EPA staff, although certain aspects of sampling (e.g., preparation for sampling and shipment, coordination with RSCC) may be delegated to other project personnel (e.g., Regional Laboratory Services Coordinator and Field Operations Leader).

With regard to sampling, the SM's specific responsibilities include:

- Preparation of EPA-approved FSAP (including QA/QC protocols and SAS analytical protocols) for CLP analysis;
- Coordination with RAR to order sample containers
- For non-CLP analysis, coordination with the PMO Laboratory Services Manager (LSM) through the Regional Laboratory Services Coordinator (RLSC) to identify the laboratory and analytical protocols, and management of the non-CLP Laboratory's subcontract.

- Obtaining required EPA and NUS document forms, site logbook and sample logbook;
- Assigning and preparing the sampling team.

Field Operations Leader (FOL) - responsible for thorough understanding of CLP (or non-CLP) requirements and retains overall responsibility for the correct collection, bottling, documentation, preservation, and shipment of samples to the analytical laboratories, including notification of RSCC and SMO of sample shipment. Some of these responsibilities may be delegated to a sampling technician.

Field Sampling Technicians - responsible for correctly collecting samples, filling out the required sample documentation, traffic reports and chain-of-custody forms and following the directions of the Field Sampling and Analysis Plan, relevant NUS Procedures, and the FOL regarding sample collection, preservation, and shipment methods.

EPA Remedial Project Manager (RPM) - The designated EPA representative for the work assignment, the RPM is responsible for EPA's activity in all phases of the assignment. With regard to sampling, the RPM is responsible for:

- Assisting with regard to site entry;
- Contacts with the state agencies and responsible parties in the local community;
- Providing sample documentation forms (e.g., chain-of-custody, seals, tags), and
- Approval of plans, subcontracts, and reports; data validation and data entry.

5.0 PROCEDURES

5.1 OVERVIEW

Sampling and analysis, as conducted in accordance with EPA and NUS procedures and requirements, are extremely complex operations. From 6 to 12 agencies, organizations, or offices are involved in the overall program, and each has its own procedures and requirements. There are at least eight separate and distinct administrative and management activities needed to establish a sampling and analysis program These are:

1 Planning
2 Logistics
3 Subcontracting
4 Site activities, including sampling, drilling, surveying, test pit excavation, boring etc
5 Packaging and shipping, including documentation
6 Analysis
7 Data Validation
8 Reporting

Activities 1 and 2 should be covered in detail in the FSAP and are the responsibility of the SM, FOL, and other staff assigned to the project. Activity 3 should be initiated by the SM, through the NUS Contracting Officer at PMO, during the RI/FS Initial Tasks and Activities

Activities 4 and 5 are field activities to be conducted by the NUS field personnel Activity 6 may be conducted by CLP (not including field analysis, which will be conducted by field personnel), and Activity 7 may be conducted by various branches of EPA (see Attachment J) Finally Activity 8 is the responsibility of the NUS contractor for that work assignment

Frequent communications with the offices and organizations involved are necessary to maintain effective coordination. Throughout the entire operation, quality assurance and quality control requirements must be satisfied in accordance with the Quality Assurance Program Plan. Extensive documentation is needed to assure adequate management tracking of samples through the complex system and to maintain a chain-of-custody record for litigation purposes. Attachment A presents a summary of the timing of activities for scheduling CLP samples.

5.1.1 Sampling Equipment

Proper and sufficient sampling equipment is a basic necessity for a successful sampling effort. Attachment I contains an equipment checklist which should be used by the SM or FOL when preparing for a sampling program. To avoid delays in the sampling programs, it is in the interest of the SM to provide this equipment request with sufficient advance notice (usually 2 to 3 weeks minimum).

Additionally, if Special Analytical Services (SAS) are requested, the FSAP must include specific methods and protocols required for these analyses. These protocols must be approved by EPA before requesting SAS from SMO.

5.2 PLANNING FOR SAMPLING ACTIVITIES

Planning for work assignments involving the collection of samples to be submitted for CLP analyses consists of several major steps:

- Develop Field Operations Plan;
- Schedule CLP Analysis;
- Obtain CLP Sample Bottles;
- Obtain Sample Shipping Coolers.

The NUS Site Manager (SM) must communicate regularly with the EPA Remedial Project Manager (RPM) to ensure that site planning activities progress in a smooth and timely fashion, through each of the major steps listed above. In addition, the Regional Laboratory Services Coordinator (RLSC) must communicate regularly with the EPA Regional Sample Control Center (RSCC) to ensure smooth approval and coordination of the sampling effort. Responsibilities in each step are discussed in turn.

5.2.1 Field Sampling and Analysis Plan

The Field Sampling Analysis Plan (FSAP) is the major document outlining all planned sampling activities for a RI/FS, including elements of site-specific quality assurance. For non-RI/FS work assignments which nevertheless involve field work and analysis of samples (e.g., PA/SI, oversight, confirmational sampling for enforcement cases), an equivalent task specific FSAP will be developed.

The FSAP must be approved by EPA before CLP sample scheduling procedures can be initiated. The FSAP must therefore be prepared by the NUS Site Manager (SM) and submitted to the EPA Remedial Project Manager (RPM) at least one month prior to the date that samples will be submitted for CLP analysis. Additionally, if Special Analytical Services (SAS) and analysis are requested, the FSAP must include specific methods and protocols required for these analyses. Requirements for SAS should be defined in consultation with chemists, engineers and risk assessment personnel during the development as Data Quality Objectives (DQO's) for inclusion in the Work Plan for the site.

Requirements for notifying EPA of sampling requirements and gaining approval of the FSAP vary among EPA Regions. The requirements of EPA Region III are outlined below.

- Region III: Region III requires that the SM prepare and submit duplicate copies of the FSAP one month in advance of planned sampling for CLP analysis. One copy must be sent to the ROM and another copy sent to the RSCC at the Central Regional Laboratory in Annapolis, MD. Following RPM and RSCC review and approval of the FSAP, the SM is authorized to schedule samples for CLP analysis through the RSCC. Because of some past difficulties in obtaining CLP capacity for high concentration (drum) samples, Region III requires 3-4 weeks notice in order to schedule these types of samples for CLP analysis.

5.2.2 General Steps in Scheduling CLP Analyses

Following EPA approval of the Field Sampling and Analysis Plan, the SM or Field Operations Leader (FOL) is authorized to contact the Regional Laboratory Services Coordinator (RLSC), who in turn contacts the EPA Regional Sample Control Center (RSCC) to schedule samples for CLP analysis. The precise information required by the RSCC in order to schedule sample analyses is discussed in Section 5.2 and on pages 52-62 of the CLP User's Guide (Reference 1 of this Guideline). However, the general steps in initiating this process include:

- SM (or FOL) contacts RLSC to submit request to RSCC for specific CLP analytical support at least one week prior to sampling dates. The SM should also request CLP and evidence documentation forms from the RSCC, e.g., Traffic Reports, Chain-of-Custody forms, evidence seals, and sample tags.

- RSCC requests Sample Management Office (SMO) to schedule samples for CLP analysis, informing SMO of the sampling requirements identified by the RLSC.

- Initiation of SMO sample scheduling activities with CLP laboratories

- SMO calls RSCC confirming sample scheduling with CLP laboratories.

- RSCC calls RLSC with information on sample scheduling.

When there is sufficient CLP capacity, this scheduling process usually takes one to three days to complete. If there is a shortage of CLP capacity, sample scheduling can take up to one week. Where CLP capacity is limited, the RSCC may allocate available capacity to another project with a higher regional priority.

One CLP laboratory assignments have been made, it is important to notify the RSCC immediately of any changes in the sampling plan or schedule. If postponements or cancellations in sampling activities are necessary, this too must be communicated to the RSCC or RPM. When possible, the RLSC should have a replacement sampling activity to substitute for the postponed or canceled activity to ensure that the assigned CLP capacity is used.

5.2.3 Obtaining CLP Sample Bottles

The CLP Sample Bottle Repository program may be used by any organization scheduling samples through the CLP, and is commonly accessed by regional and NUS contractor clients. One staff member from each region, usually the RLSC, is designated by SMO as a Repository Authorized Requestor (RAR) and only these individuals may place bottle orders through the program using a Sample Bottle Repository Delivery Order (Attachment C). Once designated, the RAR orders bottles directly from the Repository. Because the Repository can respond only to orders submitted by an SMO-designated RAR, NUS regional staff must contact SMO to request any changes in RAR designees.

CLP clients may obtain nine types of bottles for use in sampling activities. The Sample Bottle Repository Program provides bottles in numbered lots, packing in protective cardboard containers, that are pre-cleaned and QC-tested to ensure no contamination exists that may affect sample data results. The identification number of the bottle lot used for each sample should be written on the Traffic Report or other sample document form (e.g., Dioxin Shipment Record, Packing List - see Section 5.3).

There are three types of bottle orders; Routine (ten or more working days lead time for delivery), Fast-turnaround (more than three days, but less than ten days lead time for delivery). Should it be necessary to cancel an order, contact the Repository either directly or through the RAR, by telephone. Follow up with a cancellation memo to the repository (see CLP User's Guide, Reference 1 of this guideline) with a copy to the work assignment file.

Some common problems which have been experienced with the bottle repository program include:

- Bottles shipped directly to sampling locations occasionally arrive at local hotels or agencies before sampling crews. Consequently, these bottles are sometimes stored improperly, broken or lost. Shipping the bottles to the nearest Federal Express or other carrier's office, marked "For Pick-Up" will avoid these problems. Alternatively, the bottles may be shipped directly to the contractor's office, laboratory, or other field sampling staging location

- Bottle types are prepared specifically for the type of analyses specified in the CLP User's Guide (Reference 1 of this guideline). Use the correct bottle for the parameter of interest.

5.2.4 Obtaining Sample Shipping Coolers

The CLP <u>does not</u> provide sample shipping coolers. It is therefore the responsibility of the SM to obtain the required number of coolers through his/her firm prior to sampling activities

All shipping coolers should have clearly visible return address labels on the outside Shipping coolers that are labeled in this manner will be returned to the sampler by the CLP laboratory usually within 14 days following laboratory sample receipt. NUS staff should be sure that the return address label is distinct from, and not obscured by, other shipping labels.

5.3 CLP SAMPLE SCHEDULING AND COLLECTION

The two keys to using the CLP successfully are first, rapid and effective communication among the sampler, RSCC, and SMO, particularly when changes in the sampling plan are necessary, and, second, accurate completion and routing of all required documentation. The appropriate steps in sample scheduling are collection under the CLP's RAS and SAS programs are summarized below For more complete information on these activities, consult the CLP User's Guide (Reference 1 of this guideline)

5.3.1 Routine Analytical Services (RAS)

To initiate a RAS request, a SM must request the RLSC to contact the RSCC who will in turn contact the SMO by telephone with a description of the analytical requirements. It is the responsibility of the SM to maintain a working knowledge of RAS protocols and analytical services The analytical protocols described in the CLP User's Guide (Reference 1) contain specific information on sample types suited to RAS analysis, target analytes, detection limits, and other information

The RSCC will require the following information from the SM:

- Name(s), firm name, and telephone number(s) of sampling personnel.
- Name and location of the site to be sampled.
- Number of samples and matrix of each sample to be collected.
- Type of analyses required for each sample; i.e., inorganic, organic, dioxin.
- Cyanide analysis requirement (inorganics only).
- Scheduled sample collection and shipment dates.
- Nature of sampling event (i.e., investigation, monitoring, enforcement, remedial construction).
- Other pertinent information which may affect sample scheduling or shipment (i.e., potential delays due to site access, weather conditions, or drilling or sampling equipment difficulties).

Once the RAS laboratory arrangements have been made, the SMO will confirm the field investigation plans with the RSCC and identify the laboratories to which the samples will be sent. The RSCC will, in turn, pass this information back to the RLSC.

For a more detailed description of how to request RAS, see pp. 52-54 of the CLP User's Guide (Reference 1).

5.3.2 Special Analytical Services (SAS)

Analytical services other than those specified in the RAS analytical protocols may be obtained by requesting Special Analytical Services (SAS). Examples of SAS needs include quick turnaround, multiphase, or non-RAS protocol analyses. Although the RSCC will assist in identifying appropriate SAS protocols, it is the responsibility of the SM and project chemist to select and provide to the laboratory the applicable analytical protocols to be used. These protocols must also be included in the Field Sampling and Analysis Plan (FSAP) for review by ESD before SAS can be requested. The lead time requirement for requesting SAS samples may be lengthened on the basis of the availability and familiarity of these protocols. In addition to the information required for RAS, the RSCC will require the following information from the RLSC for SAS:

- Specific analyses required, appropriate analytical protocols and required detection limits
- Matrix spike and duplicate frequency.
- Justification of fast turnaround request, if applicable.
- RI/FS contractor contact person for immediate problem resolution, usually the RLSC or the lead environmental chemist assigned to the project.

Once the RSCC requests SAS by telephone, SMO will initiate SAS subcontracting procedures and assign a sequential SAS number for each sampling activity. If the request is made concurrently with a RAS request, SMO will also issue a Case Number. The RSCC will record both of these numbers (if applicable) and use them to reference the samples. The RLSC must complete a SAS Client Request Form (see Attachment D) and submit it to the RSCC prior to sample scheduling for clarification and confirmation purposes

Following is a brief summary of SAS procedures. SMs may request assistance from the RLSC or the LSM in choosing appropriate SAS protocols. For a more detailed description of how to request SAS see pp. 55-59, of the CLP User's Guide (Reference 1)

5.3.3 High Hazard Analyses

The steps in scheduling analytical services for high hazard samples are similar to those described above for SAS. High concentration samples require SAS, not RAS analysis. For a description of this option, see pp 59-61 of the CLP User's Guide (Reference 1).

> Note: Samples no longer travel to EPA EMSL/LV or NEIC for preparation; high hazard samples are now being handled directly by CLP laboratories.

5.3.4 Weekend Shipments

Occasionally, it will be necessary to ship samples on Friday afternoon or evening. If this is the case, the sampler must notify the RSCC and SMO at the latest by 3:00 p.m. eastern standard time, Friday

5.3.5 Changes in Sampling Plans

Sometimes, due to unforeseen circumstances, it will be necessary to change the sampling plan. This may entail changes in the number of samples, sample matrix, shipment date, or other items. The sampler must notify the RSCC of any changes. Do not ship any samples that differ from those described in the sampling plan without authorization of the RSCC.

5.3.6 Sample Collection, Preservation, and Holding Times

Detailed guidance on approved sampling procedures may be obtained by consulting other Standard Operating Procedures.

Samples requiring preservatives should be identified and the necessary techniques to maintain sample quality should be described in the FSAP. Common preservation techniques may include the addition of acids or other materials to the sample container, or refrigeration of the sample. Refrigerated samples require special packaging (see Section 5.5.1 below).

Regardless of the method of preservation used (if any), strict adherence to holding times is necessary. Holding times represent the maximum amount of time that a preserved sample may be held from the time of sampling until extraction or analysis without compromising the validity of the analytical results. Maximum holding times at the laboratories are specified in the CLP laboratories scopes-of-work. The difference between those times and the total maximum holding times is the time allowed for shipment to the laboratory. If the laboratory receives a sample with less than the allowable laboratory holding time remaining, the laboratory is not contractually liable for analysis within the holding time (although the laboratories will try to meet the maximum holding times). In general, the following shipping frequencies should be followed:

- Samples requiring organics analysis should be shipped the same day collected, or on the following day.
- Samples for inorganic analysis may be held until the shipping container is full. Three days is the maximum recommended period for holding of inorganic samples prior to shipping.

Different EPA regions, however, may have different requirements as to holding times for samples in the field. For a detailed description of holding times, packaging, and transportation see the CLP User's Guide (Reference 1), pp 71-73 and Appendix C of the User' Guide.

5.4 DOCUMENTATION FOR CLP AND EPA CHAIN-OF-CUSTODY

Requests for analytical services through the CLP must be documented properly Documentation serves to ensure timely, correct and complete analysis for all requested parameters, provides support data for use in potential enforcement actions, and provides a means by which results may be validated. The CLP User's Guide (Reference 1) provides descriptions of various sample documentation forms and their applicability to CLP analytical requests.

5.4.1 Traffic Reports

All RAS samples must be accompanied by a Traffic Report (TR). TRs are uniquely numbered and come in three varieties: Organic, inorganic, and High-Hazard. Following are general guidelines for TRs.

- Use one TR for each sample. A sample is a collection of material from a single point at one time and submitted for a single type of analysis (e.g., inorganic or organic) The use of multiple containers does not necessarily mean multiple samples: for example, an organic CL sample may be submitted in three containers for volatile, semi-volatile and pesticide analyses.

- Several spare TR forms should be brought to the field to replace damaged or improperly completed forms prior to sample shipment.

- The sampler should complete the following information: Case Number, site name or code, location, analytical laboratory to which the sample is shipped, firm name and sampler's name, dates of samples collection and shipment, number of sample bottles used, sample concentration (e.g., high, medium or low) and matrix, and sample bottle repository lot number.

- Samples for SAS only, (i.e., those for which no RAS is required) will be tracked using a SAS packing List (see Attachment E-4). No TR is to be completed for these samples

- For samples requiring both RAS and SAS, a TR is used with both the Case number and SAS number entered

- Samples requiring RAS dioxin analysis only, will be tracked using a Dioxin Shipment Record (see Attachment E-5), not a TR.

- Two copies of the TR go to the laboratory, one to SMO, one to the sampler's files

Examples of sample TRs are included in Attachment E of this guideline. For a detailed description of these forms and instructions on their usage, see pp. 63-64 of the CLP User's Guide (Reference 1)

5.4.2 Dioxin Shipment Record

Samples destined for the RAS dioxin program must be accompanied by the CLP Dioxin Shipment Record (DSR) These will be used in lieu of the TR for dioxin samples only. A sample form is included as Attachment E-5 of this guideline. For a description of this form, see p 65 of the CLP User's Guide (Reference 1)

5.4.3 SAS Packing List

Samples that require SAS only are to be accompanied by a SAS Packing List (PL) instead of a TR. Do not use this form for RAS-plus-SAS samples. A sample form is included as Attachment E-4 of this guideline. See p. 66 of the CLP User's Guide (Reference 1) for a description of this form

5.4.4 Sample Identification Tags

Sample identification tags are required for all samples. Check off the desired analytical parameters directly on the tag and attach it securely to the sample container. The tags will be retained by the laboratory as physical evidence that the sample was received, and may be used by EPA in litigation

Care should be taken in filling out the sample tag. Improperly completed tags require time-consuming telephone inquiries to verify the actual parameters intended.

These tags may not accurately reflect the most recent CLP protocols. Mercury, for example, is considered part of the CLP metals analysis package, but is a separate parameter on these tags. In requesting metals analysis, be sure to check mercury along with metals.

5.4.5 Chain-of-Custody

In order for analytical results to be introduced as evidence in court, the custody of samples must be maintained and documented at all times. Chain-of-custody begins with the taking of the samples in the field. A detailed description of this requirement may be found on pp 69-70 in the CLP User's Guide (Reference 1)

It is strongly recommended that a second person or persons be used to verify the accuracy and correctness of the chain-of-custody and all other documentation. The second person should cross-check the chain-of-custody form with packing lists, TRs, sample tags, and logbooks This cross-check should be done prior to shipment.

5.5 NUS Program Sample Documentation

In addition to the required EPA QA, and CLP or non-CLP laboratory documentation of samples, certain standard forms are required for NUS program sample description and documentation. These include the well sampling data sheet (for water samples taken from monitoring wells) and the sample logbook, which contains sample log sheets for all samples collected.

5.5.1 Well Sampling Data Sheet

A well sampling data sheet should be filled out whenever samples are collected from a monitoring well This form records information about the well evacuation and other parameters (see Attachment F) which may be necessary for sample validation or interpretation The well sampling data sheet should be retained in the sample logbook (see Section 5.5.2), attached to the sample logsheet(s) for that well sampling event.

5.5.2 Sample Logbook

The sample logbook is a 3-ring binder which contains sample log sheets for each sample collected and also well sampling data sheets A sample log sheet (Attachment F) is filled out for each and every sample collected This form records vital information concerning the sample source, sampling methods, sample conditions, and field measurements, and is used for sample [illegible] and report preparation. The sample log sheets are numbered in order when placed in the sample logbook, and the sample number and log sheet page numbers are recorded on the sample logbook table of contents sheet (which is placed at the front of the sample logbook) for easy reference and access.

5.6 SAMPLE SHIPMENT AND NOTIFICATION

5.6.1 Sample Packaging

Samples must be properly prepared for shipment to the recipient laboratory. This preparation includes packaging and labeling sample coolers to comply with current U.S. DOT and commercial carrier regulations. The CLP User's Guide (Reference 1) should be consulted for specific guidance in this area. Specific points to note include:

- Dioxin samples should be shipped as Poison B, rather than flammable liquid or solid
- The use of bubble wrap sample bottles, after they have been placed in plastic bags, has proven very successful in reducing breakage. The material may be purchased from GSA, local office suppliers or direct from the manufacturer (e.g., Sealed Air Corporation). Under no circumstances should earth or ice be used to cushion samples. Vermiculite or similar material should be used.

Ice or "blue ice" refrigerant packages may be packed in contact with the sample bottle, and the entire package (bottle and ice) overpacked with plastic bands and bubble wrap.

5.6.2 Use of Common Carriers

Where possible, the use of reputable, overnight couriers, such as Federal Express, DHL, Purolater, and Emery, is strongly encouraged.

5.6.3 Shipment Notification

Immediately after shipping, the sampler must notify the RLSC who will inform the RSCC that samples have been collected and shipped. Under certain circumstances, the FOL or SM can contact the RSCC directly to inform of sample shipment or problems. The sampler should be prepared to provide the following information:

- Sampler Name.
- Case Number and/or SAS Number of the project
- Batch numbers (dioxin only).
- Exact number(s) and matrices of sample(s) shipped
- Carrier and airbill number(s) for the shipment.
- Method of shipment.
- Any irregularities or anticipated problems with the samples, including special handling instructions, or deviations from established sampling procedures
- Status of the sampling project (e.g., final shipment, update of future shipping schedule)

- SMO must be notified by 3:00 p.m. eastern standard time Friday, for samples due to arrive on Saturdays. Failure to do so may result in the laboratory not having anyone on hand to accept the samples. In notifying SMO of weekend or any other deliveries, the airbill number is critical.

- Do not write the site name on the airbill. Use the CLP case number or the NUS charge number to maintain confidentiality at the laboratory.

5.7 POST-SAMPLING ACTIVITIES

Following sample collection and shipment activities, and upon return to the NUS office, the SM or designated staff member must meet the specific information requirements of Region III.

5.7.1 Region III Information Requirements

The SM or FOL must complete the Project Sample Summary Form for all samples sent through the CLP (Attachment G) and submit it to the RSCC during the week following sample collection.

5.7.2 Receipt of Data from CLP Laboratories

CLP laboratories are required to analyze RAS samples and report the data within either 30 or 40 days (depending on the specific contract). Often the analysis takes longer, depending on the total CLP sample load and other factors. CLP laboratories are required to send the analytical data directly to the region in which the samples were collected. All data must be reviewed and validated by the region or designated validation contractor before release to the SM for use, and this data review process can often take a month to complete. The EPA data review and validation process is shown in Attachment H. As the attachment indicates, at least two months pass between the time samples were collected to the time the SM receives data that is authorized for use.

6.0 REFERENCES

USEPA, 1984. User's Guide to the Contract Laboratory Program. Office of Emergency and Remedial Response, EPA, Washington, D.C. (revision planned for publication in late 1986).

U.S. Environmental Protection Agency, 1983. Method for Chemical Analysis of Water and Wastes. EPA-600/4-79-020. Environmental Monitoring and Support Laboratory, Cincinnati, Ohio

U.S. Environmental Protection Agency, 1982. Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater. EPA-600/4-82-029. Environmental Monitoring and Support Laboratory, Cincinnati, Ohio.

U.S. Environmental Protection Agency, 1982. Handbook for Sampling and Sample Preservation of Water and Wastewater. EPA-600/4-82-029. Environmental Monitoring and Support Laboratory, Cincinnati, Ohio.

U.S. Environmental Protection Agency, 1980. Samplers and Sampling Procedures for Hazardous Waste Streams. EPA-600-2/80-018. Municipal Environmental Research Laboratory, Cincinnati, Ohio

U.S. Environmental Protection Agency, 1980. QAMS 005. Interim Guidelines and Specification for Preparing Quality Assurance Project Plans

---

U.S. Environmental Protection Agency, 1984. Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air. EPA 600/4-84-041.

U.S. Environmental Protection Agency, 1983. SOP for the GC/MS Determination of Volatile Organic Compounds Collected on Tenax. EMSL/RTP-SOP-EMD-020. Environmental Monitoring and Support Laboratory, Research Triangle Park, North Carolina.

U. S. Environmental Protection Agency, 1981. Determination of Polychlorinated Biphenyls in Transformer Fluid and Waste Oils. EPA 600/4-81-045.

U.S. Environmental Protection Agency, 1985. Groundwater Technical Enforcement Guidance Document. Draft - August 1985.

EBASCO Services Incorporated; REM III Field Technical Guideline No. FT-7.04, May 5, 1986.

7.0 ATTACHMENTS

Attachment A	-	CLP Sample Scheduling Timeline
Attachment B	-	*Three-Month Sample Projection Plan*
Attachment C	-	Sample Bottle Repository Delivery Order
Attachment D	-	SAS Client Request Form
Attachment E-1	-	Inorganic Traffic Report
Attachment E-2	-	Organic Traffic Report
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Attachment E-4	-	SAS Packing List
Attachment E-5	-	Dioxin Shipment Record
Attachment F	-	Well Sample Data Sheet
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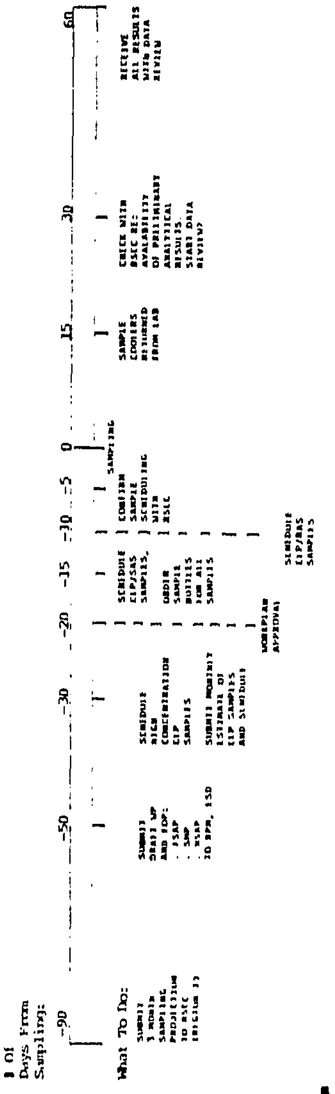
ATTACHMENT A

CLP SAMPLE SCHEDULING TIMELINE



ATTACHMENT B

PLANNED SAMPLING ACTIVITY REQUIRING CLP ANALYSES

Region ______ Site ______ Month/Year of Activity ______ Case ______

Sampling Organization (State, RLM, FIT, etc.) ______ Regional Contact ______ Telephone ______

Type of Investigation (RI/FS, Enforcement, etc.) ______ Activity Status (Definite, Conditional or Tentative) ______

Sampling Date(s) ______ Alternate Sampling Date(s) ______

Sample Shipment Date(s) ______ Alternate Shipment Date(s) ______

Routine Analytical Services Required	Media/Concentration									Total		
	Liquid or Water			Solid, Soil or Sediment			Other (SAS)					
	Low	Med	High	Low	Med	High	Low	Med	High	Low	Med	High
Full HSL Organics												
VOA Fraction Only												
Semi-VOA Fraction Only												
Pesticide/PCB Fraction Only												
Dioxin Only												
HSL Metals & Cyanide												
HSL Metals Only												

Special Analytical Services Required: Specify (Method), QA, Reporting Requirements to be Provided via SMO Client Request Form)

Submittal Status: (Current, 1, 2 or 3 Month Projection)	Notification Status: (Initial Notification or Resubmittal)	Resubmittal Status: (Resubmittal With or Without Changes)

Submitted By: ______ Date: ______ Phone: ______

Approved By: ______ Date: ______

Date Notification Received By SMO: ______

Date Laboratory(ies) Assigned: ______

ATTACHMENT C

CLP SAMPLE BOTTLE REPOSITORY
SUPERFUND DELIVERY REQUEST

Date of Request: ______________

Type of Request:
Routine ☐
Fast Turnaround ☐ ______________
Emergency ☐ ______________
(date/time request called in)

FROM (Name): ______________
Affiliation: ______________
Telephone: ______________
AR Signature: ______________

TO:
I-Chem Research Corporation
23787-F Eichler Street
Hayward, CA 94545
Phone: 415/782-3905

Ship the following items for arrival by: ______________(date)
(If applicable) Ship to arrive no earlier than: ______________(date)

Item		Description	No. of Items Per Case	No. of Cases Requested
A	80-oz	amber glass bottle	6	______
B	40-mL	glass vial	72	______
C	1-L	polyethylene bottle	12	______
D	120-mL	wide-mouth glass vial	12	______
E	16-oz	wide-mouth glass jar	12	______
F	8-oz	wide-mouth glass jar	12	______
G	4-oz	wide-mouth glass jar	12	______
H	1-L	amber glass bottle	12	______
J	32-oz	wide-mouth glass jar	12	______
K	4-L	amber glass bottle	4	______
L	500-mL	polyethylene bottle	24	______

Ship To: ______________
(provide ______________
street ______________
address) ______________
Attention. ______________

Call before Delivery ______________
(Phone No.) ______________

DISTRIBUTION: *White—Repository* *Yellow—Requestor* *Pink—SMO*

ATTACHMENT D - page 1

U.S. ENVIRONMENTAL PROTECTION AGENCY
CLP Sample Management Office
P.O. Box 818 - Alexandria, Virginia 22313
Phone: 703/557-2490 - FTS/557-2490

SAS Number

SPECIAL ANALYTICAL SERVICES
Client Request

☐ Regional Transmittal ☐ Telephone Request

A. EPA Region/Client: ____________________

B. Representative: ____________________

C. Telephone Number: ____________________

D. Date of Request: ____________________

Please provide below description of your request for Special Analytical Services under the Contract Laboratory Program. In order to most efficiently obtain laboratory capability for your request, please address the following considerations, if applicable. Incomplete or erroneous information may result in a delay in the processing of your request. Please continue response on additional sheets, or attach supplementary information as needed.

1. General description of analytical service requested: ____________________

2. Definition and number of work units involved (specify whether whole samples or fractions; whether organics or inorganics; whether aqueous or soil and sediments; and whether low, medium or high concentration): ____________________

3. Purpose of analysis (specify whether enforcement, remedial action, etc.): ____________________

4. Estimated date(s) of collection: ____________________

5. Estimated date(s) and method of shipment: ____________________

ATTACHMENT D - page 2

6. Approximate number of days results required after lab receipt of samples:

7. Analytical protocol required (attach copy if other than a protocol currently used in this program):

8. Special technical instructions (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):

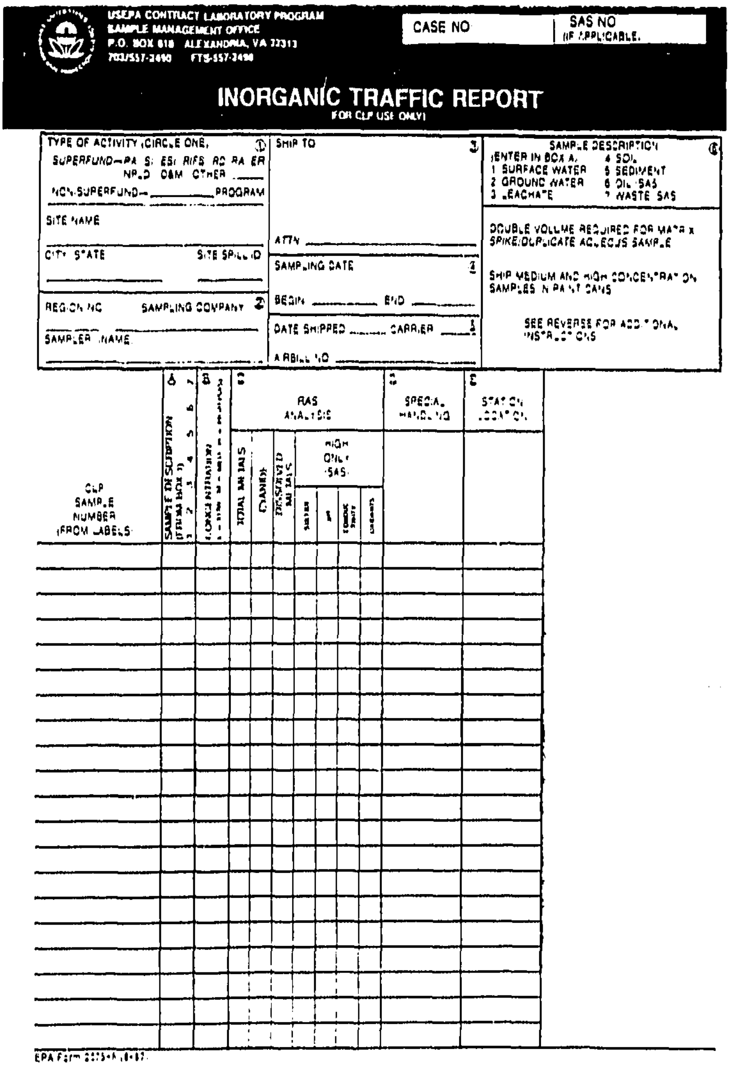
9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain of Custody documentation, etc.) If not completed, format of results will be left to program discretion.

10. Other (use additional sheets or attach supplementary information, as needed):

11. Name of sampling/shipping contact:
    Phone:

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please call us at the Sample Management Office.

ATTACHMENT E-1



USEPA CONTRACT LABORATORY PROGRAM
SAMPLE MANAGEMENT OFFICE
P.O. BOX 818 ALEXANDRIA, VA 22313
703/557-2490 FTS-557-2490

CASE NO

SAS NO (IF APPLICABLE)

INORGANIC TRAFFIC REPORT

(FOR CLP USE ONLY)

① TYPE OF ACTIVITY (CIRCLE ONE)
SUPERFUND—PA SI ESI RIFS RD RA ER NPLD O&M OTHER ____
NON-SUPERFUND—________ PROGRAM

SITE NAME

CITY, STATE ____ SITE SPILL ID ____

② REGION NO ____ SAMPLING COMPANY ____

SAMPLER (NAME) ____

③ SHIP TO

ATTN ____

④ SAMPLING DATE
BEGIN ____ END ____

⑤ DATE SHIPPED ____ CARRIER ____

AIRBILL NO ____

⑥ SAMPLE DESCRIPTION
(ENTER IN BOX A)
1 SURFACE WATER
2 GROUND WATER
3 LEACHATE
4 SOIL
5 SEDIMENT
6 OIL (SAS)
7 WASTE (SAS)

DOUBLE VOLUME REQUIRED FOR MATRIX SPIKE/DUPLICATE AQUEOUS SAMPLE

SHIP MEDIUM AND HIGH CONCENTRATION SAMPLES IN PAINT CANS

SEE REVERSE FOR ADDITIONAL INSTRUCTIONS

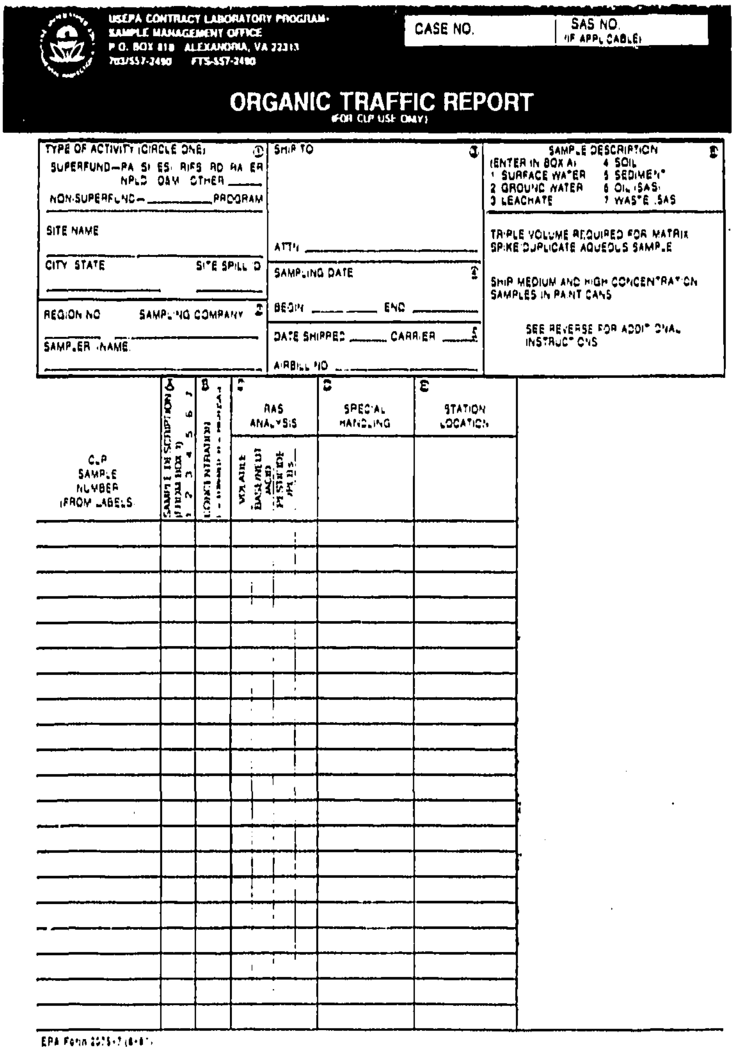
CLP SAMPLE NUMBER (FROM LABELS)	Ⓐ SAMPLE DESCRIPTION (FROM BOX 1) 1 2 3 4 5 6 7	Ⓑ CONCENTRATION	Ⓒ RAS ANALYSIS							Ⓓ SPECIAL HANDLING	Ⓔ STATION LOCATION
			TOTAL METALS	CYANIDE	DISSOLVED METALS	HIGH ONLY (SAS)					
						[illegible]	[illegible]	[illegible]	[illegible]		

EPA Form 2075-? (8-87)

WHITE — SMO COPY PINK — CLIENT COPY WHITE — LAB COPY FOR RETURN TO SMO YELLOW — LAB COPY

Subject	Number	Page
MANAGEMENT OF SAMPLING AND PREPARATION OF REQUIRED FORMS	SA-6.6	22 of 28
	Revision	Effective Date 01/01/88

ATTACHMENT E-2



USEPA CONTRACT LABORATORY PROGRAM
SAMPLE MANAGEMENT OFFICE
P.O. BOX 818 ALEXANDRIA, VA 22313
703/557-2490 FTS-557-2490

CASE NO.

SAS NO.
(IF APPLICABLE)

ORGANIC TRAFFIC REPORT

(FOR CLP USE ONLY)

TYPE OF ACTIVITY (CIRCLE ONE) ①
SUPERFUND—PA SI ESI RIFS RD RA ER
NPLD O&M OTHER ____
NON-SUPERFUND—__________ PROGRAM

SITE NAME

CITY STATE ____ SITE SPILL ID ____

REGION NO ____ SAMPLING COMPANY ____ ②

SAMPLER (NAME) ____

SHIP TO ③

ATTN ____

SAMPLING DATE ④

BEGIN ____ END ____

DATE SHIPPED ____ CARRIER ____ ⑤

AIRBILL NO ____

SAMPLE DESCRIPTION ⑥
(ENTER IN BOX A)
1 SURFACE WATER
2 GROUND WATER
3 LEACHATE
4 SOIL
5 SEDIMENT
6 OIL (SAS)
7 WASTE (SAS)

TRIPLE VOLUME REQUIRED FOR MATRIX SPIKE/DUPLICATE AQUEOUS SAMPLE

SHIP MEDIUM AND HIGH CONCENTRATION SAMPLES IN PAINT CANS

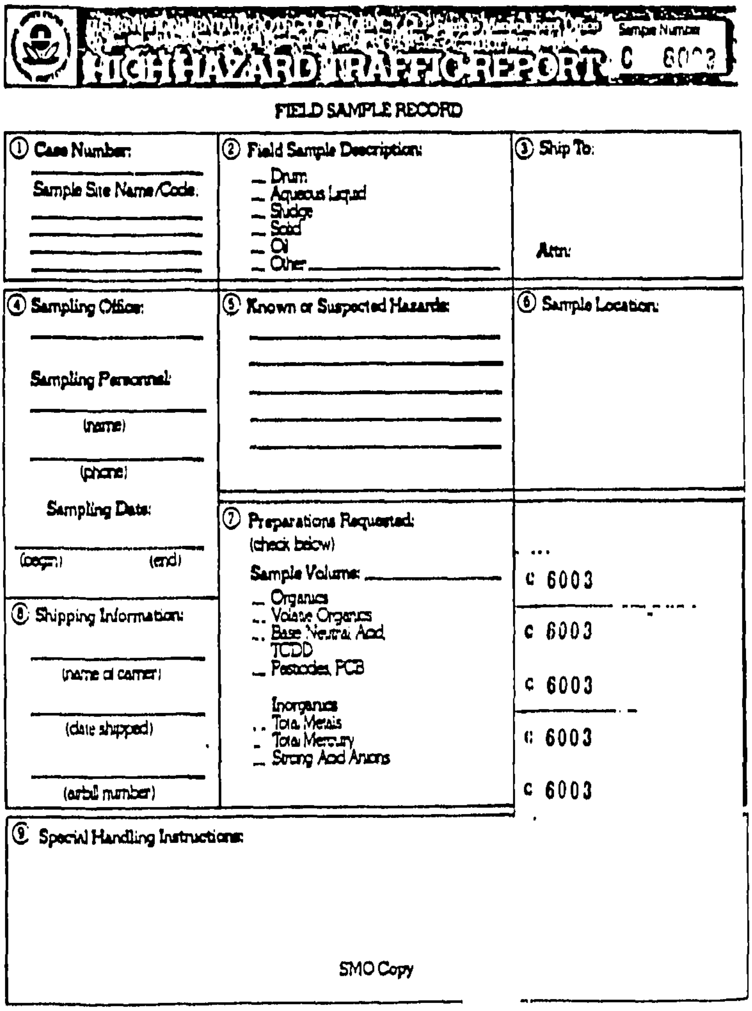
SEE REVERSE FOR ADDITIONAL INSTRUCTIONS

CLP SAMPLE NUMBER (FROM LABELS)	Ⓐ SAMPLE DESCRIPTION (FROM BOX 1) 1 2 3 4 5 6 7	Ⓑ CONCENTRATION	Ⓒ RAS ANALYSIS			Ⓓ SPECIAL HANDLING	Ⓔ STATION LOCATION
			VOLATILE	BASE/NEUT ACID	PESTICIDE PCBs		

EPA Form 2075-7 (8-87)

WHITE — SMO COPY PINK — CLIENT COPY WHITE — LAB COPY FOR RETURN TO SMO YELLOW — LAB COPY

ATTACHMENT E-3



[illegible]

HIGH HAZARD TRAFFIC REPORT

Sample Number
C 6003

FIELD SAMPLE RECORD

① **Case Number:**

Sample Site Name/Code:

② **Field Sample Description:**

- _ Drum
- _ Aqueous Liquid
- _ Sludge
- _ Solid
- _ Oil
- _ Other__________

③ **Ship To:**

Attn:

④ **Sampling Office:**

Sampling Personnel:

(name)

(phone)

Sampling Date:

(begin) (end)

⑤ **Known or Suspected Hazards:**

⑥ **Sample Location:**

⑦ **Preparations Requested:**
(check below)

Sample Volume: __________

- _ Organics
- _ Volatile Organics
- _ Base Neutral Acid TCDD
- _ Pesticides, PCB

Inorganics

- _ Total Metals
- _ Total Mercury
- _ Strong Acid Anions

c 6003

c 6003

c 6003

c 6003

c 6003

⑧ **Shipping Information:**

(name of carrier)

(date shipped)

(airbill number)

⑨ **Special Handling Instructions:**

SMO Copy

ATTACHMENT E-4

U.S. ENVIRONMENTAL PROTECTION AGENCY
CLP Sample Management Office
P.O. Box 818 - Alexandria, Virginia 22313
Phone: 703/557-2490 - FTS/557-2490

SAS Number

SPECIAL ANALYTICAL SERVICE
PACKING LIST

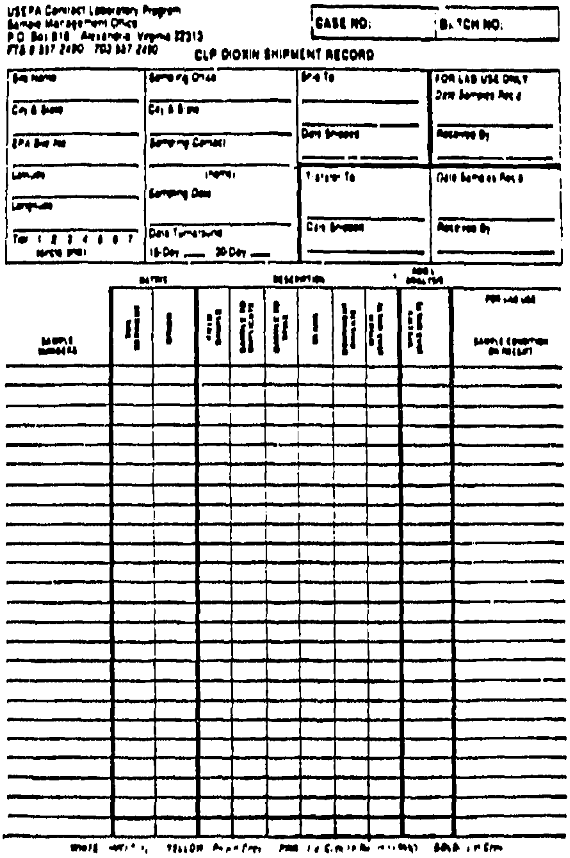
Sampling Office: ______	**Sampling Date(s):** ______	**Ship To:**	**For Lab Use Only**
Sampling Contact: ______ (name) ______ (phone)	**Date Shipped:** ______		**Date Samples Rec'd:** ______
	Site Name/Code: ______	**Attn:**	**Received By:** ______

	Sample Numbers	Sample Description - i.e., Analysis, Matrix, Concentration	Sample Condition on Receipt at Lab
1.			
2.			
3.			
4.			
5.			
6.			
7.			
8.			
9.			
10.			
11.			
12.			
13.			
14.			
15.			
16.			
17.			
18.			
19.			
20.			
			For Lab Use Only

White - SMO Copy, Yellow - Region Copy, Pink - Lab Copy for return to SMO, Gold - Lab Copy

Subject MANAGEMENT OF SAMPLING AND PREPARATION OF REQUIRED FORMS	Number SA-6 6	Page 25 of 28
	Revision	Effective Date 01/01/88

ATTACHMENT E-5



USEPA Contract Laboratory Program
Sample Management Office
P.O. Box 818 Alexandria Virginia 22313
FTS 8 557 2490 703 557 2490

CASE NO:

BATCH NO:

CLP DIOXIN SHIPMENT RECORD

Site Name	Sampling Office	Ship To	FOR LAB USE ONLY Date Samples Rec'd
City & State	City & State	Date Shipped	Received By
EPA Site No	Sampling Contact (name)	Transfer To	Date Samples Rec'd
Latitude	Sampling Date	Date Shipped	Received By
Longitude	Date Turnaround 15-Day ____ 30-Day ____		
Tier 1 2 3 4 5 6 7 (circle one)			

SAMPLE NUMBERS	MATRIX		DESCRIPTION						ADD'L ANALYSES	FOR LAB USE SAMPLE CONDITION ON RECEIPT
	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	

ATTACHMENT F

NUS CORPORATION
A Halliburton Company

SAMPLE LOG SHEET

☐ Monitoring Well Data
☐ Domestic Well Data
☐ Other ________

Page ____ of ____
Case # __________
By __________

Project Site Name ______________________ Project Site Number ________________
NUS Source No ____________________ Source Location ________________

Total Well Depth	Purge Data				
Well Casing Size & Depth	Volume	pH	S.C.	Temp (°C)	Color & Turbidity
Static Water Level					
One Casing Volume					
Start Purge (hrs)					
End Purge (hrs)					
Total Purge Time (min)					
Total Amount Purged (gal)					
Monitor Reading					
Purge Method					
Sample Method					
Depth Sampled					
Sample Date & Time	Sample Data				
	pH	S.C.	Temp (°C)	Color & Turbidity	
Sampled By					
Signature(s)	Observations / Notes				
Type of Sample ☐ Low Concentration ☐ High Concentration ☐ Grab ☐ Composite ☐ Grab - Composite					

Analysis	Preservative		Organic	Inorganic
		Traffic Report #		
		Tag #		
		AB #		
		Date Shipped		
		Time Shipped		
		Lab		
		Volume		

ATTACHMENT G

PROJECT SAMPLE SUMMARY

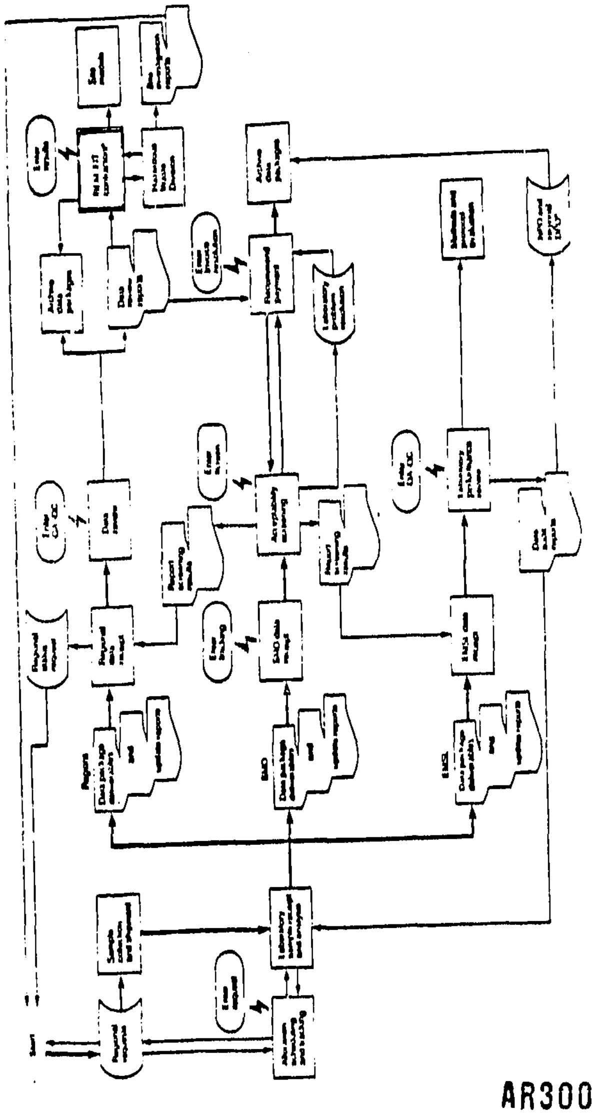
[illegible] ____________
[illegible] ____________
[illegible] ____________

DATE ____________
SAMPLERS INITIALS ____________

CASE NO / SAS NO	LABORATORY NAME	SAMPLE MEDIA	ANALYSES	NO OF SAMPLES	TRAFFIC REPORT NOs	BLANK TRAFFIC REPORT NOs	DUPLICATE TRAFFIC REPORT NOs	DATE SHIPPED

Subject MANAGEMENT OF SAMPLING AND PREPARATION OF REQUIRED FORMS	Number SA-6.6	Page 28 of 28
	Revision	Effective Date 01/01/88

ATTACHMENT H



Source: Almich, Budde and Shobe, 1986 ES&T, 20:16-21

NUS CORPORATION WASTE MANAGEMENT SERVICES GROUP	STANDARD OPERATING PROCEDURES	Number SF-1.1	Page 1 of 17
		Effective Date 01/01/88	Revision 0
		Applicability WMSG	
		Prepared Earth Sciences	
Subject ON-SITE WATER QUALITY TESTING		Approved A. K. Bomberger, P.E.	

TABLE OF CONTENTS

SECTION

1.0 PURPOSE

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

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

2.0 SCOPE

This procedure is applicable for use in an on-site groundwater quality monitoring program to be conducted during a remedial investigation or site investigation program at a hazardous or non-hazardous site. The procedures and equipment described are applicable to nearly all aqueous samples, including potable well water, monitoring well water, surface water, leachate and drummed water, etc. and are not, in general, subject to solution interferences from color, turbidity and colloidal material, or suspended matter.

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

3.0 GLOSSARY

3.1 pH MEASUREMENT

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

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

3.2 SPECIFIC CONDUCTANCE MEASUREMENT

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

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

Conductance - The conductance of a conductor 1 centimeter long and 1 square centimeter in cross-sectional area. Conductivity and specific conductance are used synonymously.

3.3 TEMPERATURE MEASUREMENT

None.

3.4 DISSOLVED OXYGEN MEASUREMENT

Galvanic Cell - An electrochemical cell in which chemical energy is spontaneously converted to electrical energy. The electrical energy produced is supplied to an external circuit.

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

3.5 OXIDATION-REDUCTION POTENTIAL MEASUREMENT

Oxidation - The process in which an atom or group of atoms loses electrons to achieve an increasing positive charge.

Reduction - The gaining of electrons by an atom or group of atoms and subsequent increase in negative charge.

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

3.6 SPECIFIC ION ELECTRODES MEASUREMENT

Specific Ion Electrode - An electrode which develops a potential difference across a membrane in response to the concentration differences for selected ions on either side of that membrane.

4.0 RESPONSIBILITIES

Site Manager - in consultation with the Project Geochemist, is responsible for determining which on-site water quality measurements can contribute to the RI, when these measurements shall be made, and the data quality objectives (DQOs) for these measurements. The Field Sampling and Analysis Plan (FSAP) should contain details of type, frequency and locations of the desired measurements.

Project Geochemist - primarily responsible for determining the type, frequency and locations for on-site water quality measurements as presented in the FSAP and for interpreting the results, including determination of which measurements are unrepresentative.

Field Operations Leader - responsible for implementing the FSAP, and also for deciding under what field conditions a particular on-site measurement will be unrepresentative or unobtainable.

Field Samplers/Analysts - responsible for the actual analyses that take place, including calibration, quality control and recording of results, as well as for the care and maintenance of the equipment in the field

5.0 GUIDELINES

5.1 MEASUREMENT OF pH

5.1.1 General

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

Measurements of pH can also be used to check the quality and corrosivity of soil and solid waste samples. However, these samples must be immersed in water prior to analysis, and specific techniques are not described.

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

5.1.2 Principles of Equipment Operation

Use of pH papers for pH measurement relies on a chemical reaction caused by the acidity or basicity of the solution with the indicator compound on the paper. Depending on the indicator and the pH range of interest, a variety of different colors can be used. Typical indicators are weak acids or bases, or both. Process chemistry and molecular transformations leading to the color change are variable and complex.

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

5.1.3 Equipment

The following equipment is needed for taking pH measurements:

- Accumet 150 portable pH meter, or equivalent.
- Combination electrode with polymer body to fit the above meter (alternately a pH electrode and a reference electrode can be used if the pH meter is equipped with suitable electrode inputs
- pH indicator paper, such as Hydrion or Alkacid, to cover the pH range 2 through 12
- Buffer solutions of pH 4, 7 and 10, or other buffers which bracket the expected pH range

5.1.4 <u>Measurement Techniques for Field Determination of pH</u>

1. *pH Meter*

The following procedure is used for measuring pH with a pH meter (Standardization is *according to manufacturers instructions*):

a. The instrument and batteries should be checked and calibrated prior to initiation of the field effort.

b. The accuracy of the buffer solutions used for field and laboratory calibration should be checked. Buffer solutions need to be changed often due to degradation upon exposure to the atmosphere.

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

d. Make sure all electrolyte solutions within the electrode(s) are at their proper levels and that no air bubbles are present within the electrode(s).

e. Immerse the electrode(s) in a pH-7 buffer solution.

f. Adjust the temperature compensator to the proper temperature (on models with automatic temperature adjustment, immerse the temperature probe into the buffer solution). Alternately, the buffer solution may be immersed in the sample and allowed to *reach temperature equilibrium before equipment calibration*. It is best to maintain buffer solution at or near expected sample temperature before calibration.

g. Adjust the pH meter to read 7.0.

h. Remove the electrode(s) from the buffer and rinse well with demineralized water. Immerse the electrode(s) in pH-4 or 10 buffer solution (depending on the expected pH of the sample) and adjust the slope control to read the appropriate pH. For best results, the standardization and slope adjustments should be repeated at least once.

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

j. Read and record the pH of the solution, after adjusting the temperature compensator to the sample temperature. pH should be recorded to the nearest 0.1 pH unit. Also record the sample temperature.

k. Rinse the electrode(s) with deionized water.

l. Keep the electrode(s) immersed in deionized water when not in use.

The sample used for pH measurement should never be saved for subsequent conductivity or chemical analysis. All pH electrodes leak small quantities of electrolytes (e.g., sodium or potassium chloride) into the solution. Precipitation of saturated electrolyte solution, especially at colder temperatures, or in cold water, may result in slow electrode response. Any visual observation of conditions which may interfere with pH measurement, such as oily materials, or turbidity, should be noted.

2. pH Paper

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

5.2 MEASUREMENT OF SPECIFIC CONDUCTANCE

5.2.1 General

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

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

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

5.2.2 Principles of Equipment Operation

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

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

5.2.3 Equipment

The following equipment is needed for taking specific conductance measurements:

- YSI Model 33 portable conductivity, meter, or equivalent
- Probe for above meter

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

5.2.4 Measurement Techniques for Specific Conductance

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

- Check batteries and calibrate instrument before going into the field.
- Calibrate the instrument daily when used. Potassium chloride solutions with a specific conductance closest to the values expected in the field should be used. Attachment A may be used for guidance.
- Rinse the cell with one or more portions of the sample to be tested or with deionized water
- Immerse the electrode in the sample and measure the conductivity. Adjust the temperature setting to the sample temperature.
- Read and record the results in a field logbook or sample log sheet.

If the specific conductance measurements become erratic, or inspection shows that any platinum black has flaked off the electrode, replatinization of the electrode is necessary. See the manufacturer's instructions for details.

Note that specific conductance is occasionally reported at temperatures other than ambient.

5.3 MEASUREMENT OF TEMPERATURE

5.3.1 General

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

5.3.2 Equipment

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

5.3.3 Measurement Techniques for Water Temperature

If a thermometer is used on a collected water sample:

- Immerse the thermometer in the sample until temperature equilibrium is obtained (1-3 minutes). To avoid the possibility of contamination, the thermometer should not be inserted into samples which will undergo subsequent chemical analysis.

- Record values in a field logbook or sample log sheet.

If a temperature meter or probe is to be used, the instrument should be calibrated according to manufacturer's recommendations with an approved thermometer before each measurement or group of closely spaced measurements.

5.4 MEASUREMENT OF DISSOLVED OXYGEN CONCENTRATION

5.4.1 General

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

The method monitoring discussed herein is limited to the use of dissolved oxygen meters only. Chemical methods of analysis (i.e., Winkler methods) are available, but require more equipment and greater sample manipulation. Furthermore, DO meters, using a membrane electrode, are suitable for highly polluted waters, because the probe is completely submersible, and are free from interference caused by color, turbidity, colloidal material or suspended matter.

5.4.2 Principles of Equipment Operation

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

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

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

5.4.3 Equipment

The following equipment is needed to measure dissolved oxygen concentration:

- YSI Model 56 dissolved oxygen monitor or equivalent.
- Dissolved oxygen/temperature probe for above monitor.
- Sufficient cable to allow the probe to contact the sample.

5.4.4 Measurement Techniques for Dissolved Oxygen Determination

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

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

- The probe should be conditioned in a water sample for as long a period as practical before use in the field. Long periods of dry storage followed by short periods of use in the field may result in inaccurate readings.

- The instrument should be calibrated in the field before each measurement or group of closely spaced measurements by placing the probe in a water sample of known dissolved oxygen concentration (i.e., determined by Winkler method) or in a freshly air-saturated water sample of known temperature. Dissolved oxygen values for air-saturated water can be determined by consulting a table listing oxygen solubilities as a function of temperature and salinity (see Attachment B).

- Immerse the probe in the sample. Be sure to provide for sufficient flow past the membrane, either by stirring the sample, or placing the probe in a flowing stream. Probes without stirrers placed in wells can be moved up and down.

- Record the dissolved oxygen content and temperature of the sample in a field logbook nor sample log sheet.

- Recalibrate the probe when the membrane is replaced, or as needed. Follow the manufacturer's instructions.

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

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

5.5 MEASUREMENT OF OXIDATION-REDUCTION POTENTIAL

5.5.1 General

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

5.5.2 Principles of Equipment Operation

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

5.5.3 Equipment

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

- Accumet 150 portable pH meter or equivalent, with a millivolt scale.
- Platinum electrode to fit above pH meter.
- Reference electrode such as a calomel, silver-silver chloride, or equivalent.

5.5.4 Measurement Techniques for Oxidation-Reduction Potential

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

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

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

- Thoroughly rinse the electrode with demineralized water.

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

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

5.6 SPECIFIC ION ELECTRODE MEASUREMENTS

5.6.1 General

Use of specific ion electrodes can be beneficial in the field for determining the presence and concentration of dissolved inorganic species which may be associated with contaminant plumes or leachate. Thus, electrodes can be used for rapid screening of water quality and determination of water migration pathways.

This procedure provides generic information for specific ion electrodes commonly used in groundwater quality monitoring programs and describes the essential elements of a field investigation program. Analytical methods using some specific ion electrodes have not been approved by the USEPA. In addition, calibration procedures and solutions, interferences and conditions and requirements for use for various electrodes vary greatly. Consequently, review of manufacturer's literature is mandatory prior to use.

5.6.2 Principles of Equipment Operation

All specific ion electrode measurements involve the use of a reference electrode, a pH meter, and a specific ion electrode (SIE). When the SIE and the reference electrode are immersed in a solution of the ion to be measured, a potential difference is developed between the two electrodes. This potential can be measured by a pH meter and related to the concentration of the ion of interest through the use of standard solutions and calibration curves.

Several different types of SIEs are in use: glass, solid-state, liquid-liquid membrane, and gas-sensing. All of the electrodes function using an ion exchange process as the potential determining mechanism. Glass electrodes are used for pH measurement. The glass in the tip of the electrode actually acts as a semi-permeable membrane to allow solution. Solid-state electrodes replace the glass membrane with an ionically-conducting membrane, (but act in essentially the same manner) while liquid-liquid membrane electrodes have an organic liquid ion exchanger contained in the pores of a hydrophobic membrane. Maintenance of the conducting interface, in combination with a reference electrode, allows completion of the electrical circuit and subsequent measurement of the potential difference. Gas-sensing electrodes have a membrane that permits the passage of gas only, thus allowing for the measurement of gas concentration. Regardless of the mechanism involved in the electrode, most SIEs are easy to use under field conditions. The sensitivity and applicable concentration range for various membranes and electrodes will vary.

5.6.3 Equipment

The following equipment is required for performing quantitative analyses using a specific ion electrode:

- A pH meter with a millivolt scale, or equivalent.
- The specific ion electrode for the parameter to be measured. A partial list of ions which can be measured includes cyanide, sulfide, ammonia, lead, fluoride and chloride.
- A suitable reference electrode to go with the above SIE.

Specific electrodes for other ions have also been developed, but are not widely used for field investigation efforts at this time. Note that of the specific electrodes referenced above, only fluoride and ammonia have analytical methods approved by the USEPA

5.6.4 Measurement Techniques for Inorganic Ions Using Specific Ion Electrodes

Different types of electrodes are used in slightly different ways and are applicable for different concentration ranges. Following the manufacturer's instructions, the general steps given below are usually followed:

- Immerse the electrode in water for a suitable period of time prior to sample analysis.

- Standardize the electrode according to the manufacturer's instructions, including necessary chemical additions for ionic strength adjustment, etc. Standard solutions normally differ by factors of ten in concentration. Constant stirring is needed for accurate readings.

- Immerse the electrode in the sample. Allow the reading to stabilize and record the results in a site logbook. Stir the sample at the same rate as the standards. Air bubbles near the membrane should be avoided, since this may cause interference in millivolt readings

(NOTE: Each SIE has substances which interfere with proper measurement. These may be eliminated using pretreatment methods as detailed by the manufacturer. It is important to know if interferences are present so that suspect readings may be noted as such.)

- If the pH meter does not read out directly, plot millivolts versus concentration for the standards and then determine sample concentration

6.0 REFERENCES

American Public Health Association, 1980. Standard Methods for the Examination of Water and Wastewater, 15th Edition, APHA, Washington, D.C.

USEPA, 1979. Methods for Chemical Analysis of Water and Wastes. EPA-600/4-79-020.

U.S. Geological Survey, 1984 National Handbook of Recommended Methods for Water Data Acquisition, Chapter 5: Chemical and Physical Quality of Water and Sediment U.S. Dept of the Interior, Reston, VA.

Ebasco Services Incorporated, REM III Field Technical Guideline FT-7.10. February 3, 1985

7.0 RECORDS

Attachment A - Specific Conductance of KC1 Solutions at 25 degrees Centigrade

Attachment B - Variation of Dissolved Oxygen Concentration in Water as a a Function of Temperature and Salinity.

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ATTACHMENT A

SPECIFIC CONDUCTANCE OF KC1 SOLUTIONS AT 25 DEGREES CENTIGRADE

Concentration	Specific	Conductance
mol/L	mg/L	HS/cm
0.0001	7.456	14.94
0.0005	37.28	73.90
0.001	074.56	147.0
0.005	372.8	717.8
0.00702	523.4	1000
0.01	745.6	1413
0.02	1591.2	2767

NOTE. This table has been modified from Standard Methods for the Examination of Water and Wastewater (1980)

ATTACHMENT B

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

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

ATTACHMENT B

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

Temperature C	Dissolved Oxygen mg/l					
	Chloride Concentration in Water					Difference/100 mg chloride
	0	5,000	10,000	15,000	20,000	
25	8.4	8.0	7.6	7.2	6.7	0.008
26	8.2	7.8	7.4	7.0	6.6	0.008
27	8.1	7.7	7.3	6.9	6.5	0.008
28	7.9	7.5	7.1	6.8	6.4	0.008
29	7.8	7.4	7.0	6.6	6.3	0.008
30	7.6	7.3	6.9	6.5	6.1	0.008
31	7.5					
32	7.4					
33	7.3					
34	7.2					
35	7.1					
36	7.0					
37	6.9					
38	6.8					
39	6.7					
40	6.6					
41	6.5					
42	6.4					
43	6.3					
44	6.2					
45	6.1					
46	6.0					
47	5.9					
48	5.8					
49	5.7					
50	5.6					

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

NUS CORPORATION

WASTE MANAGEMENT SERVICES GROUP

STANDARD OPERATING PROCEDURES

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Applicability WMSG	
Prepared Earth Sciences	
Approved A. K. Bomberger, P.E.	

Subject
SAMPLE PRESERVATION

TABLE OF CONTENTS

SECTION

1.0 PURPOSE

This procedure describes the appropriate containers to be used for samples depending on the analyses to be performed, and the steps necessary to preserve the samples when shipped offsite for chemical analysis.

2.0 SCOPE

Different types of chemicals react differently with sample containers made of various materials. For example, trace metals adsorb more strongly to glass than to plastic, while many organic chemicals may dissolve various types of plastic containers. It is therefore critical to select the correct container in order to maintain the quality of the sample prior to analysis.

Many water and soil samples are unstable, and therefore require preservation when the time interval between field collection and laboratory analysis is long enough to produce changes in either the concentration or the physical condition of the constituent(s) requiring analysis. While complete and irreversible preservation of samples is not possible, preservation does retard the chemical and biological changes that inevitably take place after the sample is collected.

Preservation techniques are usually limited to pH control, chemical addition(s) and refrigeration/freezing. Their purpose is to (1) retard biological activity, (2) retard hydrolysis of chemical compounds/complexes, (3) reduce constituent volatility, and (4) reduce adsorption effects.

3.0 GLOSSARY

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

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

4.0 RESPONSIBILITIES

Field Operations Leader - retains overall responsibility for the proper storage and preservation of samples. During the actual collection of samples, the sampling technician(s) will be directly responsible for the bottling, preservation, labeling, and custody of the samples they collect until released to another party for storage or transport to the analytical laboratory.

5.0 PROCEDURES

5.1 SAMPLE CONTAINERS

For most samples and analytical parameters either glass or plastic containers are satisfactory. In general, if the analyte(s) to be determined is organic in nature, the container should be made of glass. If the analyte(s) is inorganic, then the container should be plastic. Since container specification will depend on the analyte and sample matrix types (as indicated in Attachment A), duplicate samples should be taken when both organic and inorganic analyses are required.

Containers should be kept in the dark (to minimize biological or photooxidation/photolysis breakdown of constituent) until they reach the analytical laboratory. The sample container should allow approximately 5-10 percent air space ("ullage") to allow for expansion/vaporization if the sample is heated during transport (1 liter of water at 4°C expands by 15 ml if heated to 130°F/55°C), however, head space for volatile organic analyses should be omitted.

For CLP laboratories, containers will be obtained through the CLP Sample Management Office. For Responsible party actions or non-CLP laboratories, the laboratory should provide containers that have been cleaned according to U.S. EPA procedures. Sufficient lead time should be allowed. Shipping containers for samples, consisting of sturdy ice chests, are provided by the laboratory of the remedial investigation contractor.

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

General sample container and sample volume requirements are listed in Attachment A. Specific container requirements are listed in Attachment B.

5.2 PRESERVATION TECHNIQUES

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

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

5.2.1 Addition of Acid (H_2SO_4, HCl, or HNO_3) or Base

Addition of the following acids or bases may be specified for sample preservation; these reagents should be analytical reagent (AR) grade and should be diluted to the required concentration with double-distilled, deionized water in the laboratory, before Field sampling commences:

Acid Base	Concentration	Normality	Amount for Acidification*
HCl	1:1 dilution of concentrated HCl	6N	5-10 ml
H_2SO_4	1:1 dilution of concentrated H_2SO_4	18N	2-5 ml
HNO_3	Undiluted concentrated HNO_3	16N	2-5 ml
NaOH	400 grams solid NaOH in 870 ml water	10N	2 ml**

* Amount of acid to add (at the specified strength) per liter of water to reduce the sample pH to less than 2, assuming that the water is initially at pH 7, and is poorly buffered and does not contain particulate matter.

** To raise pH of 1 liter of water to 12.

The approximate volumes needed to acidify one liter of neutral water to a pH of less than 2 (or raise the pH to 12) are shown in the last column of the above table. These volumes are only approximate; if the water is more alkaline, contains inorganic or organic buffers, or contains suspended particles, more acid may be required. The final pH must be checked using narrow-range pH paper.

Sample acidification or base addition should proceed as follows:

- Check initial pH of sample with wide range (0-14) pH paper.
- Fill sample bottle to within 5-10 ml of final desired volume and add about 1/2 of estimated acid or base required, stir gently and check pH with medium range pH paper (pH 0-6 or pH 7.5-14, respectively).
- Add acid or base a few drops at a time while stirring gently. Check for final pH using narrow range (0-2.5 or 11-13, respectively) pH paper; when desired pH is reached, cap sample bottle and seal.

  Never dip pH paper into the sample; apply a drop of sample to the pH paper using the stirring rod.

5.2.2 Cyanide Preservation

Pre-sample preservation is required if oxidizing agents such as chlorine are suspected to be present. To test for oxidizing agents, place a drop of the sample on KI-starch paper; a blue color indicates the need for treatment. Add ascorbic acid to the sample, a few crystals at a time, until a drop of sample produces no color on the KI-starch paper. Then add an additional 0.6 g of ascorbic acid for each liter of sample volume. Add NaOH solution to raise pH to greater than 12 as described in 5.2.1. If oxidizing agents are not suspected, add NaOH as directed.

5.2.3 Sulfide Preservation

Samples for sulfide analysis must be preserved by addition of 4 drops (0.2 ml) of 2N zinc acetate solution per 100 ml sample. The sample pH is then raised to 9 using NaOH. The 2N zinc acetate solution is made by dissolving 220 g of zinc acetate in 870 ml of distilled water to make 1 liter of solution.

5.2.4 Preservation of Organic Samples Containing Residual Chlorine

Some organic samples containing residual chlorine must be treated to remove this chlorine upon collection (See Attachment A). Test the samples for residual chlorine using EPA methods 330.4 or 330.5 (Field Test Kits are available for this purpose). If residual chlorine is present, add 0.008% sodium thiosulfate (80 mg per liter of sample).

5.2.5 Field Filtration

When the objective is to determine concentration of dissolved inorganic constituents in a water system, the sample must be filtered through a non-metallic 0.45 micron membrane filter immediately after collection. A filtration system consisting of three to six filtration towers attached to a manifold and vacuum pump is recommended if much filtration is required in the field. Discard the first 20 to 50 ml of filtrate from each sample to rinse the filter and filtration apparatus. This technique minimizes the risk of altering the composition of the samples by the filtering operation. For analysis of dissolved metals, the filtrate is collected in a suitable bottle (see Section 5.1) and is immediately acidified to pH 2.0 or less with nitric acid whose purity is consistent with the measurement to be made. Inorganic anionic constituents may be determined using a portion of the filtrate that has not been acidified.

Samples used for determining temperature, dissolved oxygen, Eh, and pH should not be filtered. Do not use vacuum filtering prior to determining carbonate and bicarbonate concentration because it removes dissolved carbon dioxide and exposes the sample to the atmosphere. Pressure filtration can be done using water pressure from the well. If gas pressure is required, use an inert gas such as argon or nitrogen.

Do not filter samples for analysis of volatile organic compounds. If samples are to be filtered for analyzing other dissolved organic constituents, use a glass-fiber or metal-membrane filter and collect the samples in a suitable container (see Section 5.1). Because most organic analyses require extraction of the entire sample, do not discard any of it. After filtering, the membrane containing the suspended fraction can be sealed in a glass container and analyzed separately as soon as practicable. Total recoverable inorganic constituents may be determined using a second, unfiltered sample collected at the same time as the sample for dissolved constituents.

6.0 REFERENCES

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

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

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

Ebasco Services Incorporated; *REM III Field Technical Guideline No. FT-7.06 March 4, 1986*

7.0 ATTACHMENTS

Attachment A - General Sample Container and Preservation Requirements CERCLA RCRA Samples
Attachment B - Required Containers, Preservation Techniques, and Holding Times (3 sheets)

ATTACHMENT A
GENERAL SAMPLE CONTAINER AND PRESERVATION REQUIREMENTS

FT-7.06
REVISION 0

	SAMPLE TYPE & CONCENTRATION	CONTAINER[1]	SAMPLE SIZE	PRESERVATION[2]	HOLDING TIME[2]
WATER Organics (GC & GC/MS)	VOA	borosilicate glass	2 x 40 ml	Cool to 4°C	7 days
	Extractables Low	amber glass	2 x 2 l or 4 x 1 l	Cool to 4°C	5 days to extraction 40 days after extraction
	Medium	wide-mouth glass	4 x 32 oz	None	Same as above
Inorganics	**Metals** Low	high density (h.d.) polyethylene	1 l	HNO_3 to pH ≤2	6 months (Hg-30 days)
	Medium	wide-mouth glass	16 oz	None	6 months
	Cyanide Low	h.d. polyethylene	1 l	NaOH to pH >12	14 days
	Medium	wide-mouth glass	16 oz	None	
Organic/Inorganic	High Hazard	8 oz wide-mouth glass	6 oz	None	14 days
COD	--	h.d. polyethylene	0.5 l	H_2SO_4 to pH <2	28 days
TOC	--	h.d. polyethylene	0.5 l	HCl to pH <2	28 days
Oil & Grease	--	glass	1.0 l	H_2SO_4 to pH <2	28 dayr
Phenols	--	h.d. polyethylene	1.0 l	H_2SO_4 to pH <2	28 days
General Chemistry	--	h.d. polyethylene	1.0 l	None	---
SOIL Organics (GC & GC/MS)	VOA	2 x 120 ml (4 oz) wide-mouth glass	240 ml	Cool to 4°C	10 days
	Extractables Low/Medium	8 oz or 2 x 4 oz (120 ml) wide-mouth glass	6 oz	Cool to 4°C	10 days to extraction 40 days after extraction
Inorganics	Low/Medium	8 oz or 2 x 4 oz (120 ml) wide-mouth glass	6 oz	Cool to 4°C	NA
Organic/Inorganic	High Hazard	8 oz (120 ml) wide-mouth glass	6 oz	None	NA
Dioxin	All	4 oz (120 ml) wide-mouth glass	4 oz	None	NA
EP Toxicity	All	250 ml h.d. polyethylene	200 grams	None	NA
Air Volatile Organics	Low Medium	Charcoal Tube 7 cm long, 6 mm OD, 4 mm ID	100 l air	Cool to 4°C	NA

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1. All glass containers should have Teflon cap liners or septa.
2. See Attachment B.

SAMPLE CONTAINER AND PRESERVATION REQUIRMENTS CERCLA/RCRA SAMPLES

ATTACHMENT B
REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES

PAGE 1

Parameter No./Name	Container[1]	Preservation[2,3]	Maximum Holding Time[4]
INORGANIC TESTS:			
Acidity	P,C	Cool, 4°C	14 days
Alkalinity	P,C	Cool, 4°C	14 days
Ammonia	P,C	Cool, 4°C, H_2SO_4 to pH 2	28 days
Biochemical Oxygen Demand	P,C	Cool, 4°C	48 hours
Bromide	P,C	None required	28 days
Biochemical Oxygen Demand, Carbonaceous	P,C	Cool, 4°C	48 hours
Chemical Oxygen Demand	P,C	Cool, 4°C, H_2SO_4 to pH 2	28 days
Chloride	P,C	None required	28 days
Chlorine, Total Residual	P,C	None required	Analyze immediately
Color	P,C	Cool, 4°C	48 hours
Cyanide, Total and Amenable to Chlorination	P,C	Cool, 4°C, NaOH to pH 12, 0.6g ascorbic acid[5]	14 days[6]
Fluoride	P	None required	28 days
Hardness	P,C	HNO_3 to pH 2, H_2SO_4 to pH 2	6 months
Hydrogen Ion (pH)	P,C	None required	Analyze immediately
Kjeldahl and Organic Nitrogen	P,C	Cool, 4°C, H_2SO_4 to pH 2	28 days
Nitrate	P,C	Cool, 4°C	48 hours
Nitrate-Nitrite	P,C	Cool, 4°C, H_2SO_4 to pH 2	28 days
Nitrite	P,C	Cool, 4°C	48 hours
Oil and Grease	C	Cool, 4°C, H_2SO_4 to pH 2	28 days
Organic Carbon	P,C	Cool, 4°C, HCl or H_2SO_4 to pH 2	28 days
Orthophosphate	P,C	Filter immediately, Cool, 4°C	48 hours
Oxygen, Dissolved-Probe	G Bottle and top	None required	Analyze immediately
Oxygen, Dissolved-Winkler	G Bottle and top	Fix on site and store in dark	8 hours
Phenols	C	Cool, 4°C, H_2SO_4 to pH 2	28 days
Phosphorus (elemental)	C	Cool, 4°C	48 hours
Phosphorus, Total	P,C	Cool, 4°C, H_2SO_4 to pH 2	28 days
Residue, Total	P,C	Cool, 4°C	7 days
Residue, Filterable	P,C	Cool, 4°C	48 hours
Residue, Nonfilterable (TSS)	P,C	Cool, 4°C	7 days
Residue, Settleable	P,C	Cool, 4°C	48 hours
Residue, Volatile	P,C	Cool, 4°C	7 days
Silica	P	Cool, 4°C	28 days
Specific Conductance	P,C	Cool, 4°C	28 days
Sulfate	P,C	Cool, 4°C	28 days
Sulfide	P,C	Cool, 4°C, add zinc acetate plus sodium hydroxide to pH 9	7 days
Sulfite	P,C	None required	Analyze immediately
Surfactants	P,C	Cool, 4°C	48 hours
Temperature	P,C	None required	Analyze immediately
Turbidity	P,C	Cool, 4°C	48 hours
METALS:[7]			
Chromium VI	P,C	Cool, 4°C	24 hours
Mercury	P,C	HNO_3 to pH 2	28 days
Metals, except Chromium VI and Mercury	P,C	HNO_3 to pH 2	6 months

ATTACHMENT B
REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES

PAGE 2

Parameter No./Name	Container[1]	Preservation[2,3]	Maximum Holding Time[4]
ORGANIC TESTS:[8]			
Purgeable Halocarbons	G, Teflon-lined septum	Cool, 4°C, 0.008% $Na_2S_2O_3$[5]	14 days
Purgeable Aromatic Hydrocarbons	G, Teflon-lined septum	Cool, 4°C, 0.008% $Na_2S_2O_3$[5] HCl to pH 2[9]	14 days
Acrolein and Acrylonitrile	G, Teflon-lined septum	Cool, 4°C, 0.008% $Na_2S_2O_3$[5] adjust pH to 4-5[10]	14 days
Phenols[11]	G, Teflon-lined cap	Cool, 4°C, 0.008% $Na_2S_2O_3$[5]	7 days until extraction, 40 days after extraction
Benzidines[11]	G, Teflon-lined cap	Cool, 4°C, 0.008% $Na_2S_2O_3$[5]	7 days until extraction[13]
Phthalate Esters[11]	G, Teflon-lined cap	Cool, 4°C	7 days until extraction, 40 days after extraction
Nitrosamines[11,14]	G, Teflon-lined cap	Cool, 4°C, store in dark, 0.008% $Na_2S_2O_3$[5]	7 days until extraction, 40 days after extraction
PCBs[11] Acrylonitrile	G, Teflon-lined cap	Cool, 4°C	7 days until extraction, 40 days after extraction
Nitroaromatics and Isophorone[11]	G, Teflon-lined cap	Cool, 4°C, 0.008% $Na_2S_2O_3$[5], store in dark	7 days until extraction, 40 days after extraction
Polynuclear Aromatic Hydrocarbons[11]	G, Teflon-lined cap	Cool, 4°C, 0.008% $Na_2S_2O_3$[5], store in dark	7 days until extraction, 40 days after extraction
Haloethers[11]	G, Teflon-lined cap	Cool, 4°C, 0.008% $Na_2S_2O_3$[5]	7 days until extraction, 40 days after extraction
Chlorinated Hydrocarbons[11]	G, Teflon-lined cap	Cool, 4°C	7 days until extraction, 40 days after extraction
TCDD[11]	G, Teflon-lined cap	Cool, 4°C, 0.008% $Na_2S_2O_3$[5]	7 days until extraction, 40 days after extraction
PESTICIDES TESTS:			
Pesticides[11]	G, Teflon-lined cap	Cool, 4°C, pH 5-9[15]	7 days until extraction, 40 days after extraction
RADIOLOGICAL TESTS:			
1-5 Alpha, beta and radium	P, G	HNO_3 to pH 2	6 months

TABLE 2 Notes

(1) Polyethylene (P) or Glass (G).

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

(3) When any sample is to be shipped by common carrier or sent through the United States Mails, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172).

(4) Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid. Samples may be held for longer periods only if the permittee, or monitoring laboratory, has data on file to show that the specific types of samples under study are stable for the longer time, and has received a variance from the Regional Administrator.

(5) Should only be used in the presence of residual chlorine.

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ATTACHMENT B
REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES

PAGE 3

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

(7) Samples should be filtered immediately on-site before adding preservative for dissolved metals.

(8) Guidance applies to samples to be analyzed by GC, LC, or GC/MS for specific compounds.

(9) Sample receiving no pH adjustment must be analyzed within seven days of sampling.

(10) The pH adjustment is not required if acrolein will not be measured. Samples for acrolein receiving no pH adjustment must be analyzed within 3 days of sampling.

(11) When the extractable analytes of concern fall within a single chemical category, the specified preservation and maximum holding times should be observed for optimum safeguard of sample integrity. When the analytes of concern fall within two or more chemical categories, the sample may be preserved by cooling to 4°C, reducing residual chlorine with 0.008% sodium thiosulfate, storing in the dark, and adjusting the pH to 6-9; samples preserved in this manner may be held for seven days before extraction and for forty days after extraction. Exceptions to this optional preservation and holding time procedure are noted in footnote 5 (re: the requirement for thiosulfate reduction of residual chlorine) and footnotes 12, 13 (re: the analysis of benzidine).

(12) If 1,2-diphenylhydrazine is likely to be present, adjust the pH of the sample to 4.0±0.2 to prevent rearrangement to benzidine.

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

(14) For the analysis of diphenylnitrosamine add 0.008% $Na_2S_2O_3$ and adjust pH to 7-10 with NaOH within 24 hours of sampling.

(15) The pH adjustment may be performed upon receipt at the laboratory and may be omitted if the samples are extracted within 72 hours of collection. For the analysis of aldrin, add 0.008% $Na_2S_2O_3$.

B

APPENDIX B

FORMS FOR RI ACTIVITIES

ARMY CREEK LANDFILL SITE

NUS CORPORATION WASTE MANAGEMENT SERVICES GROUP	**STANDARD OPERATING PROCEDURES**	Number SA-6.4	Page 1 of 38
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		Applicability WMSG	
		Prepared Earth Sciences	
Subject FORMS USED IN RI ACTIVITIES		Approved A. K. Bomberger, P.E.	

TABLE OF CONTENTS

SECTION

1.0 PURPOSE

This procedure contains examples of forms in current use for RI activities, and a brief explanation of the function of these forms. The intent of this guideline is simply to compile and introduce these forms, and not to provide detailed explanations of the Forms.

2.0 SCOPE

Attachment A lists the forms illustrated in this procedure. Forms identified as controlled documents are issued by EPA, are sequentially numbered, and may not be altered. Those which are not listed as *controlled documents and not required documents issued by EPA may be altered or revised for* project-specific needs, with notification of, or in consultation with ZPMO

3.0 GLOSSARY

Controlled Document - A consecutively-numbered form released by EPA or the Zone Program Management Office (ZPMO) for use on a particular work assignment. All unused forms must be returned or accounted for at the conclusion of the assignment.

4.0 RESPONSIBILITIES

Field Operations Leader - The Field Operations Leader is responsible for ensuring that the appropriate forms illustrated in this guideline are correctly used and accurately filled out. In general, the sampling technician or Field Operations Leader will fill out forms related to sample labeling, *shipment and analysis (see Section 5.1); the site geologist/geohydrologist will fill out borings logs,* groundwater level and geohydrological test form (see Section 5.2); and the Field Operations Leader, site Health and Safety Officer, or field technicians, will fill out equipment calibration and maintenance records (see Section 5.3).

5.0 PROCEDURES

5.1 SAMPLE COLLECTION, LABELING, SHIPMENT AND REQUEST FOR ANALYSIS

5.1.1 Sample Label

The sample label is a 2- by 4 inch white label with black lettering and an adhesive backing. Attachment B-1 is an example of a sample label. Blank labels may be obtained from ZPMO or Regional Offices when needed. These labels are required on every sample but are not controlled *documents. Guidelines for filling out sample labels are contained in SA-6.1*

5.1.2 Sample Identification Tag

The Sample Identification Tag (Attachment B-2) must be used with samples collected for Contract Laboratory Program (CLP) analysis. The tag is a white, heavy paper label that is attached to the neck of the sample bottle with a string or wire. The Sample Identification Tag is a controlled document, and is available from the Regional Sample Control Center (RSCC). Procedure SA-6.1 provides the steps in filling out Sample Identification Tags.

5.1.3 Chain-of-Custody Record Form

The Chain-of-Custody Record Form accompanies a sample (or group of samples) as it is transferred from person to person. This form must be used for any samples collected for chemical or geotechnical analysis, whether on-site or off-site. It is a controlled document. Each EPA Region in Zone I uses a slightly different Chain-of-Custody form. Attachment B-3 illustrates a Chain-of-Custody Record form used by Region III. This form is available from the RSCC. Procedures for filling out Chain-of-Custody Record forms are contained in SA-6.1

5.1.4 Chain-of-Custody Seal

Attachment B-4 is an example of a custody seal. The Custody seal is a 1 by 3 inch adhesive-backed label. It is part of a chain-of-custody process and is used to prevent tampering with samples after they have been collected in the field. It is used whenever samples are shipped with an accompanying Chain-of-Custody Record form. The chani-of-custody seal is available from the RSCC. Procedure SA-6.1 describes the procedures for using chain-of-custody seals.

5.1.5 Bottle Delivery Order (DO) Form

If CLP analyses are requested, a Delivery Order (DO) form (Attachment B-5) is completed by the Authorized Requestor and submitted to the CLP Sample Bottle Repository (see Procedure SA-6.6). This form is required but not a controlled document.

5.1.6 Repository Packing List (PL) Form

The Repository Packing List form (Attachment B-6) is used for CLP analyses. This form is completed by the Sample Bottle Repository when the requested sample bottles are shipped. A copy of the PL is received with the sample bottle shipment and is retained by the Authorized Requestor

5.1.8 Sample Log Sheet

A Sample Log Sheet is a notebook (3-ring binder) page that is used to record specified types of data while sampling. Attachments B-7 to B-10 are examples of Sample Log Sheets. The data recorded on these sheets are useful in describing the waste source and sample as well as pointing out any problems encountered during sampling. Guidelines for filling out the Sample Log Sheet are contained in SA-6.6. Blank Sample Log Sheets may be obtained from ZPMO or Regional Offices; these forms are not controlled documents.

5.1.9 Traffic Reports (for CLP Laboratory Analyses)

A Traffic Report (TR) is a preprinted form that is provided by the EPA Sample Management Office to each Region through the Regional Sample Control Center (RSCC). These forms are obtained from the RSCC as needed for specific work assignments. These forms are part of the EPA sample-tracking system and are used to trace the shipment of samples for CLP laboratory analysis. Presently, these forms are for two types of samples: organics (OTR) and inorganics (ITR) (see Attachments B-11 and B-12, respectively). The organics and inorganics forms are used to document and identify the collection of low- and medium-concentrations samples for organic and inorganic analysis. Up to 20 samples can be recorded on each traffic report. Guidelines for filling out traffic report forms are contained in SA-6.6

5.1.10 Traffic Report Label

The Traffic Report Label is a small prenumbered white label with black lettering and an adhesive backing. Attachment B-13 provides examples of several traffic report labels. The number which appears on a traffic report label is uniquely numbered and used to track samples for CLP analysis. In addition to the number, each label contains a designation as to the type of analysis to be performed

(VOA, etc.) or as to preservation of the sample (preserved unpreserved, etc.). Use of these labels is described in Procedure SA-6.6.

5.1.11 Special Analytical Services (SAS) Packing List

In addition to routine analytical services (RAS), some special analytical services (SAS) are available through the CLP. These may include quick turnaround or verification analyses, non-priority pollutant analyses, analyses requiring lower detection limits than RAS methods provide, or other specific analyses (e.g., EP toxicity testing). For all "all SAS" type of request (in contrast to "RAS plus SAS," see Procedure SA-6.6), the SAS Packing List (Attachment B-14) is used rather than a traffic report. SAS Packing Lists are provided by the SMO to each region through the RSCC, which provides forms as required. Use of the SAS form is further described in Procedure SA-6.6

5.1.12 Dioxin Shipment Record (DSR)

The Dioxin Shipment Record (DSR) provides a record for one shipment batch (up to 24 samples) of dioxin samples to a CLP laboratory. Samples are individually numbered using the pre-printed labels provided with the DSR (see Attachment B-15) DSRs are provided by the SMO to each region through the RSCC. DSRs must be used to track shipment of dioxin samples submitted for CLP analysis See Procedure SA-6.6 for detailed description of the use of DSRs.

5.1.13 Sample Shipping Log

The sample shipping log, shown in Attachment B-16 is required by Region III EPA and is to be completed whenever samples are shipped to a CLP Laboratory The sample shipping log is then submitted to the RSCC the week following sample collection. In addition, a project sample summary (Attachment B-17) form must be completed and submitted to the RSCC.

5.2 GEOHYDROLOGICAL AND GEOTECHNICAL FORMS

5.2.1 Groundwater Level Measurement Sheet

A groundwater level measurement sheet, shown in Attachment C-1 should be filled out for each round of water level measurements at a site. These sheets are not controlled documents and can be obtained from ZPMO or Regional Offices.

5.2.2 Data Sheet for Pumping Test (Pumping Well)

During the performance of a pumping test, a large amount of data must be recorded, often within a short time period. he pumping test data sheet (Attachment C-2) facilitates this task by standardizing the data collection format, and allowing the time interval for collection to be laid out in advance. This form is not a controlled document and can be obtained from ZPMO or Regional Offices.

5.2.3 Data Sheet for Pumping Test (Observation Well) or In-Situ Hydraulic Conductivity Test

This data sheet (Attachment C-3) is similar to that described in Section 5 2.2. however, somewhat different data must be recorded for pumping test observation wells and in-situ hydraulic conductivity tests, as shown on this sheet. This form is not a controlled document and may be obtained from ZPMO or Regional Offices

5.2.4 Packer Test Reporting Forms

A packer test reporting form shown in Attachment C-4 is used for collecting data when conducting packer tests during monitoring well drilling. These sheets are not controlled documents and can be obtained from ZPMO or Regional Offices.

5.2.5 Summary Log of Boring

During the progress of each boring, a log of the materials encountered, operation and driving of casing, and location of samples must be kept. The Summary Log of Boring (Attachment C-4) is used for this purpose. In addition, if volatile organics are monitored on cores, samples or cuttings from the borehole (using HNU or OVA detectors), the results are entered on the boring log at the appropriate depth. The boring log also provides space for entry of the laboratory sample number and the concentration of a few key analytical results. This feature allows direct comparison of contaminant concentrations with soil characteristics.

The Summary Log of Boring is not a controlled document. Blank sheets are available from ZPMO or Regional Offices

5.2.6 Monitoring Well Construction Details Form

A Monitoring Well Construction Details Form must be completed for every monitoring well installed. This form contains specific information on length and type of well riser pipe and screen, backfill, filter sand and grout characteristics, and surface seal characteristics. This information is important in evaluating the performance of the monitoring well, particularly in areas where water levels show temporal variation, or where there are multiple (immiscible) phases of contaminants. Depending on the type of monitoring well (in overburden or bedrock), different forms are used (see Attachments C-5 through C-9).

The Monitoring Well Construction Details Form is not a controlled document. Blank sheets are available from ZPMO or Regional Offices. Guidelines on completing this form are contained in GH-1.7

5.2.7 Test Pit Log

When a test pit or trench is constructed for investigative or sampling purposes, a Test Pit Log must be filled out by the responsible field geologist or sampling technician. Test Pit Logs (Attachment C-8) are not controlled documents and are available from ZPMO or Regional Offices

5.3 *EQUIPMENT CALIBRATION AND MAINTENANCE FORMS*

5.3.1 Equipment Calibration Log

The calibration or standardization of monitoring, measuring or test equipment is necessary to assure the proper operation and response of the equipment, to document the accuracy, precision or sensitivity of the measurement, and determine if correction should be applied to the readings. Some items of equipment require frequent calibration, other infrequent. Some are calibrated by the manufacturer, other by the user

Each instrument requiring calibration has its own Equipment Calibration Log (Attachment D-1) which documents that the manufacturer's instructions were followed for calibration of the equipment, including frequency and type of standard or calibration device. This form is not a controlled document.

APPENDIX B

FORMS FOR RI ACTIVITIES

ATTACHMENT B-1

SAMPLE LABEL

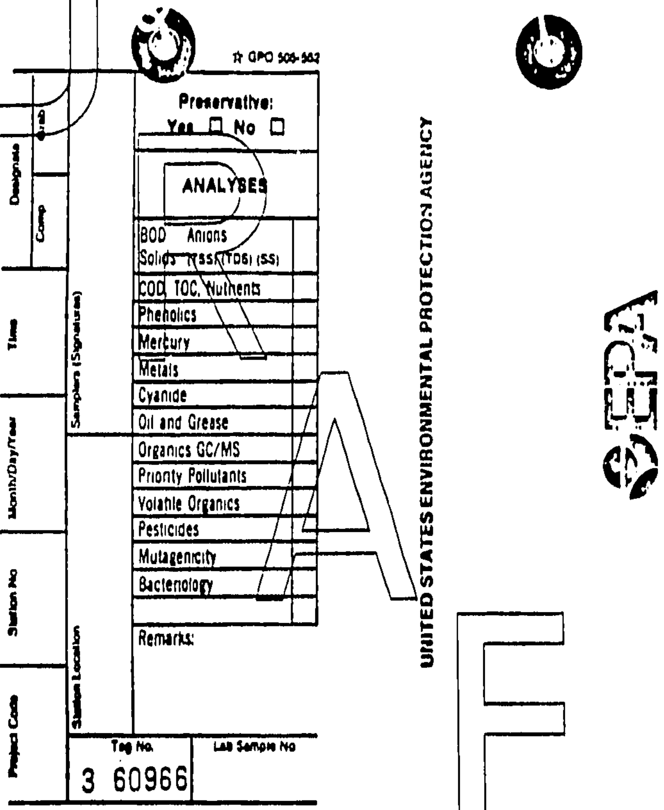
NUS
CORPORATION
LABORATORY SERVICES GROUP
5350 CAMPBELLS RUN ROAD
PITTSBURGH, PA 15205

NUS SAMPLE #

Client Name ______________________
Date Sampled ____________ Time ________
Client Sample # ____________________
Sample Source ______________________
____

ATTACHMENT B-2

SAMPLE IDENTIFICATION TAG



☆ GPO 505-562

Preservative: Yes ☐ No ☐

ANALYSES

BOD Anions	
Solids (TSS)(TDS)(SS)	
COD, TOC, Nutrients	
Phenolics	
Mercury	
Metals	
Cyanide	
Oil and Grease	
Organics GC/MS	
Priority Pollutants	
Volatile Organics	
Pesticides	
Mutagenicity	
Bacteriology	

Remarks:

Project Code

Station No

Month/Day/Year

Time

Designate: Comp / Grab

Station Location

Samplers (Signatures)

Tag No.	Lab Sample No
3 60966	

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

EPA

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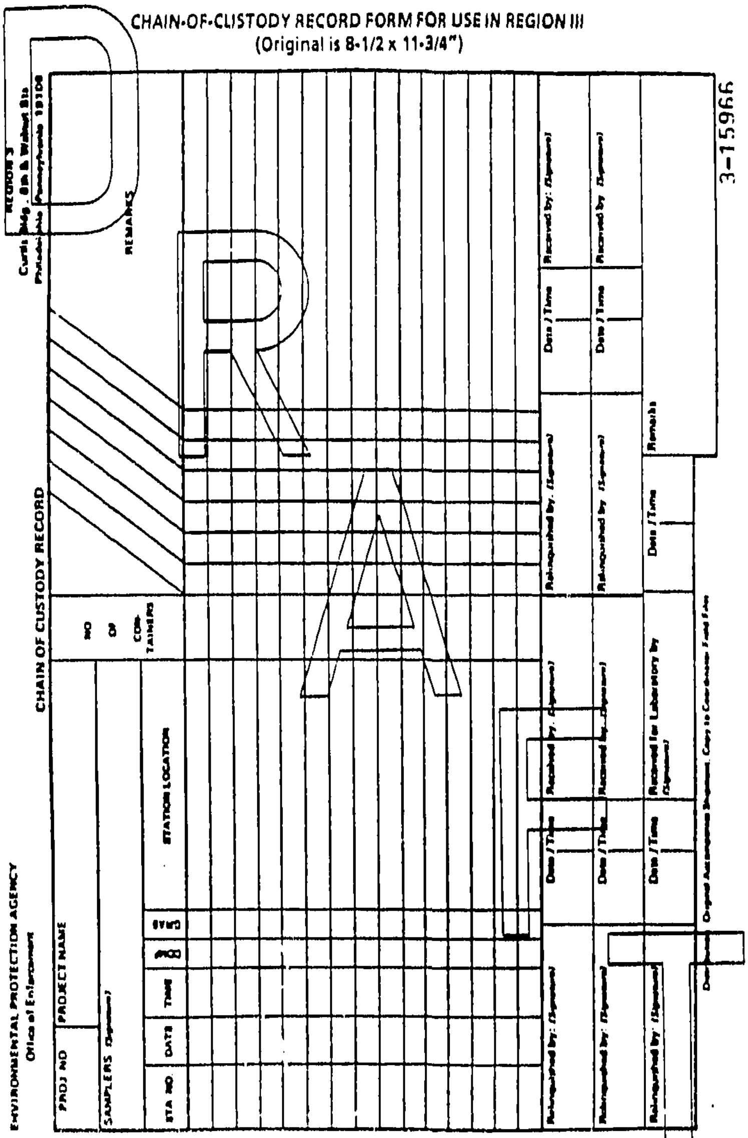
ATTACHMENT B-3

CHAIN-OF-CUSTODY RECORD FORM FOR USE IN REGION III
(Original is 8-1/2 x 11-3/4")



ENVIRONMENTAL PROTECTION AGENCY
Office of Enforcement

CHAIN OF CUSTODY RECORD

REGION 3
Curtis Bldg., 6th & Walnut Sts.
Philadelphia, Pennsylvania 19106

PROJ NO	PROJECT NAME					NO OF CON-TAINERS	REMARKS
SAMPLERS (Signature)							
STA NO	DATE	TIME	COMP	GRAB	STATION LOCATION		

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

Distribution: Original Accompanies Shipment; Copy to Coordinator Field Files

DRAFT

3-15966

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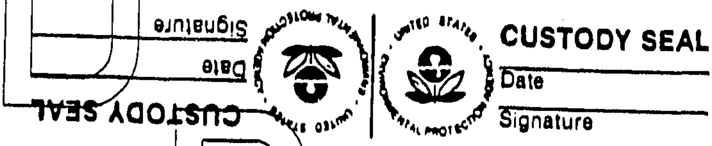
ATTACHMENT B-4

CHAIN-OF-CUSTODY SEAL



CUSTODY SEAL

Date

Signature

CUSTODY SEAL
Date
Signature

ATTACHMENT B-5
CLP SAMPLE BOTTLE REPOSITORY
SUPERFUND DELIVERY REQUEST

REQUEST NO. ____________

Date of Request: ____________

Type of Request:
Routine [ ]
Fast Turnaround [ ] ____________
Emergency [ ] ____________
(Date/Time request called in)

From (Name): ____________
Affiliation: ____________
Telephone: ____________
AR Signature: ____________

TO:
I-Chem Research Corporation
23787-F Eichler Street
Hayward, CA 94545
Phone: 415/782/3095

Ship the following items for arrival by: ____________ (Date)
(If applicable) Ship to arrive no earlier than: ____________ (Date)

Item		Description	No. of Items Per Case	No. of Cases Requested
A	80-oz.	amber glass bottle	6	
B	40-ml	glass vial	72	
C	1-liter	polyethylene bottle	12	
D	120-ml	wide-mouth glass vial	12	
E	1-oz.	wide-mouth glass jar	12	
F	8-oz.	wide-mouth glass jar	12	
G	4-oz.	wide-mouth glass jar	12	
H	1-liter	amber glass bottle	12	
J	32-oz.	wide-mouth glass jar	12	
K	4-liter	amber glass bottle	4	
L	500-ml	polyethylene bottle	24	

Ship To: ____________
(Provide ____________
street address) ____________
Attention: ____________
Call before delivery: ____________
(Phone No.): ____________

DISTRIBUTION: *White-Repository* *Yellow-Requestor* *Pink-SMO*

ATTACHMENT B-6

SUPERFUND SAMPLE BOTTLE REPOSITORY
PACKING LIST

REPOSITORY
I-CHEM Research
23787-F Eichler St.
Hayward, CA 94545
Telephone: 415/782-3905

Delivery Order No. ____________
Type of Order: ____________
Time: ____________ (emergency only)

DESTINATION (from Delivery Order)

Name: ____________
Address: ____________
____
____
To be delivered by: ____________
Telephone No: ____________

The materials listed below have been shipped as requested.
Date Shipped: ____________
Mode of Shipment: ____________
UPS, BOL, A/B No: ____________
Signature: ____________

Type of Shipment: ☐ Partial ☐ Complete ☐ Partial/Completes Order

Item No.	Description	No. of Cases Shipped	Lot Number(s)	QC Clearance Number(s)
1	80 oz. glass			
2	40-mL glass			
3	1-L poly			
4	120-mL glass			
5	16-oz glass			
6	8-oz glass			
7	4-oz glass			
8	1-L glass			
9	32-oz glass			

AUTHORIZED REQUESTOR USE ONLY

Sign below and forward the pink copy to SMO within 7 days of shipment receipt. Keep the gold copy for your file.

The above order was received by the designee, inspected, and accepted.

Date of Receipt: ____________ Requestor Signature: ____________

Send pink PL copy to: USEPA Sample Management Office (SMO)
P.O. Box 818
Alexandria, Virginia 22313

Distribution: White - Repository Copy
Yellow - Designee Copy
Pink - Requester Copy for Return to SMO
Gold - Requester File Copy

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ATTACHMENT B-7

NUS CORPORATION
A Halliburton Company

SAMPLE LOG SHEET

☐ Monitoring Well Data
☐ Domestic Well Data
☐ Other ________

Page ____ of ____
Case # ________
By ________

Project Site Name ________ Project Site Number ________
NUS Source No. ________ Source Location ________

	Purge Data				
Total Well Depth					
Well Casing Size & Depth	Volume	pH	S.C.	Temp. (°C)	Color & Turbidity
Static Water Level					
One Casing Volume					
Start Purge (hrs.)					
End Purge (hrs.)					
Total Purge Time (min.)					
Total Amount Purged (gal.)					
Monitor Reading					
Purge Method					
Sample Method					
Depth Sampled					

	Sample Data			
Sample Date & Time	pH	S.C.	Temp. (°C)	Color & Turbidity
Sampled By				

Signature(s)	Observations / Notes:

Type of Sample
☐ Low Concentration
☐ High Concentration
☐ Grab
☐ Composite
☐ Grab - Composite

Analysis	Preservative		Organic	Inorganic
		Traffic Report #		
		Tag #		
		AB #		
		Date Shipped		
		Time Shipped		
		Lab		
		Volume		

DRAFT

ATTACHMENT B-12

USEPA CONTRACT LABORATORY PROGRAM
SAMPLE MANAGEMENT OFFICE
P.O. BOX 818 ALEXANDRIA, VA 22313
703/557-2490 FTS-557-2490

CASE NO

SAS NO
(IF APPLICABLE)

INORGANIC TRAFFIC REPORT

(FOR CLP USE ONLY)

TYPE OF ACTIVITY (CIRCLE ONE) ① SUPERFUND—PA SI ESI RIFS RD RA ER NPLD O&M OTHER ____ NON SUPERFUND—________ PROGRAM	SHIP TO ③	SAMPLE DESCRIPTION ⑤ (ENTER IN BOX A) 1 SURFACE WATER 2 GROUND WATER 3 LEACHATE 4 SOIL 5 SEDIMENT 6 OIL (SAS) 7 WASTE (SAS)
SITE NAME	ATTN ________	DOUBLE VOLUME REQUIRED FOR MATRIX SPIKE/DUPLICATE AQUEOUS SAMPLE
CITY, STATE ____ SITE SPILL ID	SAMPLING DATE ④ BEGIN ______ END ______	SHIP MEDIUM AND HIGH CONCENTRATION SAMPLES IN PAINT CANS
REGION NO. SAMPLING COMPANY ②	DATE SHIPPED ____ CARRIER ____ ⑥	SEE REVERSE FOR ADDITIONAL INSTRUCTIONS
SAMPLER (NAME)	AIRBILL NO ________	

CLP SAMPLE NUMBER (FROM LABELS)	Ⓐ SAMPLE DESCRIPTION (FROM BOX 5) 1 2 3 4 5 6 7	Ⓑ CONCENTRATION L—LOW M—MED H—HIGH	Ⓒ RAS ANALYSIS							Ⓓ SPECIAL HANDLING	Ⓔ STATION LOCATION
			TOTAL METALS	CYANIDE	DISSOLVED METALS	HIGH ONLY (SAS)					
						[illegible]	[illegible]	[illegible]	[illegible]		

EPA Form 2075-6 (8-87)

WHITE — SMO COPY   PINK — CLIENT COPY   WHITE — LAB COPY FOR RETURN TO SMO   YELLOW — LAB COPY

ATTACHMENT B-13

TRAFFIC REPORT LABELS

MAB 342

6003

AC 865 · Soil/Sediment (VOA)

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ATTACHMENT B-14

U.S. ENVIRONMENTAL PROTECTION AGENCY
CLP Sample Management Office
P.O. Box 818 — Alexandria, Virginia 22313
Phone: 703/557-2490 - FTS/557-2490

SAS Number

SPECIAL ANALYTICAL SERVICE
PACKING LIST

Sampling Office:	**Sampling Date(s):**	**Ship To:**	**For Lab Use Only**
Sampling Contact:	**Date Shipped:**		**Date Samples Rec'd:**
(name)	**Site Name/Code:**		**Received By:**
(phone)		**Attn:**	

Sample Numbers	**Sample Description** **i.e., Analysis, Matrix, Concentration**	**Sample Condition on Receipt at Lab**
1.		
2.		
3.		
4.		
5.		
6.		
7.		
8.		
9.		
10.		
11.		
12.		
13.		
14.		
15.		
16.		
17.		
18.		
19.		
20.		

For Lab Use Only

White - SMO Copy, Yellow - Region Copy, Pink - Lab Copy for return to SMO, Gold - Lab Copy

Subject	Number	Page
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ATTACHMENT B-16

page___ of ___ EPA SAMPLE SHIPPING LOG FOR ALL SAMPLES SENT THROUGH THE CONTRACT LAB PROGRAM (12/85 version)

CASE#________ SAS#________ SITE NAME:________________________

Site Leader:____________________ EPA Project Officer:____________________

Phone:

					ORGANICS OR INORGANICS								SAS REQUEST=(details required)			
							DATA RECEIVED XX out items not requested									
QC SAMPLE INFO AND/OR COMMENTS	CONC. (low/med/high)	SAMPLE PHASE (aq/sol)	TYPE OF REQUEST org, dio inor, SAS	SAMPLE TRAFFIC REPORT NUMBER	LAB NAME	DATE SHIPPED	VOA	BNA	PEST	TCDD	METALS	CN	LAB NAME	SAS REQUEST (itemize)	DATE SHIPPED	DATA REC'D

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ATTACHMENT B-17

PROJECT SAMPLE SUMMARY

[illegible] ____________

[illegible] ____________

[illegible] ____________

DATE ____________

SAMPLER'S INITIALS ____________

CASE NO / SAS NO	LABORATORY NAME	SAMPLE MEDIA	ANALYSES	NO OF SAMPLES	TRAFFIC REPORT NO s	BLANK TRAFFIC REPORT NO s	DUPLICATE TRAFFIC REPORT NO s	DATE SHIPPED

ATTACHMENT C-1

GROUNDWATER LEVEL MEASUREMENT SHEET

Project Name: ______________________

Project No.: ______________________

Personnel: ______________________

Date: ______________________

LOCATION

Municipality: ______________________

County: ______________________

State: ______________________

Street or Map Location (If Off-Site) ______________________

WEATHER CONDITIONS

Temperature Range: ______________________

Precipitation: ______________________

Barometric Pressure: ______________________

Equipment No.: ______________________

Equipment Name: ______________________

Latest Calibration Date: ______________________

Tidally-Influenced: ☐ Yes ☐ No

Well or Piezometer Number	Date/Time	Elevation of Reference Point (Feet)*	Water Level Indicator Reading (Feet)*	Adjusted Depth (Feet)*	Groundwater Elevation (Feet)*

* All elevations to nearest 0.01 foot

Subject	Number	Page
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ATTACHMENT D-1

EQUIPMENT CALIBRATION LOG

Instrument (Name/Model No./Serial No.): ____________________

Manufacturer ____________________ Date Purchased ____________________

Calibration Date	Initial Settings	Standard/Gas Used	Procedure	Adjustments Made	Final Settings	Signature	Comments

Subject SITE LOGBOOK	Number SA-6 3	Page 4 of 4
	Revision	Effective Date 01/01/88

ATTACHMENT A
TYPICAL SITE LOGBOOK ENTRY

START TIME: ______________ DATE: ________________

SITE LEADER: ____________________________

PERSONNEL:

NUS	DRILLER	EPA

WEATHER: Clear, 68°F, 2-5 mph wind from SE

ACTIVITIES.

1. Steam jenney and fire hoses were set up.

2. Drilling activities at well ______ resumes. Rig geologist was ____________________. See Geologist's Notebook, No. 1, page 29-30, for details of drilling activity. Sample No. 123-21-S4 collected; see sample logbook, page 42. Drilling activities completed at 11:50 and a 4 inch stainless steel well installed. See Geologist's Notebook, No. 1, page 31, and well construction details for well ________

3. Drilling rig No 2 steam-cleaned at decontamination pit. Then set up at location of well ________

4. Well ________ drilled. Rig geologist was ____________________________. See Geologist's Notebook, No 2, page ______ for details of drilling activities. Sample numbers 123-22-S1, 123-22-S2, and 123-22-S3 collected; see sample logbook, pages 43, 44, and 45

5. Well ________ was developed. Seven 55-gallon drums were filled in the flushing stage. The well was then pumped using the pitcher pump for 1 hour. At the end of the hour, water pumped from well was "sand free."

6. EPA remedial project manger arrives on-site at 14:25 hours.

7. Large dump truck arrives at 14:45 and is steam-cleaned. Backhoe and dump truck set up over test pit ______________.

8. Test pit __________ dug with cuttings placed in dump truck. Rig geologist was ____________________. See Geologist's Notebook, No. 1, page 32, for details of test pit activities. Test pit subsequently filled. No samples taken for chemical analysis. Due to shallow groundwater table, filling in of test pit _____ resulted in a very soft and wet area. A mound was developed and the area roped off

9. Express carrier picked up samples (see Sample Logbook, pages 42 through 45) at 17:50 hours Site activities terminated at 18 22 hours. All personnel offsite, gate locked.

____
Field Operations Leader

FIGURE 7-2

NUS CORPORATION
ARCS III PROGRAM
TASK MODIFICATION REQUEST

EPA Work Assignment Number ____ Project Number ____ TMR Number ____

To ____________ Location ____________ Date ________

Description:

____

____

____

____

Reason for Change:

____

____

____

____

Recommended Disposition:

____

____

____

____

____

Field Operations Leader (Signature) ____ Date ____

Disposition:

____

____

____

____

____

Project Manager ____ Date ____

Distribution:
- Program Manager
- Quality Assurance Officer
- Project Manager
- Field Operations Leader

Others as required ________

____

____

____

ATTACHMENT B

PLANNED SAMPLING ACTIVITY REQUIRING CLP ANALYSES

Region ____________ Site ____________ Month/Year of Activity ________ Case ________

Sampling Organization ________ Regional Contact ____________ Telephone ________
(State, REM, FIT, etc.)

Type of Investigation ____________ Activity Status ____________
(RI/FS, Enforcement, etc.) (Definite, Conditional or Tentative)

Sampling Date(s) ____________ Alternate Sampling Date(s) ____________

Sample Shipment Date(s) ____________ Alternate Shipment Date(s) ____________

Routine Analytical Services Required	Media/Concentration									Total		
	Liquid or Water			Solid, Soil or Sediment			Other (SAS)					
	Low	Med	High	Low	Med	High	Low	Med	High	Low	Med	High
Full HSL Organics												
VOA Fraction Only												
Semi-VOA Fraction Only												
Pesticide/PCB Fraction Only												
Dioxin Only												
HSL Metals & Cyanide												
HSL Metals Only												

Special Analytical Services Required: Specify (Method, QA, Reporting Requirements to be Provided via SMO Client Request Form)

Submittal Status: ________
(Current, 1, 2 or 3 Month Projection)

Notification Status: ________
(Initial Notification or Resubmittal)

Resubmittal Status: ________
(Resubmittal With or Without Changes)

Submitted By: Date: Phone:

Approved By: Date:

Date Notification Received By SMO:

Date Laboratory(ies) Assigned:

ATTACHMENT C

CLP SAMPLE BOTTLE REPOSITORY
SUPERFUND DELIVERY REQUEST

Date of Request: ____________

Type of Request
Routine ☐
Fast Turnaround ☐ ____________
Emergency ☐ ____________
(date/time request called in)

FROM (Name): ____________
Affiliation: ____________
Telephone: ____________
AR Signature: ____________

TO
I-Chem Research Corporation
23787-F Eichler Street
Hayward, CA 94545
Phone: 415/782-3905

Ship the following items for arrival by: ____________(date)
(If applicable) Ship to arrive no earlier than: ____________(date)

Item		Description	No. of Items Per Case	No. of Cases Requested
A	80-oz	amber glass bottle	6	______
B	40-mL	glass vial	72	______
C	1-L	polyethylene bottle	12	______
D	120-mL	wide-mouth glass vial	12	______
E	16-oz	wide-mouth glass jar	12	______
F	8-oz	wide-mouth glass jar	12	______
G	4-oz	wide-mouth glass jar	12	______
H	1-L	amber glass bottle	12	______
J	32-oz	wide-mouth glass jar	12	______
K	4-L	amber glass bottle	4	______
L	500-mL	polyethylene bottle	24	______

Ship To: ____________
(provide ____________
street ____________
address) ____________
Attention: ____________
Call before Delivery ____________
(Phone No.) ____________

....

DISTRIBUTION: *White–Repository* *Yellow–Requestor* *Pink–SMO*

ATTACHMENT D - page 1

U.S. ENVIRONMENTAL PROTECTION AGENCY
CLP Sample Management Office
P.O. Box 818 - Alexandria, Virginia 22313
Phone: 703/557-2490 - FTS/557-2490

SAS Number

SPECIAL ANALYTICAL SERVICES

Client Request

☐ Regional Transmittal ☐ Telephone Request

A. EPA Region/Client: ____________________

B. Representative: ____________________

C. Telephone Number: ____________________

D. Date of Request: ____________________

Please provide below description of your request for Special Analytical Services under the Contract Laboratory Program. In order to most efficiently obtain laboratory capability for your request, please address the following considerations, if applicable. Incomplete or erroneous information may result in a delay in the processing of your request. Please continue response on additional sheets, or attach supplementary information as needed.

1. General description of analytical service requested: ____________________

2. Definition and number of work units involved (specify whether whole samples or fractions; whether organics or inorganics; whether aqueous or soil and sediments; and whether low, medium or high concentration): ____________________

3. Purpose of analysis (specify whether enforcement, remedial action, etc.): ____________________

4. Estimated date(s) of collection: ____________________

5. Estimated date(s) and method of shipment: ____________________

ATTACHMENT D - page 2

6. Approximate number of days results required after lab receipt of samples:
____

7. Analytical protocol required (attach copy if other than a protocol currently used in this program): ____________________
____
____

8. Special technical instructions (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.): ____________
____
____

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain of Custody documentation, etc.) If not completed, format of results will be left to program discretion. ____________
____
____
____

10. Other (use additional sheets or attach supplementary information, as needed):
____
____
____

11. Name of sampling/shipping contact: ____________________
Phone: ____________________

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please call us at the Sample Management Office.

C

Subject HEALTH AND SAFETY PLANS	Number HS01	Page 8 of 64
	Revision 3	Effective Date 08/15/87

APPENDIX C
HEALTH AND SAFETY PLAN

Subject HEALTH AND SAFETY PLANS	Number HS01	Page 9 of 64
	Revision 3	Effective Date 08/15/87

HEALTH AND SAFETY PLAN
BACKGROUND

Site Name: Army Creek Landfill

Address: U.S. Routes 13 and 301
New Castle County, Delaware

Client Contact: U.S. EPA-Eric Newman

Phone No.: 215-597-9238

Other Contacts: NUS Corp.-R. Stecik
GFEE-D. Sheridan

Phone No.: NUS - 412-788-1080

Phone No.: GFEE- 301-433-8832

Date Plan Requested: May 30, 1989

Purpose of Site Visit: Conduct Groundwater Sampling Task

Proposed Data of Work: Commencing June 23 - July 7, 1989

Proposed Site Investigation Team:

NUS Personnel:	Discipline/Tasks Assigned:
L. Johnson (or Designee)	Site Supervision

Other	Purpose
GFEE - Chen Yen	Field Operations Leader
GFEE - Louis Fiorucci	Site Health & Safety Officer
GFEE - Chen Yen	Field Quality Assurance Officer
GFEE -	Field Geologist/Sampler

Plan Preparation

Prepared by: Louis C. Fiorucci (HSSO) (5/30/89)

Reviewed and Approved by: Gary Beswick (/ /)

Reviewed

NUS Project Manager:

Follow Up Report:

Responsible Person: Louis C. Fiorucci

(Must fill out Follow up Report)

Facility Description: The Army Creek Landfill, formerly known as the Llangollen Landfill, is a 60 acre parcel that has been abandoned as a landfill since 1968.

Principal Disposal Method (type and location): The Army Creek Landfill was operated by New Castle County, Delaware from 1960 to 1968. The landfill accepted municipal wastes. During the eight years of operation, approximately 1.9 million cubic yards of refuse were placed in the landfill. It is estimated that 30 percent (600,000 cubic yards) of the refuse lies below the seasonal high water table. The average refuse thickness is approximately 25 feet.

According to the 1986 Feasibility Study, wastes deposited at the site included paper, cans, scrap sheet metal, bottles, wood, fabric, plastic, and glass. Although not known, it is suspected that organic solvents and metallic wastes may have been disposed at the landfill.

These wastes were disposed of using the modified area fill method. During landfilling, daily cover and intermediate cover were obtained from within the quarry and added only intermittently. Waste compaction is believed to have been minimal. Significant amounts of differential settlement have occurred at the landfill surface. The depressions that have formed due to differential settlement, the lack of significant site relief, and the silty sand characteristics of the final cover all enhance infiltration of precipitation from the landfill to the subsurface. In fact, model simulations conducted as part of the County's Feasibility Study provided predictions that at least 50 percent of the precipitation which falls onto the landfill surface percolates through the refuse.

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Subject HEALTH AND SAFETY PLANS	Number HS01	Page 11 of 64
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Unusual Features (containers, buildings, dikes, power lines, terain, etc.):

The site contains significant depressions throughout. In addition, portions of the site, along U.S. Routes 13 and 301S, have areas that drop off sharply from the top surface. Areas adjacent to the site have recovery wells and pipelines operating to remove contaminated groundwater from the shallow water table. A Conrail right-of-way exists along the northern boundary line of the site.

Status (active, inactive, unknown):

The Army Creek Landfill site is inactive as a landfill. There are active shallow water table recovery wells on the site that discharge the groundwater into Army Creek.

Locations of potential borrow sites have not yet been determined.

History (worker or non-worker injury; complaints from public; previous agency action):

No known worker or non-worker injury incidence data are available. Sometime after the landfill closed, contaminants exited the landfill via local groundwater and such contaminants were detected at residential wells. EPA instituted emergency action and installed groundwater monitoring wells and recovery wells. The recovery wells kept contaminated water from advancing to populated areas. In, 1986, EPA issued a Record of Decision following a Feasibility Study by New Castle County.

Monitoring used on previous site work; previous sampling data:

Monitoring performed on-site during both site investigations and the Feasibility Study included real time instrumental monitoring for organic vapors and natural gas releases from the fill. The results of such monitoring indicated that some organic vapor was detected, and that trace amounts of natural gas (methane) were detected in some of the monitoring well head spaces. The types and models of instruments used in the Feasibility Study are not known, but may have included the use of an HNU (PID) unit for organic vapor, and an explosive gas and oxygen meter like a LEL/O_2 unit. On April 26, 1988, site visitors walked the site and monitored the near breathing zone area for organic vapors with an OVA(FID) and ambient air for explosive limits with an LEL/O_2 type (comparable) instrument.

Subject HEALTH AND SAFETY PLANS	Number HS01	Page 12 of 64
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Previous Monitoring On Site (Previous Sampling Data Continued):

(1) The following constituents are present in ground water: benzene, 1, 2,-dichloropropane, methylene chloride, 2,4-dinitrotoluene, n-nitrosodimethylamine, 2,4,6,-trichlorophenol, bis(2 chloroethyl)-ether, and chlorodibromomethane, beryllium, cadmium, chromium, lead, mercury, nickel, iron, and manganese (Tables B1-B4).

(2) The following constituents have been found in surface water: (Tables B5-B8). Organics included phenol, bis (2-ethylhexyl) phthalate, butyl benzylphthalate and di-n-butylphthalate. Inorganics detected were aluminum, barium, cobalt, copper, iron, maganese, zinc, boron, calcium, magnesium and sodium (Tables B6,B8). Copper, silver, chromium, zinc, and manganese were found downstream at higher concentrations than upstream. Cadmium and mercury were the only parameters exceeding water quality criteria upstream and downstream of the pond (Tables B6, B8).

(3) The following constituents have been found in the Army Creek Sediments: iron, manganese, chromium, zinc, lead and barium (Table B9).

No organic compounds were detected in ambient air monitoring during site investigations. It should be mentioned that the air space over groundwater in some monitoring wells contained levels of organic compound vapors. Xylene exhibited the highest level of 200 ppb, and subsequent modeling resulted in a maximum ambient concentration down field of 2 ppb in extremely stable air conditions. When the concentrations are evaluated in ambient air mixing, the levels of contaminants in air drop to the parts per trillion range.

TABLE B1

ORGANIC COMPOUNDS IN RECOVERY WELLS EXCEEDING DRINKING WATER CRITERIA*

Well	Parameter	Max. Conc. (ug/l)	Criteria (ug/l)
RW-1	Benzene	12.0	5.0[a]
	1,2-dichloropropane	40.6	6.0[b]
	Methylene Chloride	32.0	0.19[c]
RW-10	2,4-dinitrotoluene	44.0	0.11[d]
	N-nitrosodimethylamine	<10.0	0.0014[d]
	1,2-dichloroethane	51.0	5.0[a]
RW-11	Benzene	<10.0	5.0[a]
RW-12	Benzene	10.3	5.0[a]
	1,2-dichloropropane	25.1	6.0[b]
	Methylene Chloride	21.2	0.19[c]
	2,4,6-trichlorophenol	3.0	1.8[e]
	Bis(2-chloroethyl)ether	<10.0	0.03[d]
RW-13	1,2-dichloropropane	26.8	6.0[b]
	Methylene Chloride	21.3	0.19[c]
	Bix(2-chloroethyl)ether	46.0	0.03[d]
	Benzene	37.0	5.0[a]
RW-14	Bis(2-chloroethyl)ether	<10.0	0.03[d]
	2,4-dinitrotoluene	38.0	0.11[d]
28	Benzene	40.0	5.0[a]
29	Benzene	45.0	5.0[a]
31	Benzene	150.0	5.0[a]
	Methylene Chloride	16.3	0.19[c]
	Chlorodibromomethane	19.2	0.19[c]
	2,4-dinitrotoluene	116.0	0.11[d]

* = All Laboratories (1983-1985)

a = Proposed Primary MCL (EPA, 13 November 1985)

b = Proposed RMCL (EPA, 13 November 1985)

c = Water Quality Criteria for Human Health (Fish and Drinking Water); concentration of total halomethanes (CWA) -.

d = Water Quality Criteria for Human Health - Fish and Drinking Water (CWA)

e Water Quality Criteria for Human Health - Adjusted for Drinking Water (CWA)

TABLE B2

Army Creek Landfill
New Castle, Delaware *
Ground Water Quality 1983

Total Concentration (mg/l)	Well 31 Upgradient	Well 54 Upgradient	Well 21 Downgradient	Well 26 Downgradient	Drinking Water Criteria
Antimony	<0.010	<0.010	<0.010	<0.010	0.146[a]
Arsenic	<0.002	<0.002	<0.002	<0.002	0.050[b]
Beryllium	<0.0005	<0.0005	<0.0005	<0.0005	0.0000037[c]
Cadmium	0.0006	0.001	0.0006	0.0015	0.010[b]
Chromium **	0.0214	0.0151	0.0134	0.0288	0.050[b]
Copper	0.0018	0.0133	0.0032	0.0104	1.3[f]
Lead	0.0101	0.0019	0.0018	0.0013	0.050[b]
Mercury	0.0022	0.0004	0.0002	0.0001	0.002[b]
Nickel	0.050	0.070	0.060	0.050	0.0154[c]
Selenium	<0.050	<0.050	<0.050	<0.050	0.010[b]
Silver	<0.0005	<0.0005	<0.0005	0.0018	0.050[b]
Thallium	<0.002	<0.002	<0.002	<0.002	0.0138[c]
Zinc	0.010	0.030	0.030	0.020	5.0[d]
Iron	2.510	0.220	1.140	3.650	0.3[e]
Manganese	0.110	0.040	0.010	0.080	0.050[e]

* = Analyzed by New Castle County Laboratory
** = Containers probable source of chromium 1983, leached chromium from polyurethane
a = Water Quality Criteria for Human Health - Fish and Drinking Water (CWA)
b = Interim Primary MCL (EPA, 13 November 1985)
c = Water Quality Criteria for Human Health - Adjusted for Drinking Water Only (CWA)
d = Water Quality Criteria - Organoleptic - Fish and Drinking Water (CWA)
e = Secondary MCL
f = Proposed MCL (EPA, 13 November 1985)

TABLE B3

ARMY CREEK LANDFILL
NEW CASTLE, DELAWARE

MONITORING WELL INORGANIC WATER QUALITY 1983*

Total Concentration (mg/l)	MW-1	MW-3	MW-4	MW-6	MW-9	MW-10	MW-11	MW-12	MW-13	MW-14	27	28	29	31	Drinking Water Criteria	Surface Water Criteria
Antimony	<0.001	<0.001	<0.001	<0.001	<0.005	<0.001	<0.001	0.0064	<0.001	0.0048	<0.001	0.0061	<0.005	<0.001	0.146^{a}	1.6^{c*}
Arsenic	0.0092	0.010	0.0158	0.0047	<0.001	0.014	0.0146	<0.003	0.0036	<0.0003	0.0204	0.0021	<0.001	0.005	0.050^{b}	0.010^{c}
Beryllium	<0.0005	<0.0005	<0.0005	<0.0005	0.0052	<0.0005	<0.0005	<0.0005	<0.0005	<0.0005	<0.0005	0.0034	0.0012	<0.0005	0.0000039^{d}	0.0053^{c*}
Cadmium	0.0056	0.0014	0.004	0.006	0.0009	0.0054	0.0051	0.0035	0.0021	0.0016	0.0038	0.0011	0.0009	0.0028	0.010^{b}	0.10^{e}
Chromium**	<0.005	0.0088	0.0099	0.0147	0.0173	0.0123	0.0156	0.0185	<0.005	<0.005	<0.005	0.0733	0.0647	<0.005	0.050^{b}	0.150^{e}
Copper	0.0089	0.0213	0.0084	0.0104	0.0095	0.0105	0.0132	0.0142	0.0144	0.009	0.0197	0.0344	0.008	0.026	1.3^{h}	0.50^{e}
Lead	0.0053	0.0037	<0.001	0.0084	0.0034	0.0055	0.0042	0.0017	0.0033	0.0034	0.0058	0.003	0.005	0.0131	0.050^{b}	0.150^{e}
Mercury	0.0018	0.0016	0.0016	0.0047	NR	0.0007	0.0031	0.0028	0.0023	0.001	0.004	NR	NR	0.0011	0.002^{b}	0.005^{e}
Nickel	0.310	0.080	0.230	0.120	0.016	0.040	0.170	0.090	0.250	0.050	0.150	0.0029	0.0023	0.210	0.0134^{d}	1.0^{e}
Selenium	0.0031	0.0041	0.0151	<0.002	<0.200	0.0079	0.0209	<0.002	<0.002	<0.002	0.0267	<0.200	<0.200	<0.002	0.010^{b}	0.020^{e}
Silver	0.0029	0.0027	0.0018	0.0013	0.0003	0.0088	0.0019	0.0037	0.0039	0.0004	0.0086	0.0118	0.0005	0.0012	0.050^{b}	0.10^{e}
Thallium	<0.001	<0.001	<0.001	<0.001	<0.005	<0.001	0.0067	0.001	0.0042	0.0013	0.005	0.0065	<0.005	<0.001	0.0178^{d}	0.04^{c*}
Zinc	0.020	0.030	0.010	0.010	<0.010	0.030	0.010	0.010	0.010	0.020	0.020	0.020	0.010	0.010	5.0^{f}	1.0^{e}
Iron	4.30	4.60	53.0	11.2	21.7	5.48	6.90	16.8	11.30	8.0	16.6	47.6	60.2	10.4	0.3^{g}	2.0^{e}
Manganese	0.95	0.52	1.04	1.35	1.3	0.48	0.51	1.03	0.20	0.79	0.47	1.59	1.56	1.21	0.50^{g}	--

* - Analyzed by New Castle County Laboratory

** - Containers probable source of chromium III; leaches chromium from polyurethane

NR - No Results
a - Water Quality Criteria for Human Health - Fish and Drinking Water (OWA)
b - Interim Primary MCL (EPA, 13 November 1985)
c - Criteria after Freshwater Aquatic Life (OWA)
d - Water Quality for Human Health - Adjusted for Drinking Water Only (OWA)
e - State of Delaware Water Quality Criteria for Discharges (1983)
f - Water Quality Criteria; Organoleptic - Fish and Drinking Water (OWA)
g - Secondary MCL
h - Proposed MCL (EPA, 13 November 1985)
* - Apparent Threshold Limit Values (OWA)

TABLE B4

Army Creek Landfill
New Castle, Delaware
Ground Water Quality
EPA September 1981 Sampling

Total Concentration (mg/l)	Well 58 Upgradient	Well 56 Upgradient	Well 28 Downgradient	Well 26 Downgradient	Well 30 Downgradient	RW-9 Recovery	RW-11 Recovery	Well 29 Recovery	Drinking Water Criteria	Surface Water Criteria
Antimony	<0.005	<0.005	<0.005	0.007	<0.005	<0.005	<0.005	<0.005	0.146^{a*}	1.6^{c*}
Arsenic	<0.002	<0.002	<0.002	<0.002	<0.002	<0.002	<0.002	<0.002	0.050^{b}	0.04^{c}
Beryllium	0.020	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	0.000003^{g}	0.0053^{c*}
Cadmium	<0.005	0.0066	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	0.010^{b}	0.1^{e}
Chromium	0.258	<0.100	<0.100	<0.100	<0.100	<0.100	<0.100	<0.100	0.05^{b}	0.150^{e}
Copper	0.490	0.090	<0.050	0.190	<0.050	<0.050	<0.050	<0.050	1.3^{g}	0.5^{e}
Lead	0.190	0.021	0.018	0.720	0.019	<0.002	0.010	<0.002	0.050^{b}	0.150^{e}
Mercury**	0.032	<0.0002	<0.0002	0.0002	<0.0002	<0.0002	<0.0002	<0.002	0.002^{b}	0.005^{e}
Nickel	0.154	<0.050	<0.050	0.083	<0.050	<0.050	<0.050	<0.050	0.0154^{d}	1.0^{e}
Selenium	<0.002	<0.002	<0.002	<0.002	0.003	<0.002	<0.002	<0.002	0.010^{b}	0.02^{e}
Silver	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	0.05^{b}	0.1^{e}
Thallium	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010	0.0128^{d}	0.04^{c*}
Zinc	0.133	0.093	0.044	0.188	0.056	<0.050	<0.050	<0.050	5.0^{f}	1.0^{e}

** Analyzed beyond the holding time

a = Toxicity value for protection of potable water supplies
b = Primary Drinking Water Standard
c = Secondary Drinking Water Standard
d = Water Quality Criteria for Human Health – Adjusted for Drinking Water Only (OHA)
e = State of Delaware Water Quality Criteria for Discharges (OHA)
f = Water Quality Criteria, Organoleptic – Fish and Drinking Water (OHA)
g = Proposed MCL (EPA, 13 November 1985)
* = Adjusted Threshold Limit Values (OHA)

TABLE B5

Army Creek Landfill
New Castle, Delaware
Army Creek and Pond Water Quality
EPA/FIT Sampling 9-11 November 1981
Organic Concentrations (ug/l)

Station	Pyrene	PCB-1248	Fluoranthene	Bis (2-ethylhexyl) Phthalate	Di-n-butyl Phthalate	Benzo (a) anthracene	Chrysene	Anthracene	Phenanthrene	Benzene (a) pyrene	Benzo (b) Fluoranthene	Benzo (k) Fluoranthene	Benzo (ghi) perylene
I (Rt. 13)	ND	ND	ND	*	*	ND	ND	ND	ND	ND	ND	ND	ND
II (RR tracks)	*	ND	*	*	*	*	*	*	*	ND	ND	ND	ND
III (nr. RW-9) (discharge)	*	ND	*	*	*	*	*	*	*	ND	ND	ND	ND
IV (nr. RW-4) (discharge)	*	ND	*	*	*	*	*	*	*	*	*	*	*
V (nr. Amoco)	ND	350.	*	*	*	ND	ND	*	*	ND	ND	ND	ND

ND = Not Detected

* Indicates chemical present in sample

TABLE F6

Army Creek Landfill
New Castle, Delaware
Army Creek and Pond Water Quality
Inorganic Concentrations (mg/l)
EPA/FIT Sampling 9-11 November 1981

Total Conc.	Station I (Rt. 13)	Station II (RR tracks)	Station III (nr. RW-9 discharge)	Station IV (nr. RW-4 discharges)	Station V (nr. Amoco)	Surface Water Criteria
Aluminum	0.150	0.830	0.330	0.150	0.830	—
Chromium	<0.010	<0.010	<0.010	<0.010	<0.010	0.021^b
Barium	0.060	0.170	0.270	0.090	0.310	—
Beryllium	<0.002	<0.002	<0.002	<0.002	<0.002	0.0153^b
Cadmium	<0.005	<0.005	<0.005	<0.005	<0.005	0.000025^a
Cobalt	<0.010	0.010	0.010	<0.010	0.050	0.022^a
Copper	<0.020	<0.020	<0.020	<0.020	0.200	1.000^b
Iron	1.760	2.160	25.000	0.840	167.000	—
Lead	<0.010	<0.010	<0.010	<0.010	<0.010	0.17^a
Nickel	<0.020	<0.020	<0.020	<0.020	<0.020	1.8^a
Manganese	0.270	0.590	0.830	0.130	0.470	—
Zinc	0.050	0.050	0.020	0.020	0.150	0.32^a
Boron	0.050	0.080	<0.010	0.030	<0.010	—
Vanadium	<0.010	<0.010	<0.010	<0.010	<0.010	—
Calcium	21.800	13.600	22.100	20.700	11.600	—
Magnesium	2.700	1.700	2.500	2.800	5.800	—
Sodium	17.900	28.300	34.500	26.300	13.00	—
Arsenic	<0.010	<0.010	<0.010	<0.010	<0.010	0.04^b
Antimony	<0.020	<0.020	<0.020	<0.020	<0.020	1.6^a
Selenium	<0.010	<0.010	<0.010	<0.010	<0.010	0.26^c
Thallium	<0.010	<0.010	<0.010	<0.010	<0.010	0.04^a
Mercury	<0.001	<0.001	<0.001	<0.001	<0.001	0.0000017^b
Tin	<0.020	<0.020	<0.020	<0.020	<0.020	—
Silver	<0.020	<0.020	<0.020	<0.020	<0.020	0.0011^a

a = Criteria for Freshwater Aquatic Life - total recoverable assuming a hardness of 100. mg/l as $CaCO_3$ (EPA)
b = Criteria for Freshwater Aquatic Life
c = Acute Toxicity Criteria for Freshwater Aquatic Life (EPA)
d = Apparent Threshold Limit Values (EPA)

TABLE E7

Army Creek Landfill
New Castle, Delaware
Army Creek Water Quality

Base/Neutral Organic Compounds (ug/l)*

Location	Date	Bis(2-ethylhexyl) Phthalate	Di-n-butyl Phthalate
Upstream	Sept. 1983	13.2	83.6
Downstream	Sept. 1983	NP	40.1
Surface Water or Criteria		3.0^{a*}	3.0^{a*}

* Analyzed by New Castle County Laboratory

NP = Not Found

a = Criteria for Freshwater Aquatic Life for Phthalate Esters (CWA)
* = Apparent Threshold Limit Values (CWA)

TABLE B8

Army Creek Landfill
New Castle, Delaware
Army Creek Water Quality

Total Inorganic Concentrations (mg/l)

Location	Date	Antimony	Arsenic	Beryllium	Cadmium	Chromium**	Copper	Lead	Mercury	Nickel	Selenium	Silver	Thallium	Zinc	Iron	Manganese
Upstream	Sept. 1983	<0.010	<0.002	<0.0005	0.0016	0.015	0.0064	0.0127	0.0005	0.110	<0.050	<0.0005	<0.002	0.030	2.78	0.180
Downstream	Sept. 1983	<0.010	<0.002	<0.005	0.0015	0.0176	0.0068	0.0077	0.0001	0.080	<0.050	0.0006	<0.002	0.130	2.150	0.400
Surface Water Criteria		1.6^{b*}	0.04^{b}	0.0053^{b*}	0.000025^{a}	0.021^{b}	0.022^{c}	0.17^{c}	0.000017^{c}	1.8^{a}	0.76^{d}	0.0041^{c}	0.04^{b*}	0.32^{b}	--	--

Analyzed by New Castle County Laboratory

* Apparent Threshold Limit Values (EPA)
** Chromium probably leached from container by using HNO_3 in polyethylene containers.

a = Water Quality Criteria for Human Health - Fish and Drinking Water (EPA)
b = Criteria for Freshwater Aquatic Life (EPA)
c = Criteria for Freshwater Aquatic Life - total recoverable (compound)
d = Acute Toxicity Criteria for Freshwater Aquatic Life (EPA)

TABLE B9

Army Creek Landfill
New Castle, Delaware
Army Creek and Pond Sediment Quality
Total Inorganic Concentrations (ppb)
DNREC Sampling and Analysis

Total Concentration	Station 1	Station 2	Station 3	Station 4	Station 5	Station 6	Station 7	Station 8
Iron	21,502,000.	9,505,000	44,010,000.	19,530,000.	45,175,000.	27,962,000.	2,867,000.	18,259,000.
Manganese	163,000.	390,000.	274,000.	398,000.	845,000.	1,320,000.	24,260.	87,020.
Chromium	18,450.	10,210.	25,540.	<10,000.	14,920.	14,930.	<10,000.	11,570.
Silver	<10,000.	<10,000.	<10,000.	<10,000.	<10,000.	<10,000.	<10,000.	<10,000.
Zinc	87,270.	90,920.	274,000.	70,750.	143,000.	210,000.	22,210	93,060
Lead	21,400.	142,000.	175,000.	25,410.	70,630.	56,750.	10,110.	31,690.
Cadmium	<10,000.	<10,000.	<10,000.	<10,000.	<10,000.	<10,000.	<10,000.	<10,000.
Mercury	<500.	<500.	<500.	<500.	<500.	630.	<500	<500.
Arsenic	<3000.	<3000.	9250.	<3000.	5470.	13,510.	<3000.	<3000.
Selenium	<500.	<550.	210.	<500.	<500.	<500.	<500.	<500.
Barium	42,800.	38,310.	234,000.	73,720.	145,000.	76,660.	<10,000.	57,300.

Station 1 = Army Creek, West of Route 13
Station 2 = Army Creek, East of Route 13, Upstream of Weir
Station 3 = Army Creek, Pond Entrance
Station 4 = Army Creek, Pond Effluent
Station 5 = Army Creek, Under Railroad Bridge
Station 6 = Army Creek, Tidal Gate, East of Route 9
Station 7 = Gravel Pit Pond
Station 8 = Intermittent Stream East of US 13

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Characteristics of Waste: Wastes vary in source type, but contact with contaminated groundwater and sediment may have toxic effects.

Corrosive ____ Flammable ____ Radioactive ____

Toxic X Volatile ____ Reactive ____ Inert ____

Physical Hazards of Site:

Describe hazards (taking into account reactivity, stability, flammability, operational concerns, sampling, decontamination, etc.).

The landfill site consists of high and low spots throughout that may pose a hazard to persons walking (sprain or fall), or driving a vehicle (roll over). The steep slope on the north/northwest and west perimeters of the site pose a fall hazard to walking/riding workers. The operating groundwater recovery wells and associated piping pose a trip, collison, electrical and contaminant exposure hazard to workers on-site. The above ground well casings (both recovery wells and monitoring wells) present a collison hazard. Other hazards include sharp objects that my project up through the surface; surface soil/hand auger samplers may contact sharp objects in the fill below surface; and soils engineers may come in contact with contaminated soils or fall into Army Creek. Side slopes of the landfill adjacent to Army Creek are heavily vegetated and somewhat steep.

Personal Protection:

Personal protection used on previous site visits. Dates and scope of work included.

Information describing P.P.E. used in previous site investigations was not available in the EPA documents that have been reviewed. The Feasibility study report issued during 1986 reported the installation of ground water monitoring wells, and the sampling of soil, ground water, Army Creek water (surface), and creek sediments. Level D P.P.E. was recommended and consisted of hard hart, safety glasses, safety shoes, radiation badge, and coveralls. A site walkthrough visit was conducted in Level D protection on April 26, 1988. No organic vapors nor combustible mixtures were detected during this visit.

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SCOPE OF WORK (Task 1, Task 2, etc.)

The scope of work for the field components of the RI/FS is presented below:

TASK 1: Groundwater Sampling will be performed at the discharge end of the pipe for each of 10 recovery wells. Each pipeline extends from the well head outlet, across the landfill surface, and to the edge of the bank of Army Creek. The water splashes down RIP-RAP into Army Creek. Water samples will be taken by the field Geologist using manual grab techniques. As water samples are taken, the HSSO will monitor the breathing zone area for presence of organic vapors using either an OVA, HNU, or TIP on-line instrument, and potential explosive gas mixtures using a LEL/O_2 meter.

Contaminants and Routes of Exposure

Tables B10 and B11 present a summary of the compounds found in different environments (groundwater (gw), surface water (sf), and creek sediments (sed) on the site, the exposure route (inhal-inhalation, ing-ingestion, abs-absorption, and con-contact), some (not all) typical toxicological effects, and current OSHA permissible exposure limits.

TABLE B10

ORGANIC CONTAMINANTS

EXPOSURE COMPOUND	ROUTE	Permissible Exposure Limit	TOXICITY	Location Max. Water Conc. (ppb)	CAS No.#	C
benzene	inhal	1.00 ppm	Irritation to nose carcinogen-blood forming organs	GW 150	71-43-2	CA
1,2-dichloropropane	inhal/ing/con	75.00 ppm	Eye, skin irritation, dizziness	GW 41	78-87-5	-
Methylene Chloride	inhal/ing	500.00 ppm	Weakness, nausea, angina, carcinogen	GW 32	75-09-2	CA
2,4 dinitrotoluene	inhal/Abs/con/ing	1.5 mg/m3	Anoxia, cyanosis, carcinogen	GW 116	25321-14-6	CA
n-nitrosodimethylamine	inhal/Abs/con/ing	(1)	Nausea, vomiting, reduced function, liver, kidney carcinogen	GW LT. 10	62-75-9	CA
2,4,6-trichlorophenol	inhal/Abs/con/ing	(1)	Acute poisoning, motor weakness, cancer-dioxin	GW 3	88-06-2	CA
bis(2 chloroethyl) ether	inhal/Abs/con/ing	5.00 ppm	Irritation, nose, cough vomit, carcinogen	GW 46	111-44-4	CA
chlorodibromomethane	inhal/ing	(1)	Irritation, nose, cough potential carcinogen	GW 19.2	124-48-1	CA
Phenol	inhal/Abs/con/ing	5.00 ppm	Irritatant -- eyes, nose, throat, muscle ache, cyanosis, kidney damage, tremors	SW L.T. 10	108-95-2	-
bis(2 ethyhexyl)phthalate	inhal/ing/con	5.00 ppm	Carcinogen	SW 13.2	00117817	CA
butyl benzylphthalate	inhal/ing/con	5.00 mg/m3	Irritation, respiratory systems, GI tract	SW L.T. 10	00085687	-
di-n-butylphthalate	inhal/ing/con	5.00 mg/m3	Irritation, respiratory systems, GI tract	SW 83.6	84-74-2	-
PCB-1248	inhal/ing/con	0.5 mg/m^3	Irritation	SW 350	11097-69-1	C

Location:
GW - Groundwater
SW - Surface water
SED - Sediment
L.T. - Less than

Route:
inhal - inhalation
Abs - absorption
con - contact
ing - ingestion

C	Carcinogenicity
CH	Carcinogenticy established for humans
CA	Carcinogenicity established for animals
CSH	Carcinogenicity suspected for humans
CSA	Carcinogenicity suspected for animals

(1) No permissible exposure limit has been established.

TABLE B11

INORGANIC CONTAMINANTS

EXPOSURE COMPOUND	ROUTE	Permissible Exposure Limit(mg/m^3)	TOXICITY	Location Max. Water Conc. (ppb)	CAS NO.#	C
Beryllium	inhal	2.0ug	Respiratory Distress carcinogen	GW 1.2	7440-41-7	CA
admium	inhal/ing	0.2	Pulmonary edema, carcinogen	GW/SW 2.4	7440-43-9	CA
Chromium	inhal/con	0.1	Irritant/carcinogen	GW/SW/SED 258	7440-47-3	CA
Lead	inhal/ing/con	0.05	Insomnia, loss of weight; abdominal pain, paralysis	GW/SED 770	7439-92-1	-
Mercury	inhal/abs/con	0.1	Cough, tremor, insomnia, eye and skin irritation	GW/SW 20	7439-97-6	-
ickel	inhal/ing/con	1.00	Sensitive dermatitis, nasal cavities, pneumonitis, carcinogen	GW 310	7440-02-0	CA
Iron	inhal	10.00	Benign pneumoconiosis	GW/SW/SED 47,600	1309-37-1	-
anganese	inhal/ing	5.00	Parkinson's, insomnia, dry throat, tight chest, low back pain, fatigue	GW/SW/SED 1,350	7439-96-5	-
luminum	inhal/ing	5.00	Dermatosis, pneumoconiosi	SW 400	7429-90-5	-
arium	inhal/ing/con	0.50	Eye, nose irritations, slowed heart rate, CNS - muscle tension	SW/SED 210	7440-39-3	-
obalt	inhal/ing/con	0.10	Pneumoconiosis, vomiting	SW 25	7440-48-4	-
Copper	inhal/ing/con	0.10	Skin, eye irritant and ulceration; lung irritation, vomiting	SW 490	7440-50-8	-
inc	inhal	0.05-1	Dermatitis, fatigue	SW/SED 427	1314-13-2	-
Boron	inhal/ing	1.00	Nasal irritation, eyes, respiratory tract, CNS irritation	SW 80	1303-86-2	-

TABLE B11 (continued)

INORGANIC CONTAMINANTS

	EXPOSURE	Permissible Exposure		Location		
COMPOUND	ROUTE	Limit	TOXICITY	Max.Water Conc. (ppb)	CAS NO.3	C
Calcium (Compounds)	inhal/ing	1-5	Irritant, CNS, respirator tract irritation	SH 22,100	1317-65-3	-
Sodium (salts)	inhal/con/ing	0.05-5	Irritation to eyes, mucous membranes	SH 36,500	1310-73-2	-
Silver	inhal/con/ing	0.01	Nasal septum, skin, eye irritation	SH L.T. 20	7440-22-4	-

See the bottom of Table B10 for the key to abbreviations used in this table.

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PROTECTION AGAINST POTENTIAL HAZARDS TABLE, PAGE 1

Replaced by previous tables No. B10 and B11

PROTECTION AGAINST POTENTIAL HAZARDS

Substance	CAS No.	Environmental Concentration W/units	In Sample (Soil, water, air, waste)	Toxicity			C
				TLV	Route of Exposure	Comments	

PROTECTION AGAINST POTENTIAL HAZARDS TABLE, PAGE 2

PROTECTION AGAINST POTENTIAL HAZARDS
PAGE TWO

Substance	CAS No.	Environmental Concentration W/units	In Sample (Soil, water, air, waste)	Toxicity			C
				TLV	Route of Exposure	Comments	

C	Carcinogenicity
CH	Carcinogenicity established for humans
CA	Carcinogenicity established for animals
CSH	Carcinogenicity suspected for humans
CSA	Carcinogenicity suspected for animals

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RISK ANALYSIS - FOR EACH SITE TASK AND OPERATION

TASK 1 - Groundwater Sampling - A landfill site survey poses risks to workers from exposure to both organic and inorganic contaminants previously identified at the site. Potential exposure to these agents can come from: (1) contaminated soils and airborne dusts generated by worker's walking along the surface, (2) contaminated sediments along Army Creek and Pond disturbed while surveying, and (3) workers may walk in wet areas (low spots where water may accumulate) and be exposed to contaminated standing water or mud, (4) workers may become splashed during groundwater sampling and become exposed to contaminants via routes of inhalation, ingestion, or dermal contact, including eyes and ears. Contaminants on or near the site have been found in groundwater and surface water. The routes of exposure from most contaminants found on-site include (1) dust/vapor inhalation, (2) direct surface contact, (3) absorption through the skin, and (4) ingestion. A summary of routes of exposure, symptoms and TLV's/PEL's limits are presented in Tables 14 and 15 (previous section). Workers carrying out this task are expected to perform field tasks during June-July 1989 when the potential for heat stress is increased.

RISK ANALYSIS - FOR EACH SITE TASK AND OPERATION

(page 2)

Other Risks - Army Creek Site

Other risks to worker's health and safety at the Army Creek landfill site include unstable soil conditions and slope slides from heavy rain events.

A breach in Site Security during these field activities can produce risks to both authrorized workers and any unauthorized persons who enter the site. Unauthorized persons may not have any safety gear protection and may suffer foot, eye or head injuries and be exposed to site contaminants along the creek (sediments) and organic vapors and methane gas from the wells or depressed areas. Use of motor vehicles on site can cause injury to the driver/passengers and site investigation crews.

SITE OPERATIONS

Respiratory and dermal requirements (P.P.E. - personal protection equipment for each of the site tasks and operations to be conducted): The site has been evaluated and it has been determined that Level D. P.P.E. is required for worker protection. Such equipment is outlined on page 21 of this HASP. Prior to the commencement of any task on-site, real time organic vapor and explosion gas monitoring instruments will be used to detect potentially harmful vapors/gases above background levels. Levels higher than background will result in a withdrawal of workers from the area. Personnel working on-site performing sampling tasks will be required to wear rubber gloves, and if dusts are generated, a dust respirator will be worn by all personnel in the area. Eye goggles will also be used.

Selection criteria:

The P.P.E. to be used on-site for tasks is selected based on (1) contaminants detected from previous field studies, and (2) work to be performed under each task. Reference is made to tables 5 through 15 for a list of contaminants and concentrations found at the site. Routes of exposure include inhalation, ingestion, contact and skin absorption. Rubber gloves and boots to be used; tyvek coveralls; safety glasses, hard hat; TLD badge, dust respirator, and escape pack respirator are to be used or, in the case of the respirators, be available to field workers.

Modifications for personal protection requirements: Safety goggles are required during the groundwater sampling task.

Action levels - regarding limitations in tasks assigned, P.P.E. requirements, and/or withdrawal from site:

Since certain field efforst involve some level of excavation and disturbance of the cover, any detection of organic vapor or methane gas above background levels while tasks are being performed, will be a basis for action. If levels are above bk. detected, personnel will stop working and move to an area where background levels are seen. Additional investigative work will be performed to determine the concentration and types of volatile contaminants released before any more work is carried out. Personnel are required to carry and use an escape pack air supplied respirator to evacuate the site if elevated levels are detected. If levels

(See next page)

above background are detected, personnel shall evacuate the area. If high (explosive limit) levels of methane are detected in areas, personnel shall evacuate the area. Monitoring may proceed only after an evaluation of the conditions has been made, and appropirate P.P.E. and/or controls have been selected and provided.

Selection Criteria:
A.D. Little Guidelines for the selection of Chemical Protective clothing. There is a possibility that personnel may come in contract with low level organic solvent type materials and some metals. Personnel will need Butyl rubber boots and gloves.

Modifications of personal protection requirements will be specified if the HSSO determines that conditions have become too dusty. Dusty conditions will require upgrading to Level C, Full Face Air Purifying respirator with dust filters. Detection of organic vapors and/or LEL of 10% will require immediate evacuation from the area and the use of an escape pack respirator. Upon evaluation, Level B dermal protection upgrade and SCBA respiratory portection may be required.

Level A protection should be worn when the highest level of respiratory, skin, eye, and mucous membrance protection is needed.

____ Positive-pressure (pressure-demand), self-contained, breathing apparatus (MSHA/NIOSH approved) (REQUIRED).

____ Fully-encapsulating, chemical-resistant suit (REQUIRED).

____ Chemical resistant inner and outer gloves (REQUIRED). Type? ____

____ Chemical resistant boots with steel toe and shank: (depending on suit boot construction, worn over or under suit boot) (REQUIRED).

____ Thermal Luminescent Detector (TLD) Badge for Radiation (REQUIRED)

____ Personal radiation detector

____ Hard hat (under suit).

____ Coveralls (under suitsj).

____ Two-way radio communications (intrinsically safe) (REQUIRED).

Level B protection should be selected when the highest level of respiratory protection is needed, but a lesser level of skin and eye protection. Level B protection is the minimum level recommended on initial site entries until hazards have been further identified and defined by monitoring, sampling, and other reliable methods of analysis. Personnel equipment corresponding with those findings may then be utilized.

____ Positive-pressure (pressure demand), self-contained, breathing apparatus (MSHA/NIOSH approved) (REQUIRED).

____ Chemical resistant clothing (overalls and long sleeved jacket, coveralls, hooded, two-piece, chemical splash suit, or disposable, chemical-resistant coveralls) (REQUIRED).

____ Coveralls (under splash suit)

____ TLD Badge for Radiation (REQUIRED).

____ Personal radiation detector

____ Chemical resistant inner and outer gloves (REQUIRED). Type ? ____

____ Chemical resistant boots with steel toe and shank (REQUIRED)

____ Two-way radio communications (intrinsically safe)

____ Hard hat

Level C protection should be selected when the type of hazardous airborne substance is known, concentration measured, criteria for using air-purifying respirators met, and skin and eye exposure is unlikely. Monitoring of the air must be performed to comply with OSHA regulations and to ensure respirator effectiveness.

_X_ Full face, air-purifying respirator (MSHA/NIOSH approved) with cartridge type ___ (REQUIRED)
___ Chemical resistant clothing (one-piece coverall; hooded, two-piece, chemical-splash suit, chemical resistant hood and apron, disposable chemical resistant coveralls) (REQUIRED)
___ TLD Badge for Radiation (REQUIRED)
___ Personal radiation detector
_X_ Chemical resistant inner and outer gloves (REQUIRED). Type? Butyl/Nitrile
___ Boots, steel toe and shank, chemical-resistant (REQUIRED)
___ Two-way radio communications (intrinsically safe)
___ Hard hat
___ Escape mask (respirator)

Level D is primarily a work uniform. It should not be worn on any site where respiratory or skin hazards exist.

_X_ Protective coveralls and protective gloves. Type? Tyvek coveralls and Butyl Rubber boots and gloves.
_X_ TLD Badge for Radiation (REQUIRED)*
_X_ Personal radiation detector
_X_ Boots or shoe with steel toe and shank (REQUIRED)
_X_ Hard hat
_X_ Safety eye wear - Glasses & Goggles

* Use of Radiation Detection Instruments while in field.

REQUIRED LEVEL(S) OF PROTECTION

Task	Name	Respiratory	Clothing	Gloves*	Boots	Other Modifications
Team Leader	L. Fiorucci	5 minute escape pack	T	B	B	Dust Respirator for Excavation
Site Safety Officer	L. Fiorucci	5 minute escape pack	T	B	B	Dust Respirator for Excavation
Samplers	C. Yen	5 minute escape pack	T	B	B	Dust Respirator for Excavation
Other	Geologist	5 minute escape pack	T	B	B	Dust Respirator for Excavation
Decon	GFEE	5 minute escape pack	T	B	B	Dust Respirator for Excavation

Tyvek = T
Latex = L
Saranex = S
Butyl = B
Neoprene = N
Viton = V
Covers = C
Other = Other (specify)

* Latex normally used, but Butyl will be used for water sampling tasks.

Operations and Monitoring/Sampling Equipment Checklist

Type of Equipment	Number Needed	Calibrated	Field Ready
Photo Ionization Detector	1		
Explosive Gas Meter/			
Oxygen Meter	1		
Radiation Survey meter	1		

Operating Procedures and Methods for Surveillance:

The site HSSO or designee will accompany field personnel while carrying out each task to monitor background levels for organic vapor and identify any areas where oxygen may be insufficient. Personnel entering test pits will be fitted with a life-line and harness for rapid removal in event of side slope failure or release of toxic materials in the confined space. Test pits will be tested for combustible gases and the presence of sufficient oxygen (> 19.5%) to support life. Ambient air around surface soil samplers and boundary monument installers will be monitored for organic vapor and combustible gases.

Test pit side walls will be sloped according to OSHA regulations 1926.651-.652 Subpart P. Such slopes are determined based on soil characteristics. As appropriate, side wall shoring may be provided. If organic vapor levels are above background in test pit areas, Level B, SCBA respiratory protection will be provided.

Methods of Maintenance and Calibration

All equipment maintenance and calibration efforts shall be conducted by P. J. Lohr at the NUS (GFEE) Harrisburg facility. These efforts shall be performed in accordance with the following NUS health and Safety Standard Operating Procedures

____ No. ME01; Use, Calibration, and Maintenance of the HNU PI-101
X No. ME05; Combustible Gas Indicator
____ No. ME02; Use, Calibration, and Maintenance of the OVA 128
X Other. Specify procedures: Oxygen Meter (combined with Combustible Gas Indictor) Use manufacturers calibration procedures. Calibration work will be documented.
X Radiation Detector
X Photoionization Detector

Perimeter Establishment: at landfill

Site Secured Yes

Map of Contaminated Zones X

Perimeter Identified X

Contaminated Zones Identified X

Exact locations of perimeter to be determined by site survey.

Contaminated zones need to be identified. The contaminant reduction zone and support zones are expected to be near the facility entrance along U.S. Routes 13/301S. Such zones need to be established prior to commencement of any site work.

Site Control Measures:

The following procedures and measures shall be observed to minimize the potentials for contaminant transfer and personnel exposures (i.e., site security, site zonation, etc.). If applicable, attach maps.

All personnel assigned to the site to perform tasks or support field work will be (1) instructed in field procedures applicable to this site, (2) follow CPEE/NUS procedures for sampling water, (3) checked by the HSSO (or designee) for compliance with HASP requirements and P.P.E., (4) Supervised by HSSO/FOL for compliance with proper procedures to decon equipment between sampling events and decon of safety gear in the contaminant reduction zone.

The work areas will be roped off to avoid accidental or intentional entry and subsequent injury. The HSSO and FOL will establish proper barricades for the site (whole) and for each task in the respective areas.

Ionizing Radiation:

Normal background 0.01 to 0.02 mR/hr. If less than 2 mR/hr, continue investigation with caution. If greater than 2 mR/hr, evacuate site. Note: normal background is 10 to 20 counts per minute (CPM).

Site Standard Operating Procedures (SOPs)

The following SOPs shall be observed by all personnel during site activities:

Personnel on-site shall comply with Standard Operating Procedures (SOP's) provided by NUS or GFEE.

TASK 1 - Groundwater Sampling - The site will be visited by GF FOL, Health and Safety Officer, and a Field Q/A and Geologist. It is expected that the Sampling Team will be kept together, and accompanied by the Field Operations Leader and Health and Safety Site Officer. The group provides a means to respond to emergency situations if they occur and should provide a means to anticipate and avoid visible dangers--each warning the other. The GF group will operate and carry a real time organic vapor analyzer and lower explosive limit/oxygen meter for the detection of hazardous materials. Site visitors shall not smoke, eat, or drink while on the site. As indicated in the Task 1 SOP, contaminated sampling tools shall be decontaminated with appropriate cleaners, rinsed: and secured, P.P.E. (gloves, boots) shall be decontaminated along with the tools so that cross contamination can be minimized and potential exposure to workers reduced. After work is completed (daily), the crew shall decontaminate gloves, boots and tools, etc., discard the Tyvek suit and secure safety gear for subsequent work. After exiting the site, all members shall wash face and hands. No hand-to-mouth contact is permitted during site activities. No facial hair is allowed which interferes with mask fit. No flames or open fires will be permitted on-site. No working outdoors will be permitted during electrical storms. All subcontractor personnel will be responsible for employing safe operating procedures and complying with OSHA regulations while conducting field activities. All ARCS health and safety requirements plus the contents of this HASP must be followed by all applicable site workers. Work areas must be partitioned off by some method to combat unauthorized entry. The HSSO will will post an 8½"x14" poster, e.g., similar to the one attached to this HASP, at a comspicuous place visible to all employees at the site.

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Confined Space Entry (CSE) Procedures

Specify the nature of any CSE procedures to be performed, and the procedures and restrictions required to protect the involved personnel.

The CSE procedures will support the field effort to take soil samples from a test pit/trench in the soils borrow area. The nature of this soil sampling may require that a person enter the pit/trench. The confined space entry procedures consist of (1) obtaining an entry permit, (2) describing the specific area and pit, (3) isolating the pit, (4) providing necessary protective equipment (Level D P.P.E. plus safety harness/life line plus extraction A-Frame hoist Unit) and shoring of pit sides, (5) pre-entry atmospheric monitoring, (6) continuous atmospheric monitoring, (7) identification of the entry team (pit man and stand-by , personnel, (8) emergency procedures and first aid equipment location, (9) specific training to enter pit, rescue pit man, and administer first aid. Specific attention will be paid to 29 CFR-1926.650-653 subpart P, that descr excavations, trenching, and shoring. Purging of the confined space will be provided if atmospheric testing indicates: (1) oxygen levels are deficient (<19.5%), (2) LEL measurements are at or greater than 10% LEL, and (3) organic vapor measurements are detected above background. The permit for CS/CE operations is shown as Attachment A. (HS03).

ATTACHMENT A

WORK PERMIT FOR CS/LE OPERATIONS

EXPIRATION DATE: ____________

Location of CS/LE: ______________________________________

Description of task: ______________________________________

Identified Hazards (see checklist): ______________________________________

Personnel Assigned
Name: ______________________ duties: ______________
Name: ______________________ duties: ______________
Name: ______________________ duties: ______________
Name: ______________________ duties: ______________
Name: ______________________ duties: ______________

Special Equipment Required ______________________________________

Special Safety Requirements/Procedures ______________________________________

Initial Atmospheric Tests:

		AT ENTRY	INSIDE DEPTHS 4'	8'	12'	16'	20'
oxygen levels		____	____	____	____	____	____
combustible gas		____	____	____	____	____	____
toxics	____	____	____	____	____	____	____
	____	____	____	____	____	____	____
	____	____	____	____	____	____	____

Level of protection	____	Ventilation Functions	____
Adequate PPE Supply	____	Tools & Equipment Approved	____
Isolation Complete	____	Lighting & Elec. Approved	____
Rescue Equipment	____	Communications	____

Permit Prepared by: ______________________ ____________
Health and Safety Officer Date

Permit Approved by: ______________________ ____________
Office Health and Safety Officer Date

MEDICAL

All subcontracting personnel whose presence is required on site must first be examined by a licensed physician (or under the supervision of a licensed physician) in accordance to OSHA standard 29 CFR 1910.120 and 1910.134. The physician's clearance for site work on the Army Creek Landfill site shall be documented, and reviewed by the OHSS before the individual(s) is (are) permitted to be onsite. NUS personnel, whose work may require their presence in areas where potential exposures to hazardous materials exist, shall participate in the NUS medical monitoring program as specified in the NUS Health and Safety Manual, Subject: Medical Program, No. HS-3.0. All medical examinations performed for NUS personnel and NUS subcontracting personnel for these purposes shall be conducted in accordance with OSHA General Industry standards 29 CFR 1910.120, and 1910.134.

Medical examination contents are at the discretion of the examining physician. However, it may be necessary to identify specific tests for certain sites, based on the requirements of the OHSS in consultation with the company physician and the site Health and Safety Officer. Any site-specific test to be performed for personnel are as identified on page 18 of this Health and Safety Plan.

Special Medical Surveillance Procedures:

Parameters	Action Level	Biological Medium	Test Method

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EMERGENCY REFERENCE
(Post Onsite)

Site: Army Creek Landfill

Project No.: GF 25680.001 , NJC 7101

Emergency Information:	911-Delaware	
Local Resources:	NUS: Park West Two	(412) 788-1040
Office:	GFEE: Village of Cross Keys	(301) 433-8832.
Ambulance (Name):	New Castle	(302) 573-2800
Hospital (Name):	Christiana Hospital	(302) 733-1601
Police (Local or State):	Troup 9*	(302) 571-3075
Fire Department (Name): + Rescue	New Castle	(302) 573-2800
Radio Channel:	Citizens Band	CH-9
Nearest Phone:	Popeye Rest./Exxon	(302) 328-4700/(302) 322-6073
Project Manager:	David Sheridan	(301) 433-8832
Site Health and Safety Officer:	Louis Fiorucci	(301) 433-8832
Alternate Site Health and Safety Officer:	Mitchell Weber	(717) 763-7211
*Notify Intelligence Unit Prior to Start of Work.		(302) 736-5996

Emergency Contacts (Medical and Health):

- Dr. Michael Hodgsen (NUS Consulting Physician - University of Pittsburgh)
  Office: (412) 648-3240/Dr. Julio Rivera - Johns Hopkins (301) 338-3704
- Office Health and Safety Supervisor GFEE - Robin Pepperman
  Office: (717) 763-7211 X456
- Director of Health, Safety, and Training NUS - Richard C. Gerlach
  Office: (412) 788-1080 X390
  Home: (412) 531-8014
- Poison Information Center: New Castle, DE 302-573-2800
- National Response Center (for Environmental Emergency Only): 1-800-424-8802
- Office: NUS: (412) 788-1080; GFEE: (717) 763-7211

Directions to Hospital (Attach Map): Travel North on Route 13/40 to New Churchman's Road, turn left onto New Churchman's Road, watch for signs to Christiana Hospital - 3/4 mile on left - Map Attached.

DELAWARE - 911

American Red Cross

First Aid

EMERGENCY TELEPHONE NUMBERS	
Police	302-323-4411
Fire Department	302-573-2800
Doctor	412-648-3240
Ambulance	302-573-2800
Hospital	302-733-1601
Poison Control Center	302-573-2800

BITES <u>Animal Bites</u> - Thoroughly wash the wound with soap and water. Flush the area with running water and apply a sterile dressing. Immobilize affected part until the victim has been attended by a physician. See that the animal is kept alive and in quarantine. Obtain name and address of the owner of the animal.

<u>Insect Bites</u> - Remove "stinger" if present. Keep affected part down below the level of the heart. Apply ice bag. For minor bites and stings apply soothing lotions, such as calamine.

BURNS AND SCALDS <u>Minor Burns</u> - DO NOT APPLY VASELINE OR GREASE OF ANY KIND. Apply cold water applications until pain subsides. Cover with a dry, sterile gauze dressing. Do not break blisters or remove tissue. Seek medical attention.

<u>Severe Burns</u> - Do not remove adhered particles of clothing. Do not apply ice or immerse in cold water. Do not apply ointment, grease or vaseline. Cover burns with thick sterile dressings. Keep burned feet or legs elevated. Seek medical attention immediately.

<u>Chemical Burns</u> - Wash away the chemical soaked clothing with large amounts of water. Remove victim's chemical soaked clothing. If dry lime, brush away before flushing. Apply sterile dressing and seek medical attention.

CRAMPS <u>Symptoms</u> Cramps in muscles of abdomen and extremities. Heat exhaustion may also be present.

<u>Treatment</u> - Same as for heat exhaustion.

CUTS Apply pressure with sterile gauze dressing, and elevate the area until bleeding stops. Apply a bandage and seek medical attention.

EYES <u>Foreign Objects</u> - Keep the victim from rubbing his his eye. Flush the eye with water. If flushing fails to remove the object, apply a dry, protective dressing and consult a physician.

<u>Chemicals</u> - Flood the eye thoroughly with water for 15 minutes. Cover the eye with a dry pad and seek medical attention.

FAINTING Keep the victim lying down. Loosen tight clothing. If victim vomits, roll him onto his side or turn his head to the side. If necessary wipe out his mouth. Maintain an open airway. Bathe his face gently with cool water. Unless recovery is prompt, seek medical attention.

FRACTURES Deformity of an injured part usually means a fracture. If fracture is suspected, splint the part. DO NOT ATTEMPT TO MOVE INJURED PERSON, seek medical attention immediately.

FROSTBITE <u>Symptoms</u> - Just before frostbite occurs skin may be flushed, then change to white or grayish-yellow. Pain may be felt early then subsides. Blisters may appear, affected part feels very cold and numb.

<u>Treatment</u> - Bring victim indoors, cover the frozen area, provide extra clothing and blankets. Rewarm frozen area quickly by immersion in warm water---NOT HOT WATER. DO NOT RUB THE PART. Seek medical attention immediately.

HEAT EXHAUSTION Caused by exposure to heat - either sun or indoors. <u>Symptoms</u> - Near normal body temperature. Skin is pale and clammy. Profuse sweating, tiredness, weakness, headache, perhaps cramps, nausea, dizziness, and possible fainting.

<u>Treatment</u> - Keep in lying position and raise victim's feet. Loosen clothing, apply cool wet cloths. If conscious, give sips of salt water (1 teaspoon of salt per glass) over a period of one hour. If vomiting occurs, discontinue the salt water. Seek medical attention immediately.

SUNSTROKE <u>Symptoms</u> - Body temperature is high (106 degrees F or higher). Skin is hot, red, and dry. Pulse is rapid and strong. Victim may be unconscious.

<u>Treatment</u> - Keep victim in lying position with head elevated. Remove clothing and repeatedly sponge the bare skin with cool water or rubbing alcohol. Seek medical attention immediately.

POISONING Call the poison control center for instruction on immediate care. If victim becomes unconscious, keep the airway open. If breathing stops give artificial respiration, by mouth to mouth breathing. Call an emergency squad as soon as possible.

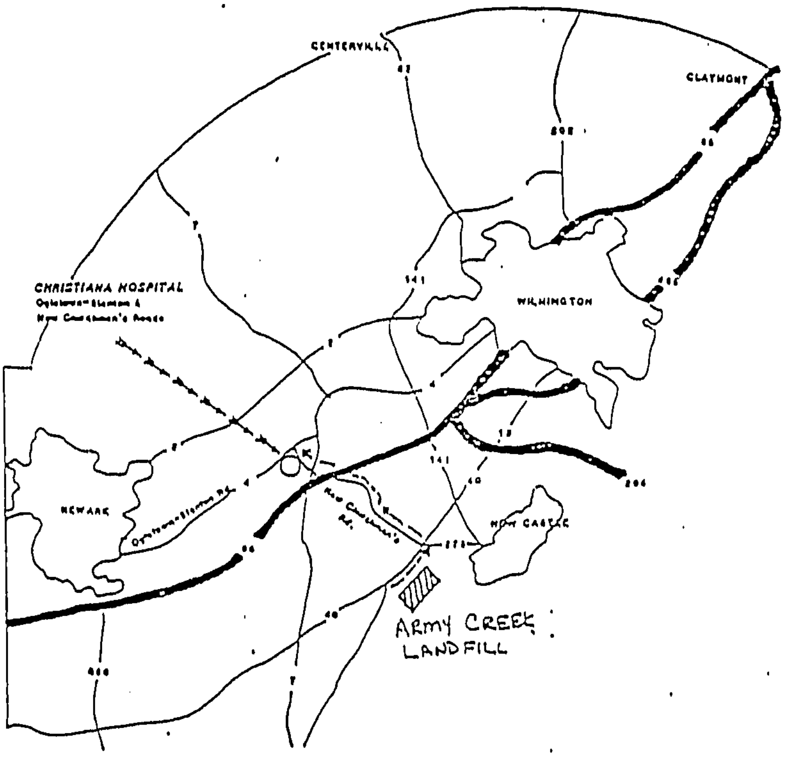
POISON IVY Remove contaminated clothing, wash all exposed areas thoroughly with soap and water followed by rubbing alcohol. If rash is mild, apply calamine or other soothing skin lotion. If a severe reaction occurs, seek medical attention.

PUNCTURE WOUNDS If puncture wound is deeper than skin surface, seek medical attention. Serious infection can arise unless proper treatment is received.

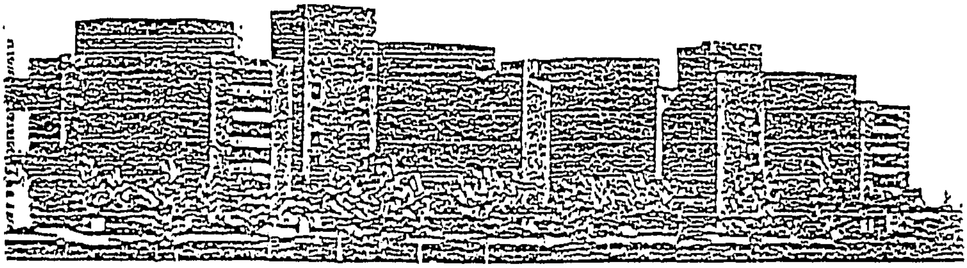
SPRAINS Elevate injured part and apply ice bag or cold packs. DO NOT SOAK IN HOT WATER. If pain and swelling persist, seek medical attention.

UNCONSCIOUSNESS Never attempt to give anything by mouth. Keep victim lying flat, maintain open airway. If victim is not breathing provide artificial respiration by mouth to mouth breathing and call an emergency squad as soon as possible.

DIRECTIONS TO CHRISTIANA HOSPITAL



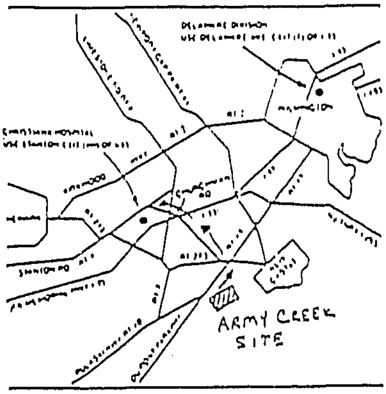
modern hospitals will be just minutes away.



Claymont	20 min.	Newark	11 min.	Salem (NJ)	26 min.
Naamans & 202	21 min.	✈ New Castle	12 min.	Centerville	25 min.
Elkton (Maryland)	18 min.	Middletown	28 min.	Kennett Square (PA)	30 min.
Hockessin	20 min.	Delaware City	19 min	Wilmington	15 min.

stimated times only. Under normal driving conditions

When The Medical Center of Delaware's* new Christiana Hospital opens in Stanton, quality hospital care will be close to home for everyone in this area. The chart above should give ou a pretty good idea in minutes of just how close you will be to truly modern hospital care. The 780-bed Christiana Hospital represents the tate of the art in hospital technology and patient care. The Medical Center* is the state's, and region's only Level One Trauma Center apable of handling any critical illness or injury. uch emergency care is already available at our Delaware Division which will be remodeled uring 1985 and be renamed The Wilmington ospital. Our rebuilding program assures modern facilities and quality hospital care well into the next century. You will see that Christiana ospital is wholly modern, bright and comfortable.



THE MEDICAL CENTER
OF DELAWARE*
We're bringing quality care close to home.
*Formerly The Wilmington Medical Center

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FIRST AID

Emergency Resources   911 DELAWARE

Ambulance	New Castle	(302)573-2800
Hospital (Emergency Room)	Christiana Hospital	(302)733-1601
Fire	New Castle	(302)573-2800
Police*	Troup 9-State Police	(302)571-3075
Poison Control Center	New Castle	(302)655-3389
Airport	Wilmington, DE	(302)323-2680
Explosive Ordinance Disposal Unit**	New Castle Co.	(302)571-7962 or 911
Site Water Supply	Popeye Rest. Exxon	(302)328-4700 (302)322-6073
Site Telephone	-	
Site Radio	Two-Way Radio System	
Site Other	-	

*Call 911 first; ** Contact: Lt. Stanley Yackowski

Emergency Contacts

OHSS-	(Day)	Robin Pepperman	(717)763-7211
	(Other)	Louis Fiorucci	(301)433-8832
Office Physician	(Office)	Michael Hodgson	(412-648-3240
	(~~Beeper~~)	Julio Rivera	(301)338-3704
DHST-	(Day)	Richard Gerlach	(412)788-1080 X390
	(Other)		(412)531-8014
WMSG Physician - Dr. Hodgeson	(Office)		(412) 648-3240

Alternates: Dr. Karpf and Sue Comisky

Hospital: Travel North on Route on 13/40 to New Churchman's Road, turn left onto New Churchman's Road, watch for signs to Christian Hospital: 3/4 mile on left.

Map attached? Yes ✓ No ____

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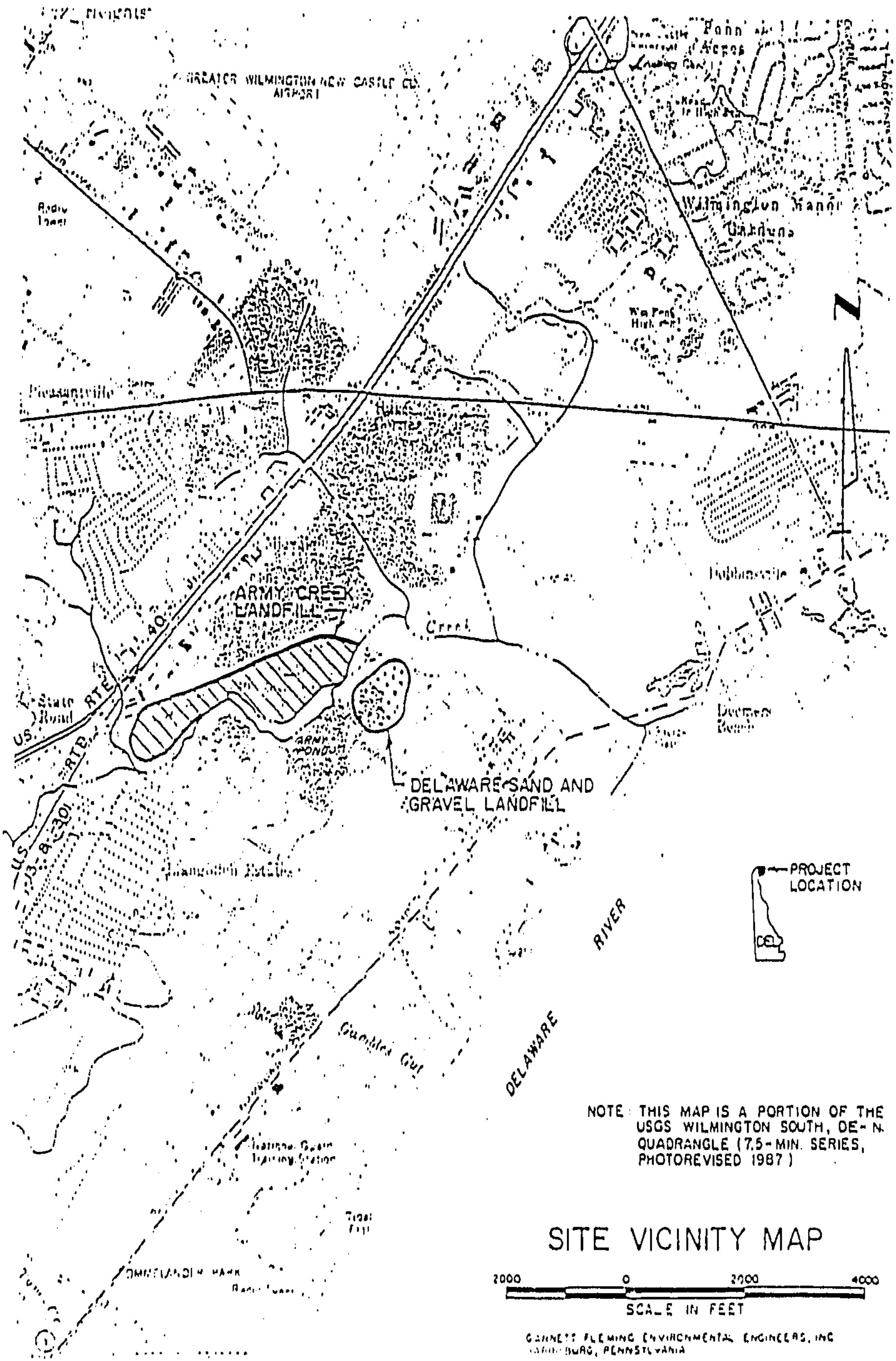
Alternate Emergency Facilities to be noted: Will be determined by Site HSCO prior to the commencement of field operations plan tasks.

Procedures for inclement weather: No excavation during lightning, and rain storms.

Emergency Procedure for Overt Personnel Exposure:

- Skin Contact: Remove contaminated clothing. Wash immediately with water. Use soap if available.
- Inhalation: Remove from contaminated atmosphere. Artificial respiration is necessary. Transport to hospital.
- *Ingestion: Never induce vomiting on an unconscious person. Also never induce vomiting* when acids, alkalis, or petroleum products are suspected. Contact the poison control center.



NOTE: THIS MAP IS A PORTION OF THE USGS WILMINGTON SOUTH, DE-N. QUADRANGLE (7.5-MIN. SERIES, PHOTOREVISED 1987)

SITE VICINITY MAP

GANNETT FLEMING ENVIRONMENTAL ENGINEERS, INC
HARRISBURG, PENNSYLVANIA

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FIGURE

Medical Data Sheet

This form must be completed by all onsite, personnel prior to the commencement of activities, and shall be kept in the site command post during site activities. This form must be delivered to any attending physician when medical assistance is needed.

Site Army Creek Landfill

Name ____________________ Home Telephone (   ) __________

Address ____________________

Age ______ Height ____ Weight ____

Name of next of kin ____________________ Telephone (   ) __________

Drug allergies or other allergies ____________________

Previous Illnesses or Exposures to Hazardous Substances:

Current Medication (prescription and non-prescription):

Medical Restrictions ____________________

Name, address and phone number of personal physician ____________________

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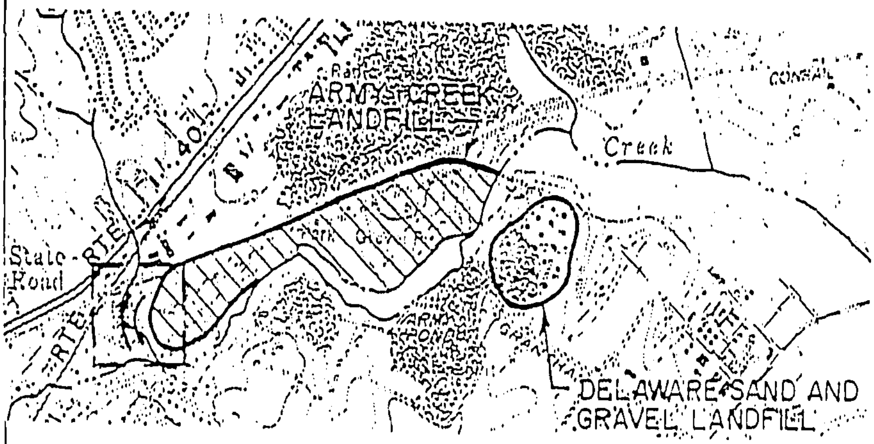
DECONTAMINATION PROCEDURES

Map of site showing restricted access zones, protection levels, decontamination areas, equipment layout, and clean zones enclosed?

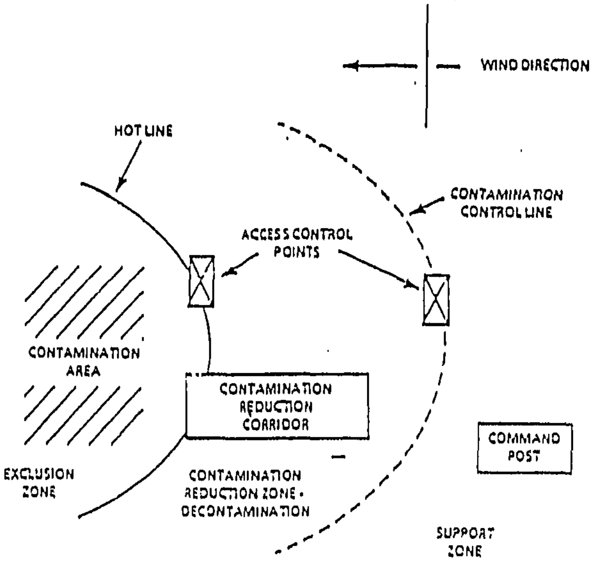
Yes X No ____

If not, sketch below



Explanation: The support zone will be East of the landfill, near Route 13/[illegible] and the site fence line. The CRZ will be a segment located between the Support Zone and the Landfill or Exclusion Zone.

Perimeter Establishment:



The above is a general diagram of a site set-up for work on hazardous waste sites. It shall be established for each work station as the project progresses.

Site Control Measures:

The following procedures and measures shall be observed to minimize the potentials for contaminant transfer and personnel exposures (e.g., site security, site zonation, etc.). If applicable, attach maps.

Personnel shall partition off the immediate area around all work sites with cones and ropes or by some other method, to control the access to these areas.

Decontamination Procedures:

P.P.E. Decontamination involves the wash and rinse of butyl rubber boots and gloves, hard hat and safety glasses. If removed from package and used, escape respirator and Air Purifying Respirators (APR) will be removed, rubber pieces cleaned and returned to packages. The general decontamination process is shown on the following page.

Decontamination of Sampling Bottles and Equipment:

Decontamination of sampling equipment is described in the Project Operations Plan for both inorganic and organic contaminants.

Decontamination Modification (personnel, surfaces, materials, instruments, equipment, etc.):

None are expected at this time, but HSM will make decisions and changes appropriate with changes in types of contaminants encountered.

Disposal Procedures

On-Site:

Tyvek coveralls will be discarded daily and will be placed in double bags and then in covered disposal drums.

Off-Site:

Decontamination procedures are as illustrated below:

WASH AND RINSE OUTER GARMENTS
↓
REMOVE TAPE
↓
REMOVE OUTER GLOVES
↓
REMOVE [illegible] . SUIT
↓
REMOVE BOOT COVERS
↓
REMOVE APR (IF WORN)
↓
REMOVE INNER GLOVES
↓
FULL BODY WASH AS SOON AS POSSIBLE

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EMPLOYEE TRAINING ASSIGNMENTS

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TRAINING CONDUCTED ON SITE

Attendees	Subject-Coverage	Instructor	Date

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EMERGENCY PLAN

Through the course of site activities, potentials for emergency response efforts exist. Pre-emergency planning (such as determining and contacting appropriate offsite emergency response agencies) is the responsibility of the Site Health and Safety Officer. (HSSO) This information is included in the emergency information sections of this Health and Safety Plan. This person is also responsible for determining and documenting the following information.

(A) *Personnel Roles, Lines of Authority and Communication*

Personnel	Responsibilities
Louis Fiorucci - HSSO	Contact Emergency Organizations
Chen Yen - Q/A Officer	Organizing Emergency Response to victim on-site and provide first aid to victims.

(B) *Emergency Recognition and Prevention*

The HSSO will monitor the site work activities by observing and evaluating practices relative to the POP and HASP. In addition the HSSO will monitor the work areas for presence of unsafe conditions, i.e., organic vapor, deficient oxygen, potential explosive mixtures, unsafe physical conditions and unsafe practices.

(C) *Site Evacuation Routes, Procedures, Safe Distances, and Places of Refuge*

Site evacuation routes will be determined prior to site activities described in the Project Operations Plan.

Emergency Procedures

The following standard emergency procedures will be used by on-site personnel. The Site Health and Safety Officer shall be notified of any on-site emergencies and be responsible for ensuring that the appropriate procedures are followed:

1) Personnel Injury: Upon notification of an injury on the Site, the FOL and Site Health and Safety Officer will assess the nature of the injury. If the cause of the injury or loss of the injured person does not affect the performance of site personnel, operations may continue. If the injury increases the risk to others, all site personnel shall move off site for further instructions. Activities on-site will stop until the added risk is removed or minimized.

2) Fire/Explosion: In the event of a fire or explosion on-site, a verbal emergency signal will be given and all affected personnel shall assemble off site. The fire department shall be notified and all personnel moved to a safe distance from the involved area.

3) Personal Protective Equipment Failure: If any site worker experiences a failure or alteration of protective equipment that effects the protection factor, that person shall immediately leave the Site. Re-entry shall not be permitted until the equipment has been repaired or replaced.

4) Other Equipment Failure: If any other equipment on-site fails to operate properly, the Site Manager and Site Health and Safety Officer shall be notified and then determine the effect of this failure on continuing operations on-site. If the failure affects the safety of personnel or prevents completion of the Work Plan tasks, all personnel shall leave the Site until the situation is evaluated and appropriate actions taken.

In all situations, when an on-site emergency results in evacuation of the Site, personnel shall not re-enter until: a) The conditions resulting in the emergency have been corrected, b) The hazards have been reassessed, c) The HASP has been reviewed, d) Project Personnel have been briefed on any changes in the HASP.

(D) Site Security and Control During Emergencies

(E) Emergency Decontamination Procedures

(F) Emergency Altering and Response Procedures

(G) PPE and Emergency Equipment

(H) Emergency Personnel Training Requirements

(I) Emergency Physician Access Plan

(1) MONDAY THROUGH FRIDAY, 8:00 A.M. - 4:00 P.M.

Dial the (412) 648-3240 number. When answered state that:

(a) you are calling from NUS Corporation;

(b) *this is an emergency call.*

Program staff will be alerted how to contact the physician designated to provide emergency coverage on that day. Collect calls will be accepted.

(2) EVENINGS, WEEK-ENDS AND HOLIDAYS:

Dial the (412) 648-3240 number. An operator from the answering service will answer the telephone. *Do the following.*

(a) Tell the operator that you are calling from NUS Corporation.

(b) Tell the operator that this is an emergency call.

(c) Give her your name.

(d) Give her the telephone number where the physician is to call. Be certain that she has written the correct number (area code and seven digits).

(e) If you do not receive a call back within 15 minutes, place a second call to (412) 648-3240.

Collect calls will be accepted.

(3) SITUATIONS WHERE EMPLOYEE REQUIRES IMMEDIATE TRANSPORT TO A HOSPITAL:

If the situation is life-threatening, i.e., cardiac arrest or person not breathing, call the emergency medical services system and transport the person to the nearest hospital with advanced life support capabilities.

- *Report the accident to the Site Safety Officer, and the Office Health and Safety Supervisor*
- Develop safe operating procedures to prevent a recurrence
- File incident report with Manager of Health and Safety Department in Pittsburgh, PA (NUS) and Harrisburg, PA (GFEE).

(J) Site Topography, Layout, and Prevailing Weather Conditions (attach map). Explanation:

____

____

____

____

____

____

(K) Procedures for Contacting Local, State, and Federal Agencies to Report Site Incidents

____

____

____

____

____

____

(L) Employee Alarm System

The following methods will be utilized to notify onsite personnel of the appropriate procedures

Signal	
______________	Work cessation
______________	Onsite emergency situation
______________	Lower background noise to speed communication
______________	Beginning emergency procedures
Other (specify) ______________	______________
______________	______________

(M) Emergency First Aid Procedures (see Page 28)

Other: ______________________________

____

____

____

____

FIELD TEAM REVIEW

Must be signed by each field team member prior to the first site visit.

I have read and understand the contents of this HASP and will comply to its provisions, requirements, and restrictions.

Site ARMY CREEK LANDFILL

Name (PRINT)	Signature	Date

SITE SAFETY FOLLOW UP REPORT

This section must be filled out and returned to the HSSO after each site visit or task.

Site Name: Army Creek Landfill

Person responsible for follow up report: ____________________

Actual date of work: ____________________

Actual Site Investigation Team:

[illegible] Personnel:	Responsibility:

Other:	Purpose:

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PERSONAL PROTECTIVE EQUIPMENT

• Level of Respiratory Protection Used	Activity Performed

• Field Dress	Activity

MONITORING EQUIPMENT

HNU

- Background reading ____________
    - Readings above background? ____________
    - Location of high readings ____________

____

____

Radiation

- Readings above background? _____ Yes _____ No
- If yes, specify where readings were found and what action was taken.

____

____

____

GENERAL SAFETY

Were any safety problems encountered while on site?

Explain: ____

____

____

____

____

____

____

____

ACCIDENT REPORT INFORMATION

Did any team member report:	Yes	No
• Chemical exposure	____	____
• Illness, discomfort, or unusual symptoms	____	____
• Environmental problems (heat, cold, etc.)	____	____

Explain: ____

____

____

____

____

____

____

____

Was an Employee Exposure/Injury Incident Report Completed? ___ Yes ___ No

SITE SAFETY REVIEW - CHANGES AND OVERALL EVALUATION

(To be Completed for Each Field Change in Plan)

Was the Safety Plan Followed as presented? ____ yes ____ no

Describe, in detail, all changes to the Safety Plan:

____

____

____

____

____

Reason for changes: ______________________________

____

____

____

____

Follow-up, Review and Evaluation Prepared by: ____________ Date ________

Discipline ______________________

Approved by: Site Manager ____________________ Date ________

Site Safety Officer ____________________ Date ________

Approved by: Office Health & Safety Supervisor ____________ Date ________

Evaluation of Site Safety Plan

Was the Safety Plan adequate? ____ yes ____ no

What changes would you recommend?

____

____

____

____

HEAT STRESS MONITORING LOG

Date	Name	Weight Change	Pulse Rate	Blood Pressure	Blood Pressure	WBGT

FIRST AID SUPPLY USAGE FORM

Project No.	Date	Item(s) Used	Kit No.

Please submit this form as soon as possible to to the NUS/WMSG Equipment Manager for first aid supply replenishment.

Subject	Number	Page
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EMERGENCY ESCAPE PACK LOG

Site: Army Creek Landfill

Location: New Castle, Delaware

Dates of Investigation:

User	Date of Use	SCBA #	Satisfactory Check-Out (Yes/No - Initials)	Date Cleaned

SCBA Performance Comments:

Site Manager

Date

Return to HSO at Completion of Activity

(ULTRA TWIN) RESPIRATOR LOG

Site: Army Creek Landfill

Location: New Castle, Delaware

Dates of Investigation: ______________________

User	Date of Use	Cleaned and Inspected Prior To Use (Initials)	Cartridges Changed Prior to Use (Yes/No)	Total Hours On Cartridge

______________________ Site Manager

______________________ Date

Return to HSO at Completion of Activity

INCIDENT REPORT

Report No. ______

Site: Army Creek Landfill   Project No. ______

Location: New Castle, Delaware

Date of Report: ______   Preparer's Name: ______

Name and Address of Injured: ______   SSN: ______   Age: ______

______   Sex: ______

Years of Service: ______   Time of Present Job: ______   Title/Classification: ______

Division/Department: ______   Date of Incident: ______   Time: ______

Incident Category:

___ Motor Vehicle

___ Chemical Exposure

___ Property Damage

___ Near Miss

___ Fire

___ Other

Severity of Injury or Illness:

___ Non-disabling

___ Medical Treatment

___ Disabling

___ Fatality

Amount of Damage: $ ______   Property Damage: ______

Estimated Number of Days Away from Job: ______

Nature of Injury or Illness: ______

Classification of Injury:

___ Fractures

___ Dislocations

___ Sprains

___ Abrasions

___ Lacerations

___ Punctures

___ Bites

___ Respiratory Allergy

___ Heat Burns

___ Chemical Burns

___ Radiation Burns

___ Bruises

___ Blisters

___ Toxic Respiratory Exposure

___ Toxic Ingestion

___ Cold Exposure

___ Frostbite

___ Heat Stroke

___ Heat Exhaustion

___ Concussion

___ Faint/Dizziness

___ Toxic Respiratory

___ Dermal Allergy

Part of Body Affected: ______

Degree of Disability: ______

Date Medical Care was Received: ______

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Where Medical Care was Received: ________________________

Address (if off site): ________________________

Incident Location

Causative agent most directly related to accident (object, substance, material, machinery, equipment, conditions): ________________________

Was weather a factor? ________________________

Unsafe mechanical/physical/environmental condition at time of accident (Be specific):

Unsafe act by injured and/or others contributing to the accident (Be specific, must be answered):

Personal factors (improper attitude, lack of knowledge or skill, slow reation, fatigue):

Level of personal protection equipment required in Site Safety Plan:

Modifications: ________________________

Was injured using required equipment: ________________________

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If not, how did actual equipment use differ from plan?

What can be done to prevent a recurrence of this type of accident (modification of machine; *mechanical guards; correct environment; training*)?

Detailed narrative description (how did accident occur, why; objects, equipment tools used, circumstances, assigned duties). Be specific:

(Use back of sheet, as required)

Witnesses to accident:

Signature of Preparer

Signature of Site Manager

Department Appraisal and Recommendation

In your opinion, what actions or equipment contributed to this accident?

Your recommendation:

Date: Signature of Department Manager

FOR HEALTH AND SAFETY USE ONLY

Temporary Total	________	Permanent Partial	________
Death or Permanent Total	________		
Started losing time	________	Part of Body	________
Returned to work	________	Percent loss or loss of use	________
Time charge	________	Time charge	________
Compensation	$________	Medical	$________
Other	$________	total	$________

Name and Address of Hospital ____________________

____

Name and Address of Physician ____________________

____

cc: OHSS
Administrative Manager
DHST
Medical Consultant

INCIDENT FOLLOW-UP

Date of Incident: ____________________

Name: ____________________ Employee No. ____________________

Site: Army Creek Landfill

Brief description of incident: ____________________

Outcome of incident: ____________________

Physician's recommendations: ____________________

Date returned to work: ____________________

ATTACH ANY ADDITIONAL INFORMATION TO THIS FORM

cc: OHSS
Administrative Manager
DHST
Medical Consultant

JOB SAFETY & HEALTH PROTECTION

The Occupational Safety and Health Act of 1970 provides job safety and health protection for workers by promoting safe and healthful working conditions throughout the Nation. Requirements of the Act include the following:

Employers

All employers must furnish to employees employment and a place of employment free from recognized hazards that are causing or are likely to cause death or serious harm to employees. Employers must comply with occupational safety and health standards issued under the Act.

Employees

Employees must comply with all occupational safety and health standards, rules, regulations and orders issued under the Act that apply to their own actions and conduct on the job.

The Occupational Safety and Health Administration (OSHA) of the U.S. Department of Labor has the primary responsibility for administering the Act. OSHA issues occupational safety and health standards, and its Compliance Safety and Health Officers conduct jobsite inspections to help ensure compliance with the Act.

Inspection

The Act requires that a representative of the employer and a representative authorized by the employees be given an opportunity to accompany the OSHA inspector for the purpose of aiding the inspection.

Where there is no authorized employee representative, the OSHA Compliance Officer must consult with a reasonable number of employees concerning safety and health conditions in the workplace.

Complaint

Employees or their representatives have the right to file a complaint with the nearest OSHA office requesting an inspection if they believe unsafe or unhealthful conditions exist in their workplace. OSHA will withhold, on request, names of employees complaining.

The Act provides that employees may not be discharged or discriminated against in any way for filing safety and health complaints or for otherwise exercising their rights under the Act.

Employees who believe they have been discriminated against may file a complaint with their nearest OSHA office within 30 days of the alleged discrimination.

Citation

If upon inspection OSHA believes an employer has violated the Act, a citation alleging such violations will be issued to the employer. Each citation will specify a time period within which the alleged violation must be corrected.

The OSHA citation must be prominently displayed at or near the place of alleged violation for three days, or until it is corrected, whichever is later, to warn employees of dangers that may exist there.

Proposed Penalty

The Act provides for mandatory penalties against employers of up to $1,000 for each serious violation and for optional penalties of up to $1,000 for each nonserious violation. Penalties of up to $1,000 per day may be proposed for failure to correct violations within the proposed time period. Also, any employer who willfully or repeatedly violates the Act may be assessed penalties of up to $10,000 for each such violation.

Criminal penalties are also provided for in the Act. Any willful violation resulting in death of an employee, upon conviction, is punishable by a fine of not more than $10,000, or by imprisonment for not more than six months, or by both. Conviction of an employer after a first conviction doubles these maximum penalties.

Voluntary Activity

While providing penalties for violations, the Act also encourages efforts by labor and management, before an OSHA inspection, to reduce workplace hazards voluntarily and to develop and improve safety and health programs in all workplaces and industries. OSHA's Voluntary Protection Programs recognize outstanding efforts of this nature.

Such voluntary action should initially focus on the identification and elimination of hazards that could cause death, injury, or illness to employees and supervisors. There are many public and private organizations that can provide information and assistance in this effort, if requested. Also, your local OSHA office can provide considerable help and advice on solving safety and health problems or can refer you to other sources for help such as training.

Consultation

Free consultative assistance, without citation or penalty, is available to employers, on request, through OSHA supported programs in most State departments of labor or health.

More Information

Additional information and copies of the Act, specific OSHA safety and health standards, and other applicable regulations may be obtained from your employer or from the nearest OSHA Regional Office in the following locations:

Atlanta, Georgia
Boston, Massachusetts
Chicago, Illinois
Dallas, Texas
Denver, Colorado
Kansas City, Missouri
New York, New York
Philadelphia, Pennsylvania
San Francisco, California
Seattle, Washington

Telephone numbers for these offices, and additional area office locations, are listed in the telephone directory under the United States Department of Labor in the United States Government listing.

Washington, D.C.
1985
OSHA 2203

Bill Brock
William E. Brock, Secretary of Labor

U.S. Department of Labor
Occupational Safety and Health Administration

Under provisions of Title 29, Code of Federal Regulations, Part 1903 [illegible]
in a conspicuous place where notices to [illegible]

D

APPENDIX D

QUALITY ASSURANCE PROGRAM
FOR
AIR MONITORING INSTRUMENTATION

QUALITY ASSURANCE PROGRAM

for

AIR MONITORING INSTRUMENTATION

Introduction

The objective of this Quality Assurance Program is to assure high instrument reliability in situations where airborne contaminants could present a significant threat to health and safety. The accomplishment of this objective depends upon the following:

1. Periodic checks of instrument operation, calibration, and maintenance to provide an indication of unsatisfactory performance.
2. Proper training of personnel involved with instrumentation.

The following manuals and log books are utilized in the operation of the Quality Assurance Program.

1. Operations and Procedures Manual
2. Field Usage Log Book
3. General Information Manual
4. Calibration and Maintenance Log Book

1. Operations and Procedures Manual

This manual sets forth standard operating procedures for air monitoring instrumentation utilized in various types of site investigations. It also defines the responsibilities of personnel involved with the operation and maintenance of this instrumentation. Reliable and efficiently operating instrumentation depends upon:

1. Regular preventive maintenance and calibration.
2. Proficient personnel, trained in the operation of the instrumentation.

All personnel, whether operating, maintaining, calibrating or repairing instruments should be properly trained in the particular task that they are performing. The manual should be consulted regularly to assure that standard procedures are being followed and that all pertinent data are being recorded. If adhered to, this regularly scheduled preventive maintenance and calibration program should assure instrument reliability and reduce unnecessary downtime.

2. Field Usage Log Book

Operational checks are to be performed before each day's use and may include field (secondary) calibrations. The purpose of these checks is to verify to the instrument operator that the instrument is operating properly.

The Field Usage Log Book should be used to record the status of each operational check.

3. General Information Manual

The General Information Manual summarizes all instrument information for quick access. It includes the following:

I. Summary
  - Model
  - Serial Number
  - Gannett Fleming I.D. Number
  - Vendor Information
    - Address
    - Sales Person
    - Phone
  - Purchase Date
  - Purchase Price
  - P.O. Number

II. Purchase Information
  - Purchase Orders
  - Invoices

III. Parts Information
  - Parts List
  - Costs
  - Ordering Procedures

IV. Correspondence

V. Manual

VI. Miscellaneous
  - Warranties
  - Brochures

4. Calibration and Maintenance Log Book

Preventive maintenance checks and primary calibrations are to be performed at periodic intervals (usually monthly) during which the instrument is removed from the field. The purpose of the maintenance checks and calibrations is to assure reliable operation and minimize downtime of instrumentation. All maintenance and calibration data should be recorded in the Preventive Maintenance and Calibration Log Book. In addition, all corrective maintenance required should be recorded on the instrument repair log sheets.

NUS CORPORATION WASTE MANAGEMENT SERVICES GROUP	HEALTH AND SAFETY STANDARD OPERATING PROCEDURES	Number ME01 Page 1 of 11 Effective Date 08/15/87 Revision 2 Applicability WMSG Prepared Health and Safety Group
Subject HNU PI-101 ORGANIC VAPOR METER		Approved R. C. Gerlach, CIH

CONTENTS

Subject HNU PI-101 ORGANIC VAPOR METER	Number ME01	Page 2 of 11
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1.0 PURPOSE

To establish procedures for the use, maintenance, and calibration of the HNU-101 Organic Vapor Meter.

2.0 SCOPE

Applies to each usage of the HNU PI-101 photoionization detector by NUS/WMSG personnel.

3.0 DEFINITIONS

None.

4.0 RESPONSIBILITIES

Office Health and Safety Supervisor (OHSS) - The OHSS shall insure that the user has been appropriately trained and certified in the usage of the HNU instrument. He/she shall also insure that the instrument is properly maintained and calibrated prior to its release for field service.

Instrument User - The user should be personally secure that he/she has been adequately trained and that he understands the operation and limitations of the instrument. He/she is further responsible to insure that the appropriate probe(s) have been selected for compounds to be found on site and that the instrument has been properly calibrated and is working properly.

5.0 PROCEDURES

5.1 Principle of Operation

The HNU System portable photoionizer detects the concentration of many organic gases as well as a few inorganic gases. The basis for detection is the ionization of gaseous species. The incoming gas molecules are subjected to ultraviolet (UV) radiation, which is energetic enough to ionize many gaseous compounds. The molecule is transformed into charged-ion pairs creating a current between two electrodes. Each molecule has a characteristic ionization potential, which is the energy required to remove an electron from the molecule, yielding a positively charged ion and the free electron. The instrument measures this energy level.

5.2 Instrument Configuration

Three probes, each containing a different UV light source, are available for use with the HNU. Probe energies are 9.5, 10.2, and 11.7eV. All three detect many aromatic and large-molecule hydrocarbons. The 10.2 and 11.7eV probes, in addition, detect some smaller organic molecules and some halogenated hydrocarbons. The 10.2eV probe is the most useful for environmental response work, as it is more durable than the 11.7eV probe and detects more compounds than the 9.5eV probe.

5.3 Calibration

The primary HNU calibration gas is benzene (or isobutylene, a benzene equivalent). The span potentiometer knob is turned to 9.8 for benzene calibration. A knob setting of zero increases the sensitivity to benzene approximately tenfold. The instrument's response can be adjusted to give more accurate reading for specific gases and eliminate the necessity for calibration charts.

5.4 Specialized Uses

While the primary use of the HNU is as a qualitative instrument, it can also be used to detect certain contaminants, or at least to narrow the range of possibilities. Noting instrument response to a contaminant source with different probes can eliminate some contaminants from consideration. For instance, a compound's ionizing potential may be such that the 9.5eV probe produces no response, but the 10.2 and 11.7eV probes do elicit a response. Also, HNU does not detect methane or hydrogen cyanide.

5.5 Instrument Advantages

The HNU is easy to use in comparison to many other types of monitoring instrumentation. Its range detection limit is also in the low parts per million range. The response time is rapid; the meter needle reaches 90 percent of the indicated concentration in 3 seconds for benzene. It can be zeroed in a contaminated atmosphere.

5.6 Cautions

The instrument can monitor only certain vapors and gases in air. Nonvolatile liquids, toxic solids, particulates and many other toxic gases and vapors cannot be detected. Because the types of compounds that the HNU can detect is only a fraction of the chemicals possibly present at a field site, a zero reading does not necessarily signify the absence of air contaminants.

The instrument is non-specific, and its response to different compounds is relative to the calibration setting Instrument readings may be higher or lower then the true concentration. This can be an especially serious problem when monitoring for total contaminant concentrations if several different compounds are being detected at once. In addition, the response of this instrument is not linear over the entire detection range. Care must therefore be taken when interpreting the data All identifications should be reported as tentative until they can be confirmed by more precise analysis. Concentrations should be reported in terms of the calibration gas and span potentiometer of gas-select-knob setting

The instrument cannot be used as an indicator for combustible gases or oxygen deficiency

6.0 REFERENCES

1. HNU Systems, Inc. Instruction Manual for Model PI 101 Photoionization Analyzer, 1975

2. E & E FIT Operation and Field Manual: HNU Systems PI 101 Photoionization Detector and Century Systems (Foxboro) Model OVA-128 Organic Vapor Analyzer

3. Personal Communication with Fran Connel, HNU Systems, Inc., January 4, 1984

7.0 ATTACHMENTS

ATTACHMENT A - Start-up and Shutdown Procedure
ATTACHMENT B - Maintenance and Calibration Schedule
ATTACHMENT C - Calibration Procedure
ATTACHMENT D - Cleaning the UV Light Source Window
ATTACHMENT E - Cleaning the Ionization Chamber
ATTACHMENT F - Troubleshooting
ATTACHMENT G - Field Calibration (Memorandum)

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ATTACHMENT A

START-UP AND SHUTDOWN PROCEDURES

Start Up

1. Attach the probe to the readout unit. Match the alignment key, then twist the connector clockwise until a distinct locking is felt.

2. Turn the FUNCTION switch to the battery check position. Check to ensure that the indicator reads within or beyond the green battery arc on the scale plate. If the indicator is below the green arc, or if the red LED comes on, the battery must be charged prior to using.

3. To zero the instrument, turn the FUNCTION switch to the STANDBY position and rotate the ZERO POTENTIOMETER until the meter reads zero. Wait 15-20 seconds to ensure that the zero adjustment is stable. If not, then readjust.

4. Check to see that the SPAN POTENTIOMETER is set at the appropriate setting for the probe being used.

5. Set the FUNCTION switch to the desired ppm range.

6. Listen for the fan operation to verify fan function.

7. Check instrument with an organic point source (such as a magic marker) prior to usage to verify instrument function

Shut Down

1. Turn FUNCTION Switch to OFF.

2. Place the instrument on the charger.

ATTACHMENT B

MAINTENANCE AND CALIBRATION SCHEDULE

Function	Frequency
• Routine Calibration	Prior to each use*
• Factory Check-out and Calibration	Yearly or when malfunctioning
• Wipe Down Read-Out Unit	After each use
• Clean UV Light Source Window	Every month or as use and site conditions dictate*
• Clean the Ionization Chamber	Monthly
• Recharge Battery	After each use

* It is recommended that for activities where the HNU-PI-101 is in the field for extended periods of time that calibration gas be brought into the field to check calibration. However, if necessary, a single calibration conducted prior to an activity will be considered acceptable for periods of use up to three days, after which calibration gas must again be used.

ATTACHMENT C

CALIBRATION PROCEDURE

Calibration Procedure 1

1. Run through start up procedures as per Attachment 1.
2. Fill a sampling bag with HNU calibration gas of known contents.
3. Allow sample bag contents to be drawn into the probe and check response in ppm.
4. If the reading deviates ± 15 percent from the concentration of the calibration gas, the instrument requires maintenance.
5. Each office must develop a mechanism for the documentation of calibration results. This documentation includes:
    a. date inspected
    b. person who calibrated the instrument
    c. the instrument number (Serial # or Other ID #)
    d. the result of the calibration (ppm, probe ev, span pot setting)
    e. identification of the calibration gas (source, type, concentration)

Calibration Procedure 2 (for HNU Calibration Canisters Equipped with a Regulator)

1. Run through start up procedures as per Attachment 1.
2. Connect a sampling hose to the regulator outlet and the other end to the sampling probe of the HNU.
3. Crack the regulator valve.
4. Take reading after 5-10 seconds.
5. If the reading deviates ± 15 percent from the concentration of the calibration gas, the instrument requires maintenance
6. Calibration documentation should be as in #5 above.

ATTACHMENT D

CLEANING THE UV LIGHT SOURCE WINDOW

1. Turn the FUNCTION switch to the OFF position and disconnect the sensor/probe from the Read Out/Control unit.

2. Remove the exhaust screw located near the base of the probe. Grasp the end cap in one hand and the probe shell in the other. Separate the end cap and lamp housing from the shell.

3. Loosen the screws on the top of the end cap and separate the end cap and ion chamber from the lamp housing, taking care that the lamp does not fall out of the lamp housing.

4. Tilt the lamp housing with one hand over the opening such that the lamp slides out of the housing into your hand.

5. The lamp window may now be cleaned with any of the following compounds using lens paper:

   a. HNu Cleaning Compound-All lamps except the 11.7 eV
   b. *Carbon tetrachloride-All lamps except the 11.7 eV*
   c. Methanol-All lamps

6. Following cleaning, reassemble by first sliding the lamp back into the lamp housing. Place the ion chamber on top of the housing making sure the contacts are properly aligned.

7. Place the end cap on top of the ion chamber and replace the two screws. Tighten the screws only enough to seal the O-ring. *Do Not Overtighten.*

8. Line up the pins on the base of the lamp housing with pins inside the probe shell, and side the housing assembly into the shell. It will only fit one way.

ATTACHMENT E

CLEANING THE IONIZATION CHAMBER

1. Turn the FUNCTION switch to the OFF position and disconnect the sensor/probe from the Read Out/Control unit.

2. Remove the exhaust screw located near the base of the probe. Grasp the end cap in one hand and the probe shell in the other. Separate the end cap and lamp housing from the shell

3. Loosen the screws on the top of the end cap and separate the end cap and ion chamber from the lamp housing, taking care that the lamp does not fall out of the lamp housing.

4. The ion chamber may now be cleaned according to the following sequence:

   a. acetone rinse with agitation (10 min.), then dry (preferably with oven at 100°C).

   b. *methanol rinse with agitation (10 min.), then dry (preferably with oven at 100°C).*

5. Place the ion chamber on top of the housing making sure the contacts are properly aligned.

7. Place the end cap on top of the ion chamber and replace the two screws. Tighten the screws only enough to seal the O-ring. Do Not Overtighten.

8. Line up the pins on the base of the lamp housing with pins inside the probe shell, and slide the housing assembly into the shell. It will only fit one way.

ATTACHMENT F

TROUBLESHOOTING

To be performed by qualified technician only.

1. No meter response in any switch position (including BATT CHK)

   A. Broken meter movement

      (1) Tip instrument rapidly from side to side. Meter needle should move freely, and return to zero.

   B. Electrical connection to meter is broken

      (1) Check all wires leading to meter and clean the contacts of quick-disconnects.

   C. Battery is completely dead

      (1) Disconnect battery and check voltage with a volt-ohm meter.

   D. If none of the above solves the problem, consult the factory.

2. Meter responds in BATT CHK position, but reads zero or near zero for all others.

   A. Power Supply defective

      (1) Check power supply voltages per Figure 11 of the HNu owners manual. If any voltage is out of specification, consult the factory.

   B. Input transistor or amplifier has failed

      (1) Rotate zero control; meter should deflect up/down as control is turned.

      (2) Open probe. Both transistors should be fully seated in sockets.

   C. Input signal connection broken in probe or readout.

      (1) Check input connector on printed circuit board. The input connector should be firmly pressed down.

      (2) Check components on back side of printed circuit board. All connections should be solid, and no wires should touch any other object.

      (3) Check all wires in readout for solid connections.

OVA 128

ORGANIC VAPOR ANALYZER

1.0 PURPOSE

To establish guidelines and procedures for the operation, maintenance and calibration of the OVA 128 Organic Vapor Analyzer. General information is included in Section 3.0. Detailed procedures regarding daily operation, maintenance and calibration are contained in Section 4.0.

2.0 RESPONSIBILITIES

The Instrument Operator is responsible for:

- being adequately trained and should understand the limitations of the instrument being used.
- determining that the instrument has been calibrated according to the manufacturer's instructions and is working properly.
- adhering to operational and calibration checks as specified by directions in the operations and procedures manual.

The Equipment Manager is responsible for:

- being sure that all instruments are operating properly and calibrated according to manufacturer's instructions before being tagged for use.
- training all operators in the use of instruments.
- keeping the operations and procedures manual up to date.

3.0 GENERAL INFORMATION

3.1 PRINCIPLE OF OPERATION

The OVA 128 Analyzer is designed to detect and measure hazardous organic vapors and gases and utilizes the principle of hydrogen flame ionization for detection and measurement of organic vapors. The instrument measures organic vapor concentration by producing a response to an unknown sample, which can be related to a gas of known composition to which the instrument has previously been calibrated.

Survey Mode

During normal survey mode operation, a continuous sample is drawn into the probe and transmitted to the detector chamber by an internal pumping system.

The sample stream is metered and passed through particle filters before reaching the detector chamber. Inside the detector chamber, the sample is exposed to a hydrogen flame which ionizes the organic vapors. When most organic vapors burn, they leave positively charged carbon-containing ions. An electric field drives the ions to a collecting electrode. As the positive ions are collected, a current corresponding to the collection rate is generated. This current is measured with a linear electrometer preamplifier which has an output signal proportional to the ionization current. A signal conditioning amplifier is used to amplify the signal from the preamp and to condition it for subsequent meter or external recorder display.

Gas Chromatograph Mode

In the Gas Chromatograph (GC) mode, a small sample of ambient air is injected into a chromatographic column and carried through the column by a stream of hydrogen gas. Contaminants with different chemical structures are retained on the column for different lengths of time (known as retention times) and hence are detected separately by the flame ionization detector. A strip chart recorder can be used to record the retention times, which are then compared to the retention times of a standard with known chemical constituents. The sample can be injected into the column either from the air-sampling hose or directly from a gas-tight syringe.

3.2 CALIBRATION

The OVA is internally calibrated to methane by the manufacturer. When measuring methane, it indicates the true concentration. In response to all other detectable compounds, however, the instrument reading may be higher or lower than the true concentration. Relative response ratios for substances other than methane are available. To interpret the readout correctly, it is necessary either to make calibration charts relating the instrument readings to the true concentrations or to adjust the instrument so that it reads correctly. This is done by turning the 10-turn, gas-select knob, which adjusts the response of the instrument. The knob is normally set at 300 when calibrated to methane. Secondary calibration to another gas is done by sampling a known concentration of the gas and adjusting the gas-select knob until the instrument reading equals the true concentration.

3.3 LIMITATIONS

The OVA has an inherent limitation in that it can detect only organic molecules. Also, it should not be used at temperatures lower than about 40°F because gases condense in the pump and column. It has no temperature control, and since retention times vary with ambient temperatures for a given column, absolute determinations of contaminants are difficult. Despite these limitations, the GC mode can often provide tentative information on the identity of contaminants in air without relying on costly, time-consuming laboratory analysis.

3.4 CAUTIONS

The instrument can monitor only certain vapors and gases in air. Many non-volatile liquids, toxic solids, particulates, and other toxic gases and vapors cannot be detected. Because the types of compounds that the OVA can potentially detect are only a fraction of the chemicals possibly present at an incident, a zero reading does not necessarily signify the absence of air contaminants.

The instrument is non-specific, and its response to different compounds is relative to the calibration setting. Instrument readings may be higher or lower than the true concentrations. This can be an especially serious problem when monitoring for total contaminant concentrations if several different compounds are being detected at once. In addition, the response of this instrument is not linear over the entire detection range. Care must therefore be taken when interpreting the data. All identifications should be reported as tentative until they can be confirmed by more precise analysis. Concentrations should be reported in terms of the calibration gas and span potentiometer or gas-select knob setting.

This instrument cannot be used as an indicator for combustible gases or oxygen deficiency.

4.0 PROCEDURES

4.1 OPERATIONAL CHECKS

Operational checks are to be performed before each day's use, verifying that the instrument is functioning and calibrated properly. The Field Usage Log Book should be used to indicate the status of each operational check.

OVA 128 Organic Vapor Analyzer

- Operational Checks -

1. Connect the probe/readout assembly to the sidepack assembly.

2. Move the INSTR/BATT switch to the BATT position. The meter needle should move to a point beyond the white line.

3. Move the INSTR/BATT switch to the ON position. Allow a 5-minute warmup.

4. For measurements taken as methane equivalent (survey mode), check that GAS SELECT dial is set at 300.

5. Turn the PUMP switch ON. Check flow level on SAMPLE FLOW RATE indicator Record level in Field Usage Log Book. (Ball level significantly below 2 indicates inadequate flow.)

6. Use the CALIBRATE ADJUST knob to set the meter needle to the level desired for activating the alarm. (Usually zero)

7. Turn the VOLUME control fully clockwise.

8. Turn ALARM LEVEL adjust until audible alarm is activated.

9. Open the hydrogen TANK VALUE 1 or 2 turns and observers the reading on the HYDROGEN TANK PRESSURE indicator. Record level in Field Usage Log Book. (Approximately 150 psi of pressure is required for each hour of operation.)

10. Open the HYDROGEN SUPPLY VALVE 1 or 2 turns and observe the reading on the HYDROGEN SUPPLY PRESSURE indicator. Record level in Field Usage Log Book. (Should be between 8 and 12 psi.)

11. After approximately one minute, depress IGNITER BUTTON until hydrogen flame lights. The meter needle will travel upscale. (Do not depress igniter for more than 6 seconds. If flame does not ignite, wait one minute and try again.)

4.2 CALIBRATION

Primary Calibration

1. Remove instrument components from the instrument shell.

2. Turn of Electronics and Zero instrument on X10 scale. Gas select dial to 300.

3. Turn on Pump and Hydrogen. Ignite Flame. Go to Survey Mode.

4. Introduce a Methane Standard Near 100 ppm.

5. Adjust R-32 Trimpot on Circuit Board to make meter read to standard.

6. Turn Off hydrogen flame and adjust meter needle to read 40 ppm (calibrate @ X10) using the calibration adjust knobs.

7. Switch to X100 Scale. The meter should indicate 0.4 on the 1-10 meter markings (0.4 x 100 = 40 ppm). If the reading is off, adjust with R33 Trimpot.

8. Return to X10 Scale and adjust meter needle to 40 ppm with calibration adjust knob if necessary.

9. At the X10 Scale, adjust meter to read 0.4 on the 1-10 meter markings using the calibration adjust. Switch to X1 scale. The meter should read 4 ppm. If the reading is off, adjust using the R-31 Trimpot.

Secondary Calibration

1. Fill an air sampling bag with 100 ppm (Certified) methane calibration gas.

2. Connect the outlet of the air sampling bag to the air sampling line of the OVA.

3. Record the reading obtained off the meter onto the calibration record.

4.3 PUMP SYSTEM CHECK

1. With pump on, hold unit upright and observe flow gauge.

2. Ball level significantly below a reading of 2 is inadequate flow.

3. Check connections at the sample hose.

4. Clean or replace particle filters if flow is impaired or it is time for scheduled service.

5. Reassemble and retest flow.

6. If flow still inadequate, replace pump diaphragm and valves.

7. If flow normal, plug air intake. Pump should slow and stop.

8. If no noticeable change in pump, tighten fittings and retest.

9. If still no change, replace pump diaphragm and valves.

10. Document this function in the maintenance records.

4.4 BURNER CHAMBER CLEANING

1. Remove plastic exhaust port cover.
2. Unscrew exhaust port.
3. Use wire brush to clean burner tip and electrode. Use wood stick to clean teflon.
4. Brush inside of exhaust port.
5. Blow out chamber with a gentle air flow.
6. Reassemble and test unit.
7. Document this function in the maintenance records.

4.5 QUAD RING SERVICE

1. Remove OVA guts from protective shell.
2. Remove clip ring from bottom of valve.
3. Unscrew nut from top of valve.
4. Gently pull valve shaft upward and free of housing.
5. Observe rings for signs of damage-replace as necessary.
6. Lightly grease rings with silicone grease.
7. Reassemble valve-do not pinch rings during shaft insertion.
8. Document this function in the maintenance records.

4.6 HYDROGEN RECHARGING

1. High grade hydrogen (99.999%) is required.
2. Connect the fill hose to the REFILL FITTING on the side Pack Assembly, with the FILL/BLEED valve in the OFF position.
3. Open H_2 supply bottle valve.
4. Place FILL/BLEED valve on fill hose in BLEED position momentarily to purge any air out of the system.

5. Crack the instrument TANK VALVE.

6. Open REFILL valve on instrument.

7. Place FILL/BLEED valve in FILL position until the instrument PRESSURE GAUGE equalizes with the H_2 SUPPLY BOTTLE PRESSURE GAUGE.

8. Shut REFILL valve, FILL/BLEED valve, and H_2 supply bottle valves in quick succession.

9. Turn FILL/BLEED valve to BLEED until hose pressure equalizes to atmospheric pressure.

10. Turn FILL/BLEED valve to FILL Position, then to BLEED position, then to OFF.

11. Close TANK on instrument.

12. Disconnect the FILL HOSE and replace protective nut on the REFILL FITTING.

4.7 PARTICLE FILTER SERVICING

There are two points in the air sampling line of the OVA where filters have been placed to keep particulates from entering the instrument. The first filter is located in the probe assembly, and the second filter (primary filter) is located on the side pack assembly. Cleaning procedures are as follows.

1. Detach the probe assemble from the readout assembly.

2. Disassemble the probe (the components unscrew).

3. The particle filter located within the probe can be cleaned by blowing air through the filter.

4. Reassemble the probe.

5. The primary filter, located behind the sample inlet connector on the side pack assembly is accessed by removing the sample inlet connector with a thin-walled 7/16 inch socket wrench. Remove the filter and clean as above.

6. Reassemble the sample inlet fitting and filter to the side pack assembly.

7. Check sample flow rate.

5.0 MAINTENANCE AND CALIBRATION SCHEDULE

o	Check Particle Filters	Weekly or as needed
o	Check Quad Rings	Monthly or as needed
o	Clean Burner Chamber	Monthly or as needed
o	Secondary Calibration Check	Prior to project start-up
o	Primary Calibration Check	Monthly or if secondary check is off by more than ± 10%
o	Check Pumping System	Prior to project start-up
o	Replace Charcoal	120 hours of use or when background readings are higher with the inject valve down than with the inject valve up in a clean environment.
o	Factory Service	At least annually

* Instruments which are not in service for extended periods of time need not meet the above schedule. However, they must be given a complete check-out prior to their first use addressing the above maintenance items.

6.0 REFERENCES

1. Century Systems (Foxboro). Service Procedures: Organic Vapor Analyzer; 128GC

MSA MICROGARD

PORTABLE ALARM

1.0 PURPOSE

To establish guidelines and procedures for the operation, maintenance and calibration of the MicroGard Portable Alarm. General information is included in Section 3.0. Detailed procedures regarding daily operation, maintenance and calibration are contained in Section 4 0.

2.0 RESPONSIBILITIES

The Instrument Operator is responsible for:

- being adequately trained and should understand the limitations of the instrument being used.
- determining that the instrument has been calibrated according to the manufacturer's instructions and is working properly.
- adhering to operational and calibration checks as specified by directions in the operations and procedures manual.

The Equipment Manager is responsible for:

- being sure that all instruments are operating properly and calibrated according to the manufacturer's instructions before being tagged for use.
- training all operators in the use of instruments.
- keeping the operations and procedures manual up to date.

3.0 GENERAL INFORMATION

3.1 PRINCIPLE OF OPERATION

The MicroGard simultaneously monitors oxygen from 0% to 25% O_2 and combustible gas from 0% to 100% LEL via two separate visual alarms and a joint audible alarm.

Oxygen Indication

The MicroGard is ready to monitor oxygen content in the sampling area once the instrument reading stabilizes at the ambient temperature of operation and calibration is set in fresh air to 20.8%. Actual oxygen content can be continuously or intermittently monitored by using the SELECT keypad to display % Oxygen when monitoring. The

MicroGard has a High Oxygen Alarm factory pre-set at 23% and Low Oxygen Alarm factory pre-set at 19.5%. Once the MicroGard is turned ON, the oxygen alarm functions are always operable in either the % OXY or % LEL display mode.

The oxygen sensor is a galvanic type cell containing dissimilar metal electrodes in a special electrolyte. The cell is sealed with a membrane which allows oxygen to diffuse into the active area. The current generated by the cell is proportional to the oxygen partial pressure in the atmospheric sample passing over the membrane face. The generated current passes through a resistance to provide a voltage input signal for an amplifier. The output of the amplifier drives the oxygen display (through the multiplexer) and also serves as an input to the alarm decoder circuitry.

Combustible Gas Indication

After setting the combustible sensor ZERO in combustible - free air, the MicroGard is ready to monitor the presence of combustible gas in the sampling area. As long as the MicroGard is turned ON, actual combustible gas content in the sampled area is always being monitored and can be displayed continuously or intermittently by using the SELECT keypad to display the % LEL. The combustible alarm is active in both display modes and is factory pre-set at:

- 25% LEL for the high combustible alarm
- -10% LEL for the combustible sensor zero fault alarm.

The flammable properties of combustible gases are used as a basis of detection. The sensor consists of a pair of pelletized filaments called "Pelements™" arranged in an electrically balanced bridge circuit. The detector Pelement is treated with a special catalyst which causes the combustible gases to combine with oxygen at a much lower temperature than that required for normal burning. The inactive compensator Pelement is also exposed to the sample and acts to offset any changes due to flow conditions, sample temperature, pressure and/or humidity.

Combustible gases in the sample combine with oxygen at the surface of the catalyzed detector Pelement. Heat is liberated by this chemical reaction, increasing the temperature and the electrical resistance of the Pelement. This, in turn, unbalances the bridge circuit, thereby providing a voltage signal. This signal is applied to an amplifier which drives the combustible gas display (through the multiplexer) and also serves as an input to the LEL alarm decoder circuit.

3.2 CALIBRATION

The MicroGard should be calibrated with known concentrations of oxygen and pentane or methane each day before use. The instrument should stabilize for 15 minutes before calibration. Oxygen is calibrated in fresh air by adjusting the OXY S control to obtain a 20.8% reading. The combustible indicator is calibrated in two steps. First, in fresh air, the LEL Z control is adjusted to obtain a zero reading. Second, a reading of between 47% and 55% should be observed while passing a mixture of pentane in air (.75%) through the instrument. If the calibration check is not within the recommended range, the display is set to 50% by adjusting the LEL S control.

3.3 CAUTIONS AND LIMITATIONS

It is important that the instrument response be appraised by someone skilled or experienced in properly interpreting the instrument readings with respect to particular conditions, on-going operations and safe practices. For example, an atmosphere that shows no flammability hazard can still be toxic to workmen. Also, a tank or vessel which is safe before work is started may be rendered unsafe by work activities causing a temperature increase, or by stirring or handling bottom sludge in tanks.

The MicroGard Portable Alarm will respond only to those concentrations of gases or vapors which are diffused into the combustible sensor. If the combustible is a high boiling point solvent or high flash point liquid, and is tested at normal ambient temperature, a relatively low vapor concentration will be shown by the instrument. Flashpoint of a liquid is the minimum temperature at which it gives off sufficient vapor to form an ignitable mixture with the air near the surface of the liquid or within the vessel used. If a container holding such high flash point solvents is subsequently heated as by welding and soldering, or by sunlight, it is to be expected that the vapor concentration will increase, and thus the atmosphere of a vessel which was originally shown to contain only a low concentration of vapors may be rendered EXPLOSIVE.

If an attempt is made to use the MicroGard Portable Alarm for testing atmospheres contaminated with high boiling point or high flash point solvents, it is imperative that the actual boiling point or flash point of the contaminants in question be assessed against ambient temperature of the tested space.

The MicroGard is designed to measure combustible gas or vapor content in air. It will not indicate the combustible gas content in an inert gas background, furnace stack, in a reducing atmosphere or in atmospheres containing less than 10% oxygen. Further, this instrument should not be used where the oxygen concentration exceeds that of fresh air (oxygen enriched atmosphere).

Recharging must be done in a non-hazardous location, know to be free of combustible gases or vapors.

Certain materials such as silicone, silicates (such as in certain hydraulic fluids) and organic lead (such as in leaded gasoline) tend to poison the combustible gas sensor thereby giving erroneously low readings. Calibration checks should be made frequently if such materials are suspected to be present in the tested atmosphere.

The combustible gas indicator detects only combustible gases (and vapors) in the air. It will not indicate the presence of combustible airborne mists or dusts such as lubricating oils, coal dust or grain dust.

Pressurized or low pressure samples will give erroneous oxygen percent readings. For atmospheric sampling at high or lower altitudes, the oxygen system should be calibrated at the elevation where sampling is to take place.

Acid gases, such as carbon dioxide, will shorten the service life of the oxygen sensor.

When sampling with accessory sampling lines, the shortest possible length of sampling line should be used to minimize the number of times the aspirator bulb must be squeezed to obtain a valid indication.

When sampling over liquids, take care that the end of the sampling line does not touch the surface of liquid.

Do not push on center of oxygen sensor; otherwise, damage to the sensor may result.

Calibrations should be performed before each day's use to ensure that the instrument operation is dependable and its indications are accurate. A calibration check should be included as a routine inspection of the instrument. Use the MSA Calibration Kit which has been specifically designed for the calibration of this unit.

Obstruction of the sensor cover slots or dust screens in the case will cause erroneous readings. These slots must be kept open (clean).

Combustible gas readings, either negative or greater than 100% LEL*, may indicate an explosive concentration of gas beyond the accurate response range of the combustible gas sensor.

Do not use MSA Inhibitor Filters (Part No. 47740) with this instrument. Loss of sensitivity may result.

Use only genuine MSA replacement parts when performing any maintenance procedures provided in this manual. Failure to do so may seriously impair instrument performance. Repair or alteration of the MicroGard, beyond the scope of these maintenance instructions or by anyone other than a certified MSA serviceman, could cause the product to fail to perform as designed and persons who rely on this product for their safety could sustain severe bodily injury or death.

Any substitution of components may impair intrinsic safety.

The minimum and maximum calibrating and operating temperatures are 0°F (-18°C) and 122°F (50°C), respectively. The oxygen response time increases in temperatures beyond the compensated range, particularly at temperatures below 32°F (0°C); therefore, allow extra time to obtain accurate calibrations and measurement.

Use down to 0°F (-18°C) is possible when calibrated at the temperature and if more sampling time is allowed for slower sensor response (approximately 3 minutes without a sample lime).

4.0 PROCEDURES

4.1 OPERATIONAL CHECKS

Operational checks are to be performed before each day's use, verifying that the instrument is functioning and calibrated properly. The Field Usage Log Book should be used to indicate the status of each operational check.

MSA MicroGard Portable Alarm
- Operational Checks -

1. Press the ON/OFF button to turn the instrument ON. Verify that the display functions and does not indicate "BAT".

2. Verify that Combustible Gas Alarm Light is flashing and alarm is sounding.

3. Press RESET keypad. Wait for instrument to stabilize and record % LEL reading. If display does not indicate 000% LEL, adjust the COMB Z (zero) control to obtain a correct zero reading.

4. Press SELECT keypad. Record % OXY reading. If display does not indicate 20.8% OXY, adjust the OXY S (span) control to obtain the correct span reading.

5. Perform a calibration check according to the procedure in Section 5.2. Record the calibration gas concentration, the BEFORE reading if no adjustment is required and the

BEFORE and AFTER readings if a calibration adjustment is necessary

6. When sampling is completed for the day, record the approximate number of hours of usage, press BATT VOLTS keypad and record voltage and check sensor dust screen for possible blockage. (Clean according to the procedure in ____________________.)

4.2 CALIBRATION PROCEDURES

Combustible Gas

1. In fresh air, with the instrument "warmed up", press the SELECT keypad until % LEL is displayed.

2. Adjust the LEL Z control until a zero reading is obtained.

3. Position sampling/calibration adapter over the sensor openings at the top of the MicroGard. NOTE: Do NOT have aspirator bulb and tubing attached to the adapter.

4. Attach the flow control to the calibration gas tank.

5. Attach the tubing furnished with the Calibration Kit between the flow control and the sampling/calibration adapter connection.

6. Open the flow control valve on the gas tank to pass the gas through the MicroGard.

   - As the % LEL reading increases, note the reading at which the combustible alarm activates. This point is factory-set at 25% LEL.

   - When the combustible display stabilizes, the reading for .75% pentane-in-air should be between 47% and 55%.

7. If the calibration check reading is not within 47% and 55%, set the display reading to 50% by adjusting the LEL S control located under the calibration cover.

8. Close the flow control valve.

9. Remove the sampling/calibration adapter from the MicroGard.

10. Recheck the zero reading in fresh air and repeat steps 1 through 9 if fresh air reading is not equal to zero.

11. Remove the flow control from the calibration gas tank.

5.0 MAINTENANCE AND CALIBRATION SCHEDULE

	Function	Frequency
o	Calibrate	Daily
o	Clean Case	As Needed
o	Clean Sensor Screens	As Needed
o	Charge Battery	Daily

6.0 REFERENCES

MSA. Preliminary Instruction Manual: MicroGard Portable Alarm; 1987.

PHOTOVAC

TIP II PHOTOIONIZATION DETECTOR

1.0 PURPOSE

To establish guidelines and procedures for the operation, maintenance and calibration of the TIP II Analyzer. General information is included in Section 3.0. Detailed procedures regarding daily operation, maintenance and calibration are contained in Section 4.0.

2.0 RESPONSIBILITIES

The Instrument Operator is responsible for:

- being adequately trained and should understand the limitations of the instrument being used.
- determining that the instrument has been calibrated according to the manufacturer's instructions and is working properly.
- adhering to operational and calibration checks as specified by directions in the operations and procedures manual.

The Equipment Manager is responsible for:

- being sure that all instruments are operating properly and calibrated according to manufacturer's instructions before being tagged for use.
- training all operators in the use of instruments.
- keeping the operations and procedures manual up to date.

3.0 GENERAL INFORMATION

3.1 PRINCIPLE OF OPERATION

The TIP photoionization detector continuously monitors gas or vapor concentrations in air. A small pump draws sample air into an ionization chamber which contains a miniature lamp and two electrodes. One electrode is connected to an electrometer. The chamber is flooded with light and an electric voltage is applied across the electrodes. As the pump draws gases into the chamber, ions are formed and an electric current is set up and measured by the electrometer.

The standard ultraviolet lamp used in TIP II has an energy of about 10 6 electron volts (eV) and any molecule having an ionization potential below this value can be sensed.

Most of the light permanent gases (such as the air gases, hydrogen, helium, etc.) have ionization potentials at 12 eV or more. On the other hand, a huge number of organic chemicals which enter the air as gases or vapors, have ionization potentials below 10.5 eV. Included among this latter group are the vast majority of those compounds described as "Pollutants".

The various concentrations of pollutants are ionized, measured by the electrometer, converted to parts per million and read on the liquid crystal display on the face of the instrument. The TIP II can be used either qualitatively or as a direct reading instrument if it is calibrated with a span kit.

3.2 CALIBRATION

If used qualitatively, the only calibration required is to set the span control at a mid-range setting and zero the instrument in ambient air. As a direct reading instrument, TIP II must be calibrated with a known concentration of span gas. Also, for critical applications or if outdoor air is too heavily contaminated, bottled zero air should be used.

3.3 CAUTIONS AND LIMITATIONS

The TIP II does not distinguish between different pollutants. The signal produced represents a composite of all ionizable pollutants. A gas chromatograph is necessary to further distinguish pollutants. The instrument also does not detect methane.

The TIP II will not detect a compound if the compound's ionization potential is greater than the instrument probes energy level.

4.0 PROCEDURES

4.1 OPERATIONAL CHECKS

Operational checks are to be performed before each day's use, verifying that the instrument is functioning and calibrated properly. The Field Usage Log Book should be used to indicate the status of each operational check.

TIP II Photoionization Detector

- Operational Checks -

Qualitative

1. Press POWER switch to turn on TIP II. (A period of up to 2 minutes may be required for pump to start.)

2. Unlock ZERO and SPAN controls by turning locking rings <u>clockwise</u>.

3. Set SPAN control to 5.

4 Lock SPAN control by turning locking ring <u>counterclockwise</u>.

5. Allow TIP II to sample clean air.

6. Adjust ZERO control until liquid crystal display reads 0.00.

7. Lock ZERO control by turning locking ring <u>counterclockwise</u> - confirm that zero reading is unchanged.

8. Record approximate ZERO setting.

<u>Direct Reading</u>

1. Press POWER switch to turn on TIP II.

2. Unlock ZERO and SPAN controls by turning locking rings <u>clockwise</u>.

3. Set SPAN control to 5.

4. Allow TIP II to sample clean air.

5. Adjust ZERO control until LCD reads 0.00.

6. Connect bag of Span Gas to TIP II inlet. (See Section 4.2 for instructions on how to use the span kit.)

7 Adjust SPAN control until LCD indicates the Span Gas concentration (nominal -- 100 ppm isobutylene [2-methyl-1-propene]. Disconnect Span Gas Bag.

8. Sample clean air again and readjust ZERO control until LCD reads 0.00, if necessary.

9. Lock ZERO control by turning locking ring <u>counterclockwise</u>.

10. Record approximate ZERO setting.

11. Sample Span Gas again and readjust SPAN control until LCD indicates the Span Gas concentration, if necessary.

12. Lock SPAN control by turning locking ring <u>counterclockwise</u>. Disconnect Span Gas Bag.

13. Record approximate SPAN setting.

4.2 PHOTOVAC TA103 SPAN KIT

1. Hand tighten regulator onto Span Gas tank.
2. Turn gas bag valve counterclockwise to open.
3. Hand tighten gas bag adapter nut onto regulator.
4. Turn regulator knob counterclockwise about one-half turn to start gas flow.
5. Fill bag about one-half full.
6. Turn regulator knob fully clockwise to shut off gas flow.
7. Turn gas bag valve fully clockwise to close.
8. Remove gas bag adapter nut from regulator.
9. Remove regulator from Span Gas tank.

4.3 BATTERY CHARGING

Charge TIP II battery when "LOBAT" appears at the top left of the LCD.

1. Switch off TIP II.
2. Remove any external devices connected to the rear receptacle, or unscrew dust cover.
3. Set switch on charger to correct AC mains voltage.
4. Connect charger plug to TIP II rear receptacle. Turn knurled collar clockwise to secure plug.
5. Plug charger into AC mains.
6. Allow TIP II to charge for 16 hours.
7. Remove charger plug from TIP II and replace dust cover.

TIP II handle and the charger may be warm to the touch during charging. This is normal.

By letting TIP II fully discharge until "LOBAT" appears, then charging for 16 hours, the operating time of TIP II is maximized. Occasional over-charging for up to two days will not be detrimental to the batteries. Do not leave TIP II on charge continuously when not in use, as this will reduce battery life.

If TIP II is consistently put on charge before "LOBAT" appears, the operating time of TIP II is diminished.

5.0 MAINTENANCE AND CALIBRATION SCHEDULE

Function	Frequency
Calibrate	Daily
Charge Battery	Daily
Clean U V. Lamp Window	
Replace Dust Filter	
Adjust Tuning	

6.0 REFERENCES

PHOTOVAC Incorporated User's Manual: TIP II; Version 2.1, October, 1986.

COMBUSTIBLE GAS
AND OXYGEN ALARM

1.0 PURPOSE

To establish guidelines and procedures for the operation, maintenance and calibration of the MSA Combustible Gas and Oxygen Alarm. General information is included in Section 3.0. Detailed procedures regarding daily operation, maintenance and calibration are contained in section 4.0.

2.0 RESPONSIBILITIES

The Instrument Operator is responsible for:

- being adequately trained and should understand the limitations of the instrument being used.
- determining that the instrument has been calibrated according to the manufacturer's instructions and is working properly.
- adhering to operational and calibration checks as specified by directions in the operations and procedures manual.

The Equipment Manager is responsible for:

- being sure that all instruments are operating properly and calibrated according to manufacturer's instructions before being tagged for use.
- training all operators in the use of instruments.
- keeping the operations and procedures manual up to date.

3.0 GENERAL INFORMATION

3.1 PRINCIPLE OF OPERATION

The MSA Model 260 Combustible Gas and Oxygen Alarm is a hand carried, battery operated instrument. It is designed to sample atmospheres for combustible gases or vapors and oxygen content and warn the user when pre-determined concentrations of either are reached.

The Model 260 contains two completely separate sensors in a single housing and uses a common flow system. A small pump pulls the atmospheric sample through a manifold block in which the oxygen and combustible gas sensors are mounted. Oxygen and combustible gas concentrations are shown on separate meters located on the face of the instrument along with alarm lights. An audible alarm is activated in conjunction with either of the alarm lights.

The oxygen analyzer is a galvanic type cell containing dissimilar metal electrodes in a special electrolyte. The cell is sealed with a membrane which allows oxygen to diffuse into the active area. The current generated by the proportional to the oxygen partial pressure in the atmospheric sample passing over the face of the membrane. The generated current passes through a resistance to provide a voltage input signal for an amplifier. The output of the amplifier drives the oxygen indicating meter and also serves as an input to the alarm comparator circuity.

The flammable properties of combustible gases are used as a basis of detection for the combustible gas indicator. The sensor consists of a pair of pelletized filaments called "pelements" arranged in an electrically balanced bridge circuit. The detector pelement is treated with a special catalyst. The catalyst causes the combustible gases to combine with oxygen at much lower temperatures than would be required for normal burning. The inactive compensator pelement is also exposed to the sample flow and acts to offset any electrical changes caused by flow conditions, sample temperature, pressure and/or humidity.

Combustible gases in the sample combine with oxygen at the surface of the catalyzed detector pelement. Heat is liberated by this chemical reaction increasing the temperature of this pelement causing an associated increase in the pelement electrial resistance.

Increased resistance of the detector pelement unbalances the bridge causing a voltage change in the mid-point connection between the detector pelement and compensating pelement. This voltage signal is applied to an amplifier which drives the combustible gas indicating meter and provides an input for an alarm comparator circuit.

3.2 CALIBRATION

The Model 260 Combustible Gas Indicator is normally calibrated on pentane as being representative of the flammability characteristics of most commonly encountered combustible gases. The meter scale is calibrated from zero the 100% L.E.L. which corresponds to actual volume concentrations of 0 to approximately 1.4% pentane in air. A booklet of response curves is supplied with the Model 260. These curves may be used to interpret meter readings when sampling combustible gases other than pentane. Calibration for combustibles other than pentane may be made on special order.

3.3 CAUTIONS AND LIMITATIONS

Prior to testing potentially dangerous atmospheres with the MSA Model 260 Combustible Gas and Oxygen Alarm the user should be familiar with the first five sections of the manufacturer's instruction manual.

Cautions and limitations associated with the use of the Model 260 are as follows:

1. The Model 260 is designed to measure combustible gas or vapor content in air. It will not indicate the combustible gas content in an inert gas background, furnace stack or in a reducing atmosphere. Further, this instrument should not be used where the oxygen concentration exceeds that of fresh air (oxygen enriched atmospheres).

2. Proper readings are obtained only when the battery has a sufficient level of charge.
   a. The battery charge level should be checked occasionally throughout a testing period.
   b. Upon receiving a new Model 260, it is recommended that the battery be charged for at least 16 hours.
   c. As a regular monthly maintenance item for optimum battery service, the Model 260 should be run for 8 to 10 hours and then fully charged for 24 to 36 hours.
   d. After each day of use or if the indicator has not been used for more than 7 days, the battery should be charged for a minimum of 16 hours.
   e. Do not operate Model 260 while it is charging.
   f. Recharging must be done in a non-hazardous location, known to be free of combustible gases or vapors.

3. Certain materials such as silicone, silicates and organic lead compounds tend to posion the pelement catalyst thereby giving erroneously low readings. Calibration checks should be made frequently if such materials are suspected to be present in the tested atmosphere. (See Section III for more detail.)

4. The combustible gas indicator detects only combustible gases and vapors in air. It will not indicate the presence of combustible airborne mists or dusts such as lubricating oils, coal dust, or grain dust.

5. Pressurized or low pressure samples will give erroneous oxygen percent readings. For atmospheric sampling at higher or lower altitudes the instrument oxygen meter should be calibrated at the elevation where sampling is to take place.

6. Acid gases, such as carbon dioxide will shorten the service life of the oxygen sensor.

7. The oxygen sensor is packaged separately in an inert atmosphere. It must be installed before the Model 260 can be used.

8. When sampling with accessory sampling lines, a 50 foot sample line will increase the initial response time of the Model 260 to approximately 30 seconds and the final response to approximately 3 minutes. Two 50 foot lines connected in series will increase the response times to 60

seconds and 6 minutes respectively. Lines over 100 feet in length are not recommended.

4.0 PROCEDURES

4.1 OPERATIONAL CHECKS

Operational checks are to be performed before each day's use, verifying that the instrument is functioning and calibrated properly. The Field Usage Log Book should be used to indicate the status of each operational check.

MSA Combustible Gas
and Oxygen Alarm
- Operational Checks -

1. Turn the center ON-OFF control to the far right HORN-OFF position. (If the audible alarm is needed during the survey, stop the control momentarily in the ON position to verify operation of the horn.) Both meter pointers will move and one or both alarm lights may light.

2. Adjust the CALIBRATE O_2 (<19.5% or >25%) or ZERO LEL (>50%) to activate and verify operation of the alarms. (Only if they do not light in Step 1.)

3. The % oxygen meter should be set to 20.8% by using the CALIBRATE O_2 control.

4. The % LEL meter pointer should be set to zero by adjusting the ZERO LEL control.

5. If either of the alarm lights are lighted press the Alarm Reset button.

6. Place a finger over the sample inlet fitting or the end of the sample line probe. Observe that the flow indicator float drops out of sight indicating no flow and a leak free system. If the float does not drop, check out the flow system for leaks as described in the MSA 260 instrument manual.

7. Press the CHECK button and observe the % LEL meter. The pointer must read at 80% LEL or higher as marked by the BATTERY zone on the meter. If the pointer reading is less, the batteries must be recharged. No tests should be attempted as the instrument will not perform properly.

8. If it is desired that the audible alarm sound for combustible gas or low oxygen concentrations, turn the center ON-OFF control back one position to the ON setting.

9. Perform a calibration check as described in Section 4.2. Do not adjust the span control unless the correct range of 47 to 53% cannot be obtained.

4.2 CALIBRATION PROCEDURES

Before the calibration of the combustible gas indicator can be checked, the Model 260 must be in operating condition as described in the OPERATIONAL CHECKS, Section 4.1. Calibration check-adjustment is made as follows:

1. Attach the flow control to the recommended calibration gas tank.
2. Connect the adapter-hose to the flow control.
3. Open flow control valve.
4. Connect the adapter-hose fitting to the inlet of the instrument; after about 15 seconds the L.E.L. meter pointer should be stable and within the 47 to 53% range. If the meter pointer is not in the correct range, stop the flow, remove the right hand side cover. Turn on the flow and adjust the "S" control with a small screwdriver to obtain a reading as specified on the calibration sheet.
5. Disconnect the adapter-hose fitting from the instrument.
6. Close the flow control valve.
7. Remove the adapter-hose from the flow control.
8. Remove the flow control from the calibration gas tank.

5.0 MAINTENANCE AND CALIBRATION SCHEDULE

6.0 REFERENCES

LUDLUM
MODEL 3
SURVEY METER

1.0 PURPOSE

2.0 RESPONSIBILITIES

3.0 GENERAL INFORMATION

  3.1 PRINCIPLE OF OPERATION

  3.2 CALIBRATION

4.0 PROCEDURES

5.0 MAINTENANCE AND CALIBRATION SCHEDULE

6.0 REFERENCES

1.0 PURPOSE

To establish guidelines and procedures for the operation, maintenance and calibration of the Ludlum Model 3 Survey Meter. General information is included in Section 3.0. Detailed procedures regarding daily operation, maintenance and calibration are contained in Section 4.0.

2.0 RESPONSIBILITIES

The Instrument Operator is responsible for:

- being adequately trained and should understand the limitations of the instrument being used.
- determining that the instrument has been calibrated according to the manufacturer's instruction's and is working properly.
- adhering to operational and calibration checks as specified by directions in the operations and procedures manual.

The Equipment Manager is responsible for:

- being sure that all instruments are operating properly and calibrated according to manufacturer's instructions before being tagged for use.
- training all operators in the use of instruments.
- keeping the operations and procedures manual up to date.

3.0 GENERAL INFORMATION

3.1 Principle of Operation

The Model 3 is a portable survey instrument that provides four linear ranges from 0-200 mR/hr.

Operating features of the instrument include a unimorph speaker mounted to the instrument can with an audio ON-OFF capability, fast-slow meter response, meter reset button and a 6-position switch for selecting battery check or scale multiples of X0.1, X1, X10 and X100. Each range multiplier has its own calibration potentiometer.

Any G-M probe offered by the company will operate on this unit as well as many of the scintillator-type detectors. The instrument is set for 900-volt, G-M tube operation. For special requirements, it may be adjusted for operation with any G-M or scintillator tube between 400 and 1500 volts.

The unit is operated with two flashlight batteries for operation from 150° to approximately 32°F. For temperature operation to 0°F, either very fresh alkaline batteries or rechargeable NiCd batteries may be used. Battery drain averages 30 milliamperes.

3.2 CALIBRATION

4.0 PROCEDURES

4.1 OPERATIONAL CHECKS

1. Switch the range switch to BAT. The meter should deflect to the battery check portion of the meter scale. If the meter does not respond, recheck that the batteries have proper polarity.
2. Connect the cable to the instrument and detector.
3. Turn the instrument range switch to X100. Expose the detector to a check source. The speaker should click with the AUDIO ON-OFF switched to ON.
4. Move the range switch to the lower scales until a meter reading is indicated. The toggle switch labeled F-S should have fast response in "F" and slow response in "S."
5. Depress the RES switch. The meter should zero.
6. Proceed to use the instrument.

5.0 MAINTENANCE AND CALIBRATION SCHEDULE

6.0 REFERENCES

E

APPENDIX E

SPECIAL ANALYTICAL SERVICES

ARMY CREEK LANDFILL SITE

SUMMARY OF SAS REQUESTS AND TABLE OF CONTENTS
ARMY CREEK LANDFILL SITE

Form No.	Collection Begin	Collection End	Sample Type	No. of Samples	Blanks	Duplicates	Work Units	Analysis	Page
1	6-26-89	7-7-89	Groundwater	10	2	1	13	Full Organics TCL, (VOA, BNA, Pest/PCB)	AC-TLC-1
2	6-26-89	7-7-89	Groundwater	10	1	1	12	CLP Metals TAL (dissolved Metals, total metals and cyanide)	AC-TAL-1
3	6-26-89	7-7-89	Groundwater	10	1	1	12	Alkalinity	AC-ALK-1
				10	1	1	12	Acidity	
				10	1	1	12	Total Suspended Solids	(TSS)
				10	1	1	12	Total Dissolved Solids	(TDS)
				10	1	1	12	Total Organic Carbon	(TOC)
4	6-26-89	7-7-89	Groundwater	10	1	1	12	Sulfide	AC-HS2-1
5	6-26-89	7-7-89	Groundwater	10	1	1	12	Ammonia Total Kjeldahl Nitrogen	AC-NH4-1
6	6-26-89	7-7-89	Groundwater	10	1	1	12	Total Phosphorus Total Acid - Hydrolyzable Phosphorus Total Reactive Phosphorus Total Organic Phosphorus	AC-PO4-1
7	6-26-89	7-7-89	Groundwater	10	1	1	12	Nitrate Nitrite	AC-NO3-1
8	6-26-89	7-7-89	Groundwater	10	1	1	12	Sulfate Chloride	AC-SOCL-1

correction

SUMMARY OF SAS REQUESTS AND TABLE OF CONTENTS
ARMY CREEK LANDFILL SITE

Form No.	Collection Begin	Collection End	Sample Type	No. of Samples	Blanks	Duplicates	Work Units	Analysis	Page
1	6-26-89	7-7-89	Groundwater	10	2	1	13	Full Organics TCL, (VOA, BNA, Pest/PCB)	AC-TLC-1
2	6-26-89	7-7-89	Groundwater	10	1	1	12	CLP Metals TAL (dissolved Metals, total metals and cyanide)	AC-TAL-1
3	6-26-89	7-7-89	Groundwater	10	1	1	12	Alkalinity	AC-ALK-1
				10	1	1	12	Acidity	
				10	1	1	12	Total Suspended Solids	(TSS)
				10	1	1	12	Total Dissolved Solids	(TDS)
				10	1	1	12	Total Organic Carbon	(TOC)
4	6-26-89	7-7-89	Groundwater	10	1	1	12	Sulfide	AC-HS2-1
5	6-26-89	7-7-89	Groundwater	10	1	1	12	Ammonia Total Kjeldahl Nitrogen	AC-NH4-1
6	6-26-89	7-7-89	Groundwater	10	1	1	12	Total Phosphorus Total Acid - Hydrolyzable Phosphorus Total Reactive Phosphorus Total Organic Phosphorus	AC-PO4-1
7	6-26-89	7-7-89	Groundwater	10	1	1	12	Nitrate Nitrite	AC-NO3-1
8	6-26-89	7-7-89	Groundwater	10	1	1	12	Sulfate Chloride	AC-SOCL-1

U.S. Environmental Protection Agency
CLP Sample Management Office
209 Madison Street, Alexandria, VA 22313
PHONE: (703) 557-2490 or FTS 557-2490

SAS Number

SPECIAL ANALYTICAL SERVICES
Regional Request

☐ Regional Transmittal ☐ Telephone Request

A. EPA Region and Client: EPA Region III

B. Regional Representative: Colleen K. Walling

C. Telephone Number: (301) 266-9180

D. Date of Request:

E. Site Name: Army Creek Landfill Site
New Castle County, DE

Please provide below a description of your request for Special Analytical Services under the Contract Laboratory Program. In order to most efficiently obtain laboratory capability for your request, please address the following considerations, if applicable. Incomplete or erroneous information may result in delay in the processing of your request. Please continue response on additional sheets, or attach supplementary information as needed.

1. General description of analytical service requested:

   Quick turnaround analysis of 10 groundwater for full organics TCL (VOA, BNA, and Pest/PCB).

2. Definition and number of work units involved (specify whether whole samples or fractions; whether organics or inorganics; whether aqueous or soil and sediments; and whether low, medium, or high concentration):

   10 low concentration groundwater samples for CLP organics analyses (VOA, BNA, and Pesticide/PCB), plus 1 field duplicate, 1 field blank, and 1 trip blank (VOA only) for a total of 13 work units. See items 6, 7, 8, 9, 12, 13 and 14 for details.

3. Program (specify whether Superfund (Remedial or Enforcement), RCRA, NPDES, etc.), Justification for analysis and Site Account Number:

   Superfund RI/FS ENFORCEMENT
   Account Number

   SAS Approved By:

4. Estimated date(s) of collection:

   June 26, 1989 through July 7, 1989.

5. Estimated date(s) and method of shipment:

   June 26, 1989 through July 7, 1989.

   Samples will be shipped daily by overnight air carrier. These dates are tentative and are dependent on project remaining on schedule. Sampling may continue into the week of July 10, 1989.

6. Approximate number of days results required after lab receipt of samples:

   Data package is due 14 days from date of receipt of the last sample.

7. Analytical protocol required (attach copy if other than a protocol currently used in this program):

   Analysis by CLP SOW Organics (2/88)--diskette deliverable at SMO discretion.

8. Special technical instructions (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):

   The sample to be used for laboratory QC will be collected with extra volume and will be clearly labelled "Do QC" on the SAS packing list.

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc.). If not completed, format of results will be left to program discretion.

   All raw data, calculations, data sheets, blank results, duplicate results, SAS packing lists, copy of airbill, copy of analyst's logbooks, Chain-of-Custody forms, SAS Request forms and date of analysis for all parameters must be included, as well as, deliverables as per CLP--Organic (2/88) SOW are required in final data package.

   The cover page and all sample report forms must be labelled with SAS and Task numbers, and EPA sample numbers as they appear on Chain-of Custody and other CLP paperwork.

10. Other (use additional sheets or attach supplementary information, as needed):

11. Name of sampling/shipping contact: Emily Olds, Gannett Fleming
Environmental Engineers, Inc.

Phone: (301) 433-8832

12. Data Requirements

Parameter	Detection Limit	Precision Desired (+ or - Concentration)
Organics	As per (2/88) CLP-SOW	As per (2/88) CLP-SOW

13. QC Requirements

Audits Required	Frequency of Audits	Limits (Percent or Concentration)
Organics	As per (2/88) CLP-SOW	As per (2/88) CLP-SOW

14. Action Required if Limits are Exceeded

Organics--As per CLP-SOW 2/88

15. Request prepared by: Emily Olds

Date: June 30, 1989

16. Request reviewed by (CRL use only):

Date:

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please contact your Regional representative at the Sample Management Office.

U.S. Environmental Protection Agency
CLP Sample Management Office
209 Madison Street, Alexandria, VA 22313
PHONE: (703) 557-2490 or FTS 557-2490

SAS Number

SPECIAL ANALYTICAL SERVICES
Regional Request

☐ Regional Transmittal

☐ Telephone Request

A. EPA Region and Client: EPA Region III

B. Regional Representative: Colleen K. Walling

C. Telephone Number: (301) 266-9180

D. Date of Request:

E. Site Name: Army Creek Landfill Site
New Castle County, DE

Please provide below a description of your request for Special Analytical Services under the Contract Laboratory Program. In order to most efficiently obtain laboratory capability for your request, please address the following considerations, if applicable. Incomplete or erroneous information may result in delay in the processing of your request. Please continue response on additional sheets, or attach supplementary information as needed.

1. General description of analytical service requested:

   Quick turnaround analysis of 10 groundwater for CLP metals TAL. Samples collected in enough containers and with adequate volume to permit analysis of all inorganics, including dissolved metals (samples filtered on-site), total metals and cyanide (both samples unfiltered).

2. Definition and number of work units involved (specify whether whole samples or fractions; whether organics or inorganics; whether aqueous or soil and sediments; and whether low, medium, or high concentration):

   10 low concentration groundwater samples for CLP Inorganics analyses (Total metals, dissolved metals and cyanide), plus 1 field duplicate and 1 field blank for a total of 12 work units. See items 6, 7, 8, 9, 12, 13 and 14 for details.

3. Program (specify whether Superfund (Remedial or Enforcement), RCRA, NPDES, etc.), Justification for analysis and Site Account Number:

   Superfund RI/FS ENFORCEMENT
   Account Number

   SAS Approved By:

4. Estimated date(s) of collection:

   June 26, 1989 through July 7, 1989.

5. Estimated date(s) and method of shipment:

   June 26, 1989 through July 7, 1989.

   Samples will be shipped daily by overnight air carrier. These dates are tentative and are dependent on project remaining on schedule. Sampling may continue into the week of July 10, 1989.

6. Approximate number of days results required after lab receipt of samples:

   Data package is due 14 days from date of receipt of the last sample.

7. Analytical protocol required (attach copy if other than a protocol currently used in this program):

   Analysis by CLP SOW Inorganic (7/87)--diskette deliverable at SMO discretion.

8. Special technical instructions (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):

   The sample to be used for laboratory QC will be collected with extra volume and will be clearly labelled "Do QC" on the SAS packing list. All samples for dissolved metals must be digested.

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc.). If not completed, format of results will be left to program discretion.

   All raw data, calculations, data sheets, blank results, duplicate results, SAS packing lists, copy of airbill, copy of analyst's logbooks, Chain-of-Custody forms, SAS Request forms and date of analysis for all parameters must be included, as well as, deliverables as per CLP--Inorganic (7/87) SOW are required in final data package.

   The cover page and all sample report forms must be labelled with SAS and Task numbers, and EPA sample numbers as they appear on Chain-of Custody and other CLP paperwork.

10. Other (use additional sheets or attach supplementary information, as needed):

11. Name of sampling/shipping contact: Emily Olds, Gannett Fleming
Environmental Engineers, Inc.

Phone: (301) 433-8832

12. Data Requirements

Parameter	Detection Limit	Precision Desired (+ or - Concentration)
Inorganics	As per (7/87) CLP-SOW	As per (7/87) CLP-SOW

13. QC Requirements

Audits Required	Frequency of Audits	Limits (Percent or Concentration)
Inorganics	As per (7/87) CLP-SOW	As per (7/87) CLP-SOW

14. Action Required if Limits are Exceeded

Inorganics--As per CLP-SOW 7/87

15. Request prepared by: Emily Olds

Date: June 30, 1989

16. Request reviewed by (CRL use only):

Date:

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please contact your Regional representative at the Sample Management Office.

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SPECIAL ANALYTICAL SERVICES
Regional Request

☐ Regional Transmittal ☐ Telephone Request

A. EPA Region and Client: EPA Region III

B. Regional Representative: Colleen K. Walling

C. Telephone Number: (301) 266-9180

D. Date of Request:

E. Site Name: Army Creek Landfill Site
New Castle County, DE

Please provide below a description of your request for Special Analytical Services under the Contract Laboratory Program. In order to most efficiently obtain laboratory capability for your request, please address the following considerations, if applicable. Incomplete or erroneous information may result in delay in the processing of your request. Please continue response on additional sheets, or attach supplementary information as needed.

1. General description of analytical service requested:

   Quick turnaround analysis of 10 groundwater samples for Alkalinity, Acidity, Total Suspended Solids (TSS), Total Dissolved Solids (TDS) and Total Organic Carbon (TOC). Use EPA Method 305.1 (attachment 1) for Acidity, EPA Method 310.1 (attachment 2) for Alkalinity, EPA Method 160.2 (attachment 3) for TSS, EPA Method 160.1 (attachment 4) for TDS, and EPA/COE Method CE-81-1-method 1 (attachment 5) for TOC.

   TOC samples will be filtered and acidified in the field.

2. Definition and number of work units involved (specify whether whole samples or fractions; whether organics or inorganics; whether aqueous or soil and sediments; and whether low, medium, or high concentration):

   10 low concentration groundwater samples for the above plus 1 duplicate and 1 blank for a total of 12 work units. See items number 6, 7, 8, 9, 12, 13 and 14 for details.

3. Program (specify whether Superfund (Remedial or Enforcement), RCRA, NPDES, etc.), Justification for analysis and Site Account Number:

Superfund RI/FS Enforcement
Account Number:

SAS Approved By:

4. Estimated date(s) of collection:

June 26, 1989 through July 7, 1989.

5. Estimated date(s) and method of shipment:

June 26, 1989 through July 7, 1989.

Samples will be shipped daily by overnight air carrier. These dates are tentative and are dependent on project remaining on schedule. Sampling may continue into the week of June 10, 1989. Friday shipments are a possibility.

6. Approximate number of days results required after lab receipt of samples:

Data package due 14 days from date of receipt of the last sample for all parameters.

Alkalinity and Acidity samples must be analyzed within 13 days of VTSR. TSS and TDS must be analyzed within 6 days of VTSR. TOC must be analyzed within 48 hours of VTSR for each sample.

7. Analytical protocol required (attach copy if other than a protocol currently used in this program): All Methods Attached

Alkalinity - EPA 310.1 - EPA, March 1983. "Methods for Chemical Analysis of Water and Wastes."
Acidity - EPA 305.1 - " "
TSS - EPA 160.2 - " "
TDS - EPA 160.1 - " "
TOC - EPA 415.1 - " "

Alkalinity and Acidity- pH stock buffers, 7.0, 4.0 and 10.0, must be analyzed before each run, and readings must be documented and included in data package.

8. *Special technical instructions* (if-outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):

TDS and TSS- NBS certified Class "S" weights must be used to check the balance before each use. Results of balance check must be clearly reported, including date and time of check.

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc.). If not completed, format of results will be left to program discretion.

Each individual task as awarded must be submitted in a separate data package. The cover page and all sample report forms must be labelled with both SAS, Task numbers, and EPA sample numbers as they appear on Chain-Of-Custody and other CLP paperwork.

Raw data, calculation, data sheets, blank results, duplicate results, SAS packing list, copy of airbill, copy of analyst's logbooks, Chain-of-Custody forms, and SAS Request forms, lab custody record and date of analysis for all parameters must be included. Report forms must indicate analysis performed for each sample.

10. Other (use additional sheets or attach supplementary information, as needed):

Use case narrative for documenting problems encountered and problem resolution.

11. Name of sampling/shipping contact: Emily Olds, Gannett Fleming Environmental
Engineers, Inc.
Phone: (301) 433-8832

12. Data Requirements

Parameter	Detection Limit	Precision Desired (+ or - Concentration)
Alkalinity	4 mg/l	± 20%
Acidity	10 mg/l	± 20%
TSS	4 mg/l	± 20%
TDS	10 mg/l	± 20%
TOC	20 mg/l	± 20%

13. QC Requirements

Audits Required	Frequency of Audits	Limits (Percent or Concentration)
Blanks	1 set for each parameter analyzed with every analytical run.	Below Method Detection Limit
Duplicates	1 per batch per parameter	± 20% RPD
Class "S" Weights	weigh each weight with each batch of samples analyzed	N/A
TOC certified standard	1 per analytical run minimum of one per 20	± 30% RPD
Calibrated pH meter	1 time per run for each buffer	Manufacturer's Specifications

Balance check-NBS Certified Class "S" weights	With every analytical run	Manufacturer's Specifications

14. Action Required if Limits are Exceeded

If duplicate is out of limit, reanalyze duplicate pair once more. If still out of limit, repeat analysis of all samples along with all QC samples once more. If blank or TOC certified standard is out of limit, reanalyze all samples after corrective action has been taken to reduce blank contamination to the acceptable limit. Note in narrative. If problems occur needing further resolution notify Region III.

5. Request prepared by: Emily Olds

Date: June 30, 1989

16. Request reviewed by (CRL use only):

Date:

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please contact your Regional representative at the Sample Management Office.

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SAS Number

SPECIAL ANALYTICAL SERVICES
Regional Request

☐ Regional Transmittal ☐ Telephone Request

A. EPA Region and Client: EPA Region III

B. Regional Representative: Colleen K. Walling

C. Telephone Number: (301) 266-9180

D. Date of Request:

E. Site Name: Army Creek Landfill
New Castle County, DE

Please provide below a description of your request for Special Analytical Services under the Contract Laboratory Program. In order to most efficiently obtain laboratory capability for your request, please address the following considerations, if applicable. Incomplete or erroneous information may result in delay in the processing of your request. Please continue response on additional sheets, or attach supplementary information as needed.

1. General description of analytical service requested:

   Quick turnaround analysis of 10 groundwater samples for Sulfide. Use <u>Standard Method</u> 427D (attachment) for this analysis. Samples will arrive preserved with zinc acetate and sodium hydroxide.

2. Definition and number of work units involved (specify whether whole samples or fractions; whether organics or inorganics; whether aqueous or soil and sediments; and whether low, medium, or high concentration):

   10 low concentration groundwater samples for sulfide plus 1 duplicate, 1 blank, and 1 matrix-spiked sample for a total of 13 work units.

3. Program (specify whether Superfund (Remedial or Enforcement), RCRA, NPDES, etc.), Justification for analysis and Site Account Number:

   Superfund RI/FS Enforcement
   Account Number:

SAS Approved By:

4. Estimated date(s) of collection:

June 26, 1989 through July 7, 1989.

5. Estimated date(s) and method of shipment:

June 26, 1989 through July 7, 1989.

Samples will be shipped daily by overnight air carrier. These dates are tentative and are dependent on project remaining on schedule. Sampling may continue into the week of July 10, 1989. Friday shipments are a possibility.

6. Approximate number of days results required after lab receipt of samples:

Data packages due 14 days from date of receipt of the last sample. Sulfide must be analyzed within 7 days of VTSR.

7. Analytical protocol required (attach copy if other than a protocol currently used in this program):

Sulfide- 427D- APHA-AWWA-WPCF, 1985. "Standard Methods For the Examination of Water and Wastewater" 16th ed.

8. Special technical instructions (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):

Lab will perform filtration of samples and analysis of filtrates as described in Standard Methods 427B paragraph b.

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc.). If not completed, format of results will be left to program discretion.

The cover page and all sample report forms must be labelled with both SAS and Task numbers.

Raw data, calculation, data sheets, blank results, duplicate results, Chain-of-Custody forms, SAS Request forms, calibration curve data, copy of SAS packing list, copy of airbill, and copy of Lab analyst's logbooks.

10. Other (use additional sheets or attach supplementary information, as needed):

11. Name of sampling/shipping contact: Emily Olds, Gannett Fleming
Environmental Engineers, Inc.
Phone: (301) 433-8832

12. Data Requirements

Parameter	Detection Limit	Precision Desired (+ or - Concentration)
Sulfide	1 mg/L	± 10%

13. QC Requirements

Audits Required	Frequency of Audits	Limits (Percent or Concentration)
Blanks	1/20	Below Method Detection Limit
Duplicates	1/20	± 10% RPD
Matrix Spike	1/20	95% Confidence Interval

14. Action Required if Limits are Exceeded

Blank- If blank values exceed MDL, after appropriate action to reduce blank to less than MDL, repeat all samples and QC.

Duplicate- If duplicate is out of limits, reanalyze duplicate pair. If still outside range, repeat analysis of all samples and QC once more.

Matrix Spike- If matrix spike is outside limits, reprepare and reanalyze all associated samples.

15. Request prepared by: Emily Olds

Date: June 30, 1989

16. Request reviewed by (CRL use only):

Date:

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please contact your Regional representative at the Sample Management Office.

U.S. Environmental Protection Agency
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SAS Number ☐

SPECIAL ANALYTICAL SERVICES
Regional Request

☐ Regional Transmittal ☐ Telephone Request

A. EPA Region and Client: EPA Region III

B. Regional Representative: Colleen K. Walling

C. Telephone Number: (301) 266-9180

D. Date of Request:

E. Site Name: Army Creek Landfill
New Castle County, DE

Please provide below a description of your request for Special Analytical Services under the Contract Laboratory Program. In order to most efficiently obtain laboratory capability for your request, please address the following considerations, if applicable. Incomplete or erroneous information may result in delay in the processing of your request. Please continue response on additional sheets, or attach supplementary information as needed.

1. General description of analytical service requested:

   Quick turnaround analysis of 10 groundwater samples for Ammonia and total Kjeldahl nitrogen. Use Standard Methods 417A (distillation) followed by Standard Methods 417E (Ion-Selective Electrode) for Ammonia, and Standard Methods 420A (Digestion and Distillation) followed by Standard Methods 417E for Total Kjeldahl nitrogen. A mid-point check standard and a certified ammonia reference sample obtained by the lab must be run every 10 samples.

2. Definition and number of work units involved (specify whether whole samples or fractions; whether organics or inorganics; whether aqueous or soil and sediments; and whether low, medium, or high concentration):

   10 low concentration groundwater samples for above plus 1 field duplicate, 1 field blank and 1 matrix-spike sample for a total of 13 work units. See items 6, 7, 8, 9, 10, 11, 12, 13 and 14 for details.

3. Program (specify whether Superfund (Remedial or Enforcement), RCRA, NPDES, etc.), Justification for analysis and Site Account Number:

Superfund RI/FS
Account Number:

SAS Approved By:

4. Estimated date(s) of collection:

June 26, 1989 through July 7, 1989.

5. Estimated date(s) and method of shipment:

June 26, 1989 through July 7, 1989

Samples will be shipped daily by overnight air carrier. These dates are tentative and are dependent on project remaining on schedule. Sampling may continue into the week of July 10, 1989. Friday shipments are a possibility.

6. Approximate number of days results required after lab receipt of samples:

Data packages must be delivered within 14 days of date of receipt of the last sample.

7. Analytical protocol required (attach copy if other than a protocol currently used in this program):

Ammonia- 417, 417E-APHA-AWWA-WPCF, 1985. "Standard Methods For the Examination of Water and Wastewater 16th ed.
Total Kjeldahl Nitrogen- 420A, 417E-APHA-AWWA-WPCF, 1985 "Standard Methods For the Examination of Water and Wastewater" 16th ed.
Determine organic nitrogen by difference of total Kjeldahl Nitrogen and Ammonia.

8. Special technical instructions (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):

Include a five point calibration curve for ammonia in the range of the samples undergoing analysis. Separate curves will be needed for ammonia and total Kjeldahl nitrogen analysis. Run a mid-point check standard every 10 samples. Lab must obtain and report a certified reference standard for ammonia with its acceptance limits, lot number and SOW, and include a copy of the manufacturer's specifications with data deliverability.

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc.). If not completed, format of results will be left to program discretion.

Each individual task as awarded must be submitted in a separate data package. The cover page and all sample report forms must be labelled with both SAS and Task numbers, and EPA sample numbers as they appear on Chain-of-Custody and CLP paperwork.

All raw data, calculations, data sheets, blank results, duplicate results, lab and EPA Chain-of-Custody forms, SAS Request forms, standardization data, calibration curve data, external certified standard data, copy of SAS packing list, copy of airbill, and copy of lab analyst's logbooks with date of analysis for each run and each parameter analyzed.

10. Other (use additional sheets or attach supplementary information, as needed):

Document all problems, troubleshooting and problem resolution in the case narrative.

11. Name of sampling/shipping contact: Emily Olds, Gannett Fleming Environmental Engineers, Inc.

Phone: (301) 433-8832

12. Data Requirements

Parameter	Detection Limit	Precision Desired (+ or - Concentration)
Ammonia	0.03 mg/L	± 20%
Total Kjeldahl nitrogen	0.03 mg/L	± 20%
Organic nitrogen	0.03 mg/L	± 20%

13. QC Requirements

Audits Required	Frequency of Audits	Limits (Percent or Concentration)
Blanks	1 set for each parameter prepared	Below Method Detection Limit
Duplicates	1/20 per parameter	± 10% RPD
Matrix Spike	1/20 per parameter	95% Confidence Interval
Externally Certified Standard Performance	1/10 samples for ammonia	95% Confidence Interval
Midrange Check Standard	1/10 samples per parameter	± 10% RPD

14. Action Required if Limits are Exceeded

Blank- If blank values exceed MDL, after appropriate action to reduce blank to less than MDL, repeat all samples and QC.

Duplicate- If duplicate is out of limits, reanalyze duplicate pair. If still outside range, repeat analysis of all samples and QC once more.

Check Standard- If midrange check standard is outside limits, reprepare and reanalyze the check standard and all samples between check standards that exceed limits.

Matrix Spike- If matrix spike is outside limits, reprepare and reanalyze all associated samples.

15. Request prepared by: Emily Olds

Date: June 30, 1989

16. Request reviewed by (CRL use only):

Date:

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please contact your Regional representative at the Sample Management Office.

U.S. Environmental Protection Agency
CLP Sample Management Office
209 Madison Street, Alexandria, VA 22313
PHONE: (703) 557-2490 or FTS 557-2490

SAS Number

SPECIAL ANALYTICAL SERVICES
Regional Request

☐ Regional Transmittal ☐ Telephone Request

A. EPA Region and Client: EPA Region III

B. Regional Representative: Colleen K. Walling

C. Telephone Number: (301) 266-9180

D. Date of Request:

E. Site Name: Army Creek Landfill
New Castle County, DE

Please provide below a description of your request for Special Analytical Services under the Contract Laboratory Program. In order to most efficiently obtain laboratory capability for your request, please address the following considerations, if applicable. Incomplete or erroneous information may result in delay in the processing of your request. Please continue response on additional sheets, or attach supplementary information as needed.

1. General description of analytical service requested:

   Quick turnaround analysis of 10 groundwater samples for Total phosphorus, total acid-hydrolyzable phosphorus, Total reactive phosphorus and Total organic phosphorus. Use Standard Methods 424C, III (Persulfate Digestion Method) for Digestion for Total phosphorus (attachment 1); Standard Methods 424B (Acid, Hydrolysis) for Total Acid-hydrolyzable phosphorus (attachment 2); Standard Methods 424F (Ascorbic Acid) for colorimetric detection of all phosphorus components (attachment 3). A certified reference standard for total phosphorus must be obtained by the lab and run every 10 samples. A midpoint check standard must be run every 10 samples.

2. Definition and number of work units involved (specify whether whole samples or fractions; whether organics or inorganics; whether aqueous or soil and sediments; and whether low, medium, or high concentration):

   10 low concentration groundwater samples for the above plus 1 field duplicate, 1 field blank and 1 matrix spiked sample (total phosphorus only) for a total of 13 work units. See items 6, 7, 8, 9, 10, 12, 13 and 14 for details.

3. Program (specify whether Superfund (Remedial or Enforcement), RCRA, NPDES, etc.), Justification for analysis and Site Account Number:

Superfund RI/FS Enforcement
Account Number:

SAS Approved By:

4. Estimated date(s) of collection:

June 26, 1989 through July 7, 1989

5. Estimated date(s) and method of shipment:

June 26, 1989 through July 7, 1989.

Samples will be shipped daily by overnight air carrier. These dates are tentative and are dependent on project remaining on schedule. Sampling may continue into the week of July 10, 1989. Friday shipments are a possibility.

6. Approximate number of days results required after lab receipt of samples:

Data package must be delivered within 14 days from date of receipt of the last sample.

7. Analytical protocol required (attach copy if other than a protocol currently used in this program):

Total Phosphorus 424C,III; 424F-APHA-AWWA-WPCF, 1985.	"Standard Methods for the Examination of Water and Waste-Water" 16th ed.	
Total acid-hydrolyzable 424B; 424F-- phosphorus	"	"
Total reactive phosphorus 424F--	"	"
Total organic phosphorus--see Fig 424:1 for calculation	"	"

8. Special technical instructions (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):

Include a 5-point calibration in the range of the samples undergoing analysis for each fraction analyzed by colorimetry. Lab must obtain and report a certified reference standard for total phosphorus with its acceptance limits, lot number and SOW, and include a copy of the manufacturer's specifications with data deliverables.

Note that Total acid-hydrolyzable phosphorus and Total organic phosphorus are arrived at by subtraction as shown in Figure 424:1 (attachment 4).

A midpoint check standard must be performed every 10 samples.

9. **Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc.). If not completed, format of results will be left to program discretion.**

   Each individual task as awarded must be submitted in a separate data package. The cover page and all sample report forms must be labelled with both SAS and Task numbers.

   Raw data, calculations, data sheets, blank results, duplicate results, Chain-of-Custody forms, SAS request forms, calibration curve data, external certified standard data, copy of SAS packing list, copy of airbill, and copy of Lab analyst's logbooks.

   All calculations for each fraction must be provided.

10. **Other (use additional sheets or attach supplementary information, as needed):**

    Document all problems, troubleshooting and problem resolution in the case narrative.

11. Name of sampling/shipping contact: Emily Olds, Gannett Fleming Environmental Engineers, Inc.

    Phone: (301) 433-8832

12. Data Requirements

Parameter	Detection Limit	Precision Desired (+ or - Concentration)
Total phosphorus	10 ug/L	± 20%
Total acid-hydrolyzable phosphorus	10 ug/L	± 20%
Total reactive phosphorus	10 ug/L	± 20%
Total organic phosphorus	10 ug/L	± 20%

13. QC Requirements

Audits Required	Frequency of Audits	Limits (Percent or Concentration)
Blanks	1 set for each parameter prepared	Below Method Detection Limit
Duplicates	1/20 per parameter	± 10% RPD
Matrix Spike	1/20 per parameter	95% Confidence Interval
Externally Certified Standard Performance	1/10 samples--Total phosphorus only	95% Confidence Interval
Midrange Check	1/10 samples	± 10% RPD

14. Action Required if Limits are Exceeded

If duplicate is out of limit, reanalyze duplicate pair once more. If still out of limit, repeat analysis of all samples along with all QC samples once more. If blank or TOC certified standard is out of limit, reanalyze all samples after corrective action has been taken to reduce blank contamination to the acceptable limit. Note in narrative. If problems occur needing further resolution notify Region III.

15. Request prepared by: Emily Olds

Date: June 30, 1989

16. Request reviewed by (CRL use only):

Date:

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please contact your Regional representative at the Sample Management Office.

U.S. Environmental Protection Agency
CLP Sample Management Office
209 Madison Street, Alexandria, VA 22313
PHONE: (703) 557-2490 or FTS 557-2490

SAS Number

SPECIAL ANALYTICAL SERVICES
Regional Request

☐ Regional Transmittal

☐ Telephone Request

A. EPA Region and Client: EPA Region III

B. Regional Representative: Colleen K. Walling

C. Telephone Number: (301) 266-9180

D. Date of Request:

E. Site Name: Army Creek Landfill
New Castle County, DE

Please provide below a description of your request for Special Analytical Services under the Contract Laboratory Program. In order to most efficiently obtain laboratory capability for your request, please address the following considerations, if applicable. Incomplete or erroneous information may result in delay in the processing of your request. Please continue response on additional sheets, or attach supplementary information as needed.

1. General description of analytical service requested:

   Quick turnaround analysis of 10 groundwater samples for Nitrite and Nitrate. Use EPA Method 353.2 (attachment) for this analysis.

2. Definition and number of work units involved (specify whether whole samples or fractions; whether organics or inorganics; whether aqueous or soil and sediments; and whether low, medium, or high concentration):

   10 low concentration groundwater samples for Nitrite and Nitrate plus 1 duplicate, 1 blank, and 1 matrix-spiked sample for a total of 13 work units.

3. Program (specify whether Superfund (Remedial or Enforcement), RCRA, NPDES, etc.), Justification for analysis and Site Account Number:

   Superfund RI/FS Enforcement
   Account Number:

   SAS Approved By:
4. Estimated date(s) of collection:

   June 26, 1989 through July 7, 1989.

5. Estimated date(s) and method of shipment:

   June 26, 1989 through July 7, 1989.

   Samples will be shipped daily by overnight air carrier. These dates are tentative and are dependent on project remaining on schedule. Sampling may continue into the week of July 10, 1989.

6. Approximate number of days results required after lab receipt of samples:

   Data packages due 14 days from date of receipt of the last sample.

7. Analytical protocol required (attach copy if other than a protocol currently used in this program):

   Both Nitrate and Nitrite- EPA 353.2 - EPA March 1983, "Methods for Chemical Analysis of Water and Wastes."

8. Special technical instructions (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):

   Lab must obtain and report a certified reference standard with its acceptance limits, lot number and SOW.

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc.). If not completed, format of results will be left to program discretion.

   Each individual task as awarded must be submitted in a separate data package. The cover page and all sample report forms must be labelled with both SAS and Task numbers.

   Raw data, calculation, data sheets, blank results, duplicate results, Chain-of-Custody forms, SAS Request forms, standardization data, calibration curve data, external certified standard data, cadmium column reduction efficiency results, copy of SAS packing list, copy of airbill, and copy of Lab analyst's logbooks.

10. Other (use additional sheets or attach supplementary information, as needed):

11. Name of sampling/shipping contact: Emily Olds, Gannett Fleming Environmental Engineers, Inc.

Phone: (301) 433-8832

12. Data Requirements

Parameter	Detection Limit	Precision Desired (+ or - Concentration)
Nitrate	50 ugN/l	± 20%
Nitrite	50 ugN/l	± 20%

13. QC Requirements

Audits Required	Frequency of Audits	Limits (Percent or Concentration)
Blanks	1/20	Below Method Detection Limit
Duplicates	1/20	± 10% RPD
Matrix Spike	1/20	95% Confidence Interval
Externally Certified Standard Performance	1/batch	95% Confidence Interval
Cadmium Reduction Efficiency	1/20	90-110% Reduction

14. Action Required if Limits are Exceeded

Repeat QC sample in question; if results are still outside QC limits, reanalyze all samples for that parameter once more along with QC samples.

15. Request prepared by: Emily Olds

Date: June 30, 1989

16. Request reviewed by (CRL use only):

Date:

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please contact your Regional representative at the Sample Management Office.

U.S. Environmental Protection Agency
CLP Sample Management Office
209 Madison Street, Alexandria, VA 22313
PHONE: (703) 557-2490 or FTS 557-2490

SAS Number

SPECIAL ANALYTICAL SERVICES
Regional Request

☐ Regional Transmittal ☐ Telephone Request

A. EPA Region and Client: EPA Region III

B. Regional Representative: Colleen K. Walling

C. Telephone Number: (301) 266-9180

D. Date of Request:

E. Site Name: Army Creek Landfill Site
New Castle County, DE

Please provide below a description of your request for Special Analytical Services under the Contract Laboratory Program. In order to most efficiently obtain laboratory capability for your request, please address the following considerations, if applicable. Incomplete or erroneous information may result in delay in the processing of your request. Please continue response on additional sheets, or attach supplementary information as needed.

1. General description of analytical service requested:

   Quick turnaround analysis of 10 groundwater samples for Sulfate, and Chloride. Use EPA Method 375.4 (attachment 1) for Sulfate, and EPA Method 325.3 (attachment 2) for Chloride. Plus certified reference sample obtained by lab and a check standard every 10 samples which consists of a mid-range standard.

2. Definition and number of work units involved (specify whether whole samples or fractions; whether organics or inorganics; whether aqueous or soil and sediments; and whether low, medium, or high concentration):

   10 low concentration groundwater samples for the above plus 1 duplicate, 1 blank, and 1 matrix-spiked sample for a total of 13 work units. See items 6, 7, 8, 9, 10, 12, 13 and 14 for details.

3. Program (specify whether Superfund (Remedial or Enforcement), RCRA, NPDES, etc.), Justification for analysis and Site Account Number:

Superfund RI/FS Enforcement
Account Number:

SAS Approved By:

4. Estimated date(s) of collection:

June 26, 1989 through July 7, 1989.

5. Estimated date(s) and method of shipment:

June 26, 1989 through July 7, 1989.

Samples will be shipped daily by overnight air carrier. These dates are tentative and are dependent on project remaining on schedule. Sampling may continue into the week of July 10, 1989. Friday shipments are a possibility.

6. Approximate number of days results required after lab receipt of samples:

Data packages must be delivered within 14 days from date of receipt of the last sample.

Sulfate and Chloride must be analyzed within 26 days of VTSR for each sample.

7. Analytical protocol required (attach copy if other than a protocol currently used in this program): All Methods Attached

Sulfate - EPA 375.4 - EPA, March 1983. "Methods for Chemical Analysis of Water and Wastes."
Chloride - EPA 325.3 - " "

8. Special technical instructions (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):

Include a 5 point calibration curve for chloride analysis in the range of the samples undergoing analysis. Lab must obtain and report a certified reference standard with its acceptance limits, lot number and SOW, and include a copy of the manufacturer's specifications with data deliverables.

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc.). If not completed, format of results will be left to program discretion.

Each individual task as awarded must be submitted in a separate data package. The cover page and all sample report forms must be labelled with both SAS and Task numbers, and EPA sample numbers as they appear on Chain-of-Custody and CLP paperwork.

All raw data, calculations, data sheets, blank results, duplicate results, lab and EPA Chain-of-Custody forms, SAS Request forms, standardization data, calibration curve data, external certified standard data, cadmium column reduction, efficiency results, copy of SAS packing list, copy of airbill, and copy of Lab analyst's logbooks with date of analysis for each run and each parameter analyzed.

10. Other (use additional sheets or attach supplementary information, as needed):

    Document all problems, troubleshooting and problem resolution in the case narrative.

11. Name of sampling/shipping contact: Emily Olds, Gannett Fleming Environmental Engineers, Inc.
    Phone: (301) 433-8832

12. Data Requirements

Parameter	Detection Limit	Precision Desired (+ or - Concentration)
Sulfate	5 mg/l	± 20%
Chloride	5 mg/l	± 20%

13. QC Requirements

Audits Required	Frequency of Audits	Limits (Percent or Concentration)
Blanks	1 set for each parameter prepared	Below Method Detection Limit
Duplicates	1/20 per parameter	± 10% RPD
Matrix Spike	1/20 per paramater	95% Confidence Interval
Externally Certified Standard Performance	1/batch per paramater with every analytical run	95% Confidence Interval
Midrange Check Standard	1/batch(with every run)	± 10% RPD

14. Action Required if Limits are Exceeded

Blank- If blank values exceed MDL, after appropriate action to reduce blank to less than MDL, repeat all samples and QC.

Duplicate- If duplicate is out of limits, reanalyze duplicate pair. If still outside range, repeat analysis of all samples and QC once more.

Check Standard- If midrange check standard is outside limits, reprepare and reanalyze the check standard and all samples between check standards that exceed limits.

Matrix Spike- If matrix spike is outside limits, reprepare and reanalyze all associated samples.

Submit all data with a detailed description of problems and action taken to resolve problems. If problems persist, contact Region III for further instructions.

15. Request prepared by: Emily Olds

Date: June 30, 1989

16. Request reviewed by (CRL use only):

Date:

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please contact your Regional representative at the Sample Management Office.

ALKALINITY

Method 310.1 (Titrimetric, pH 4.5)

STORET NO. 00410

1. Scope and Application
   1.1 This method is applicable to drinking, surface, and saline waters, domestic and industrial wastes.
   1.2 The method is suitable for all concentration ranges of alkalinity; however, appropriate aliquots should be used to avoid a titration volume greater than 50 ml.
   1.3 Automated titrimetric analysis is equivalent.
2. Summary of Method
   2.1 An unaltered sample is titrated to an electrometrically determined end point of pH 4.5. The sample must not be filtered, diluted, concentrated, or altered in any way.
3. Comments
   3.1 The sample should be refrigerated at 4°C and run as soon as practical. Do not open sample bottle before analysis.
   3.2 Substances, such as salts of weak organic and inorganic acids present in large amounts, may cause interference in the electrometric pH measurements.
   3.3 For samples having high concentrations of mineral acids, such as mine wastes and associated receiving waters, titrate to an electrometric endpoint of pH 3.9, using the procedure in:
   Annual Book of ASTM Standards, Part 31, "Water", p 115, D-1067, Method D, (1976).
   3.4 Oil and grease, by coating the pH electrode, may also interfere, causing sluggish response.
4. Apparatus
   4.1 pH meter or electrically operated titrator that uses a glass electrode and can be read to 0.05 pH units. Standardize and calibrate according to manufacturer's instructions. If automatic temperature compensation is not provided, make titration at 25 ±2°C.
   4.2 Use an appropriate sized vessel to keep the air space above the solution at a minimum. Use a rubber stopper fitted with holes for the glass electrode, reference electrode (or combination electrode) and buret.
   4.3 Magnetic stirrer, pipets, flasks and other standard laboratory equipment.
   4.4 Burets, Pyrex 50, 25 and 10 ml.
5. Reagents
   5.1 Sodium carbonate solution, approximately 0.05 N: Place 2.5 ±0.2 g (to nearest mg) Na_2CO_3 (dried at 250°C for 4 hours and cooled in desiccator) into a 1 liter volumetric flask and dilute to the mark.

Approved for NPDES
Issued 1971
Editorial revision 1978

5.2 Standard acid (sulfuric or hydrochloric), 0.1 N: Dilute 3.0 ml conc H_2SO_4 or 8.3 ml conc HCl to 1 liter with distilled water. Standardize versus 40.0 ml of 0.05 N Na_2CO_3 solution with about 60 ml distilled water by titrating potentiometrically to pH of about 5. Lift electrode and rinse into beaker. Boil solution gently for 3–5 minutes under a watch glass cover. Cool to room temperature. Rinse cover glass into beaker. Continue titration to the pH inflection point. Calculate normality using:

$$N = \frac{A \times B}{53.00 \times C}$$

where:
A = g Na_2CO_3 weighed into 1 liter
B = ml Na_2CO_3 solution
C = ml acid used to inflection point

5.3 Standard acid (sulfuric or hydrochloric), 0.02 N: Dilute 200.0 ml of 0.1000 N standard acid to 1 liter with distilled water. Standardize by potentiometric titration of 15.0 ml 0.05 N Na_2CO_3 solution as above.

6. Procedure

6.1 Sample size

6.1.1 Use a sufficiently large volume of titrant (> 20 ml in a 50 ml buret) to obtain good precision while keeping volume low enough to permit sharp end point.

6.1.2 For < 1000 mg $CaCO_3$/l use 0.02 N titrant

6.1.3 For > 1000 mg $CaCO_3$/l use 0.1 N titrant

6.1.4 A preliminary titration is helpful.

6.2 Potentiometric titration

6.2.1 Place sample in flask by pipetting with pipet tip near bottom of flask

6.2.2 Measure pH of sample

6.2.3 Add standard acid (5.2 or 5.3), being careful to stir thoroughly but gently to allow needle to obtain equilibrium.

6.2.4 Titrate to pH 4.5. Record volume of titrant.

6.3 Potentiometric titration of low alkalinity

6.3.1 For alkalinity of < 20 mg/l titrate 100–200 ml as above (6.2) using a 10 ml microburet and 0.02 N acid solution (5.3).

6.3.2 Stop titration at pH in range of 4.3–4.7, record volume and exact pH. Very carefully add titrant to lower pH exactly 0.3 pH units and record volume.

7. Calculations

7.1 Potentiometric titration to pH 4.5

$$\text{Alkalinity, mg/l } CaCO_3 = \frac{A \times N \times 50{,}000}{\text{ml of sample}}$$

where:
A = ml standard acid
N = normality standard acid

7.2 Potentiometric titration of low alkalinity:

$$\text{Total alkalinity, mg/l } CaCO_3 = \frac{(2B - C) \times N \times 50{,}000}{\text{ml of sample}}$$

where:
B = ml titrant to first recorded pH
C = total ml titrant to reach pH 0.3 units lower
N = normality of acid

8. Precision and Accuracy

8.1 Forty analysts in seventeen laboratories analyzed synthetic water samples containing increments of bicarbonate, with the following results:

Increment as Alkalinity mg/liter, $CaCO_3$	Precision as Standard Deviation mg/liter, $CaCO_3$	Accuracy as Bias, %	Bias, mg/l, $CaCO_3$
8	1.27	+10.61	+0.85
9	1.14	+22.29	+2.0
113	5.28	- 8.19	-9.3
119	5.36	- 7.42	-8.8

(FWPCA Method Study 1, Mineral and Physical Analyses)

8.2 In a single laboratory (EMSL) using surface water samples at an average concentration of 122 mg $CaCO_3$/l, the standard deviation was ±3.

Bibliography

1. Standard Methods for the Examination of Water and Wastewater, 14th Edition, p 278, Method 403, (1975).
2. Annual Book of ASTM Standards, Part 31, "Water", p 113, D-1067, Method B, (1976).

ACIDITY

Method 305.1 (Titrimetric)

STORET NO. 70508

1. Scope and Application
   - 1.1 This method is applicable to surface waters, sewages and industrial wastes, particularly mine drainage and receiving streams, and other waters containing ferrous iron or other polyvalent cations in a reduced state.
   - 1.2 The method covers the range from approximately 10 mg/l acidity to approximately 1000 mg/l as $CaCO_3$, using a 50 ml sample.
2. Summary of Method
   - 2.1 The pH of the sample is determined and a measured amount of standard acid is added, as needed, to lower the pH to 4 or less. Hydrogen peroxide is added, the solution boiled for several minutes, cooled, and titrated electrometrically with standard alkali to pH 8.2.
3. Definitions
   - 3.1 This method measures the mineral acidity of a sample plus the acidity resulting from oxidation and hydrolysis of polyvalent cations, including salts of iron and aluminum.
4. Interferences
   - 4.1 Suspended matter present in the sample, or precipitates formed during the titration may cause a sluggish electrode response. This may be offset by allowing a 15–20 second pause between additions of titrant or by slow dropwise addition of titrant as the endpoint pH is approached.
5. Apparatus
   - 5.1 pH meter, suitable for electrometric titrations.
6. Reagents
   - 6.1 Hydrogen peroxide (H_2O_2, 30% solution).
   - 6.2 Standard sodium hydroxide, 0.02 N.
   - 6.3 Standard sulfuric acid, 0.02 N.
7. Procedure
   - 7.1 Pipet 50 ml of the sample into a 250 ml beaker.
   - 7.2 Measure the pH of the sample. If the pH is above 4.0, add standard sulfuric acid (6.3) in 5.0 ml increments to lower the pH to 4.0 or less. If the initial pH of the sample is less than 4.0, the incremental addition of sulfuric acid is not required.
   - 7.3 Add 5 drops of hydrogen peroxide (6.1).
   - 7.4 Heat the sample to boiling and continue boiling for 2 to 4 minutes. In some instances, the concentration of ferrous iron in a sample is such that an additional amount of hydrogen peroxide and a slightly longer boiling time may be required.

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Issued 1971
Technical revision 1974

7.5 Cool the sample to room temperature and titrate electrometrically with standard sodium hydroxide (6.2) to pH 8.2.

8. Calculations

8.1 Acidity, as mg/l $CaCO_3$ = $\frac{[(A \times B) - (C \times D)] \times 50{,}000}{\text{ml of sample}}$

where:

A = vol. of standard sodium hydroxide used in titration
B = normality of standard sodium hydroxide
C = volume of standard sulfuric acid used to reduce pH to 4 or less
D = normality of standard sulfuric acid

8.2 If it is desired to report acidity in millequivalents per liter, the reported values as $CaCO_3$ are divided by 50, as follows:

$$\text{Acidity as meq/l} = \frac{\text{mg/l } CaCO_3}{50}$$

9. Precision

9.1 On a round robin conducted by ASTM on 4 acid mine waters, including concentrations up to 2000 mg/l, the precision was found to be ±10 mg/l.

Bibliography

1. Annual Book of ASTM Standards, Part 31, "Water", p 116, D 1067, Method E(1976).
2. Standard Methods for the Examination of Water and Wastewater, 14th Edition, p 277, Method 402(4d) (1975).

RESIDUE, NON-FILTERABLE

Method 160.2 (Gravimetric, Dried at 103–105°C)

STORET NO. 00530

1. Scope and Application
   - 1.1 This method is applicable to drinking, surface, and saline waters, domestic and industrial wastes.
   - 1.2 The practical range of the determination is 4 mg/l to 20,000 mg/l.
2. Summary of Method
   - 2.1 A well-mixed sample is filtered through a glass fiber filter, and the residue retained on the filter is dried to constant weight at 103–105°C.
   - 2.2 The filtrate from this method may be used for Residue, Filterable.
3. Definitions
   - 3.1 Residue, non-filterable, is defined as those solids which are retained by a glass fiber filter and dried to constant weight at 103–105°C.

Sample Handling and Preservation
   - 4.1 Non-representative particulates such as leaves, sticks, fish, and lumps of fecal matter should be excluded from the sample if it is determined that their inclusion is not desired in the final result.
   - 4.2 Preservation of the sample is not practical; analysis should begin as soon as possible. Refrigeration or icing to 4°C, to minimize microbiological decomposition of solids, is recommended.

5. Interferences
   - 5.1 Filtration apparatus, filter material, pre-washing, post-washing, and drying temperature are specified because these variables have been shown to affect the results.
   - 5.2 Samples high in Filterable Residue (dissolved solids), such as saline waters, brines and some wastes, may be subject to a positive interference. Care must be taken in selecting the filtering apparatus so that washing of the filter and any dissolved solids in the filter (7.5) minimizes this potential interference.
6. Apparatus
   - 6.1 Glass fiber filter discs, without organic binder, such as Millipore AP-40, Reeves Angel 934-AH, Gelman type A/E, or equivalent.

     NOTE: Because of the physical nature of glass fiber filters, the absolute pore size cannot be controlled or measured. Terms such as "pore size", collection efficiencies and effective retention are used to define this property in glass fiber filters. Values for these parameters vary for the filters listed above.
   - 6.2 Filter support: filtering apparatus with reservoir and a coarse (40–60 microns) fritted disc as a filter support.

Approved for NPDES

[illegible]

NOTE: Many funnel designs are available in glass or porcelain. Some of the most common are Hirsch or Buchner funnels, membrane filter holders and Gooch crucibles. All are available with coarse fritted disc.

6.3 Suction flask.

6.4 Drying oven, 103-105°C.

6.5 Desiccator.

6.6 Analytical balance, capable of weighing to 0.1 mg.

7. Procedure

7.1 Preparation of glass fiber filter disc: Place the glass fiber filter on the membrane filter apparatus or insert into bottom of a suitable Gooch crucible with wrinkled surface up. While vacuum is applied, wash the disc with three successive 20 ml volumes of distilled water. Remove all traces of water by continuing to apply vacuum after water has passed through. Remove filter from membrane filter apparatus or both crucible and filter if Gooch crucible is used, and dry in an oven at 103-105°C for one hour. Remove to desiccator and store until needed. Repeat the drying cycle until a constant weight is obtained (weight loss is less than 0.5 mg). Weigh immediately before use. After weighing, handle the filter or crucible/filter with forceps or tongs only.

7.2 Selection of Sample Volume

For a 4.7 cm diameter filter, filter 100 ml of sample. If weight of captured residue is less than 1.0 mg, the sample volume must be increased to provide at least 1.0 mg of residue. If other filter diameters are used, start with a sample volume equal to 7 ml/cm^2 of filter area and collect at least a weight of residue proportional to the 1.0 mg stated above.

NOTE: *If during filtration of this initial volume the filtration rate drops rapidly, or if* filtration time exceeds 5 to 10 minutes, the following scheme is recommended: Use an unweighed glass fiber filter of choice affixed in the filter assembly. Add a known volume of sample to the filter funnel and record the time elapsed after selected volumes have passed through the filter. Twenty-five ml increments for timing are suggested. Continue to record the time and volume increments until fitration rate drops rapidly. Add additional sample if the filter funnel volume is inadequate to reach a reduced rate. Plot the observed time versus volume filtered. Select the proper filtration volume as that just short of the time a significant change in filtration rate occurred.

7.3 Assemble the filtering apparatus and begin suction. Wet the filter with a small volume of distilled water to seat it against the fritted support.

7.4 Shake the sample vigorously and quantitatively transfer the predetermined sample volume selected in 7.2 to the filter using a graduated cylinder. Remove all traces of water by continuing to apply vacuum after sample has passed through.

7.5 With suction on, wash the graduated cylinder, filter, non-filterable residue and filter funnel wall with three portions of distilled water allowing complete drainage between washing. Remove all traces of water by continuing to apply vacuum after water has passed through.

NOTE: Total volume of wash water used should equal approximately 2 ml per cm^2. For a 4.7 cm filter the total volume is 30 ml.

7.6 Carefully remove the filter from the filter support. Alternatively, remove crucible and filter from crucible adapter. Dry at least one hour at 103–105°C. Cool in a desiccator and weigh. Repeat the drying cycle until a constant weight is obtained (weight loss is less than 0.5 mg).

8. Calculations

8.1 Calculate non-filterable residue as follows:

$$\text{Non-filterable residue, mg/l} = \frac{(A - B) \times 1{,}000}{C}$$

where:

A = weight of filter (or filter and crucible) + residue in mg
B = weight of filter (or filter and crucible) in mg
C = ml of sample filtered

9. Precision and Accuracy

9.1 Precision data are not available at this time.

9.2 Accuracy data on actual samples cannot be obtained.

Bibliography

NCASI Technical Bulletin No. 291, March 1977. National Council of the Paper Industry for Air and Stream Improvement, Inc., 260 Madison Ave., NY.

RESIDUE, FILTERABLE

Method 160.1 (Gravimetric, Dried at 180°C)

STORET NO. 70300

1. Scope and Application
   1.1 This method is applicable to drinking, surface, and saline waters, domestic and industrial wastes.
   1.2 The practical range of the determination is 10 mg/l to 20,000 mg/l.
2. Summary of Method
   2.1 A well-mixed sample is filtered through a standard glass fiber filter. The filtrate is evaporated and dried to constant weight at 180°C.
   2.2 If Residue, Non-Filterable is being determined, the filtrate from that method may be used for Residue, Filterable.
3. Definitions
   3.1 Filterable residue is defined as those solids capable of passing through a glass fiber filter and dried to constant weight at 180°C.
4. Sample Handling and Preservation
   4.1 Preservation of the sample is not practical; analysis should begin as soon as possible. Refrigeration or icing to 4°C, to minimize microbiological decomposition of solids, is recommended.
5. Interferences
   5.1 Highly mineralized waters containing significant concentrations of calcium, magnesium, chloride and/or sulfate may be hygroscopic and will require prolonged drying, desiccation and rapid weighing.
   5.2 Samples containing high concentrations of bicarbonate will require careful and possibly prolonged drying at 180°C to insure that all the bicarbonate is converted to carbonate.
   5.3 Too much residue in the evaporating dish will crust over and entrap water that will not be driven off during drying. Total residue should be limited to about 200 mg.
6. Apparatus
   6.1 Glass fiber filter discs, 4.7 cm or 2.1 cm, without organic binder, Reeve Angel type 934-AH, Gelman type A/E, or equivalent.
   6.2 Filter holder, membrane filter funnel or Gooch crucible adapter.
   6.3 Suction flask, 500 ml.
   6.4 Gooch crucibles, 25 ml (if 2.1 cm filter is used).
   6.5 Evaporating dishes, porcelain, 100 ml volume. (Vycor or platinum dishes may be substituted).
   6.6 Steam bath.
   6.7 Drying oven, 180°C ±2°C.
   6.8 Desiccator.

Approved for NPDES
Issued 1971

6.9 Analytical balance, capable of weighing to 0.1 mg.

7. Procedure

7.1 Preparation of glass fiber filter disc: Place the disc on the membrane filter apparatus or insert into bottom of a suitable Gooch crucible. While vacuum is applied, wash the disc with three successive 20 ml volumes of distilled water. Remove all traces of water by continuing to apply vacuum after water has passed through. Discard washings.

7.2 Preparation of evaporating dishes: If Volatile Residue is also to be measured heat the clean dish to 550 ±50°C for one hour in a muffle furnace. If only Filterable Residue is to be measured heat the clean dish to 180 ±2°C for one hour. Cool in desiccator and store until needed. Weigh immediately before use.

7.3 Assemble the filtering apparatus and begin suction. Shake the sample vigorously and rapidly transfer 100 ml to the funnel by means of a 100 ml graduated cylinder. If total filterable residue is low, a larger volume may be filtered.

7.4 Filter the sample through the glass fiber filter, rinse with three 10 ml portions of distilled water and continue to apply vacuum for about 3 minutes after filtration is complete to remove as much water as possible.

7.5 Transfer 100 ml (or a larger volume) of the filtrate to a weighed evaporating dish and evaporate to dryness on a steam bath.

7.6 Dry the evaporated sample for at least one hour at 180 ±2°C. Cool in a desiccator and weigh. Repeat the drying cycle until a constant weight is obtained or until weight loss is less than 0.5 mg.

8. Calculation

8.1 Calculate filterable residue as follows:

$$\text{Filterable residue, mg/l} = \frac{(A - B) \times 1{,}000}{C}$$

where:

A = weight of dried residue + dish in mg
B = weight of dish in mg
C = volume of sample used in ml

9. Precision and Accuracy

9.1 Precision and accuracy are not available at this time.

Bibliography

1. Standard Methods for the Examination of Water and Wastewater, 14th Edition, p 92, Method 208B, (1975).

EPA 415.1

ORGANIC CARBON

(Total and Dissolved)

STORET NO. Total 00680

Dissolved 00681

1. Scope and Application

1.1 This method includes the measurement of organic carbon in drinking, surface, and saline waters, domestic and industrial wastes. Exclusions are noted under Definitions and Interferences.

1.2 The method is most applicable to measurement of organic carbon above 1 mg/l.

2. Summary of Method

2.1 Organic carbon in a sample is converted to carbon dioxide (CO_2) by catalytic combustion or wet chemical oxidation. The CO_2 formed can be measured directly by an infrared detector or converted to methane (CH_4) and measured by a flame ionization detector. The amount of CO_2 or CH_4 is directly proportional to the concentration of carbonaceous material in the sample.

3. Definitions

3.1 The carbonaceous analyzer measures all of the carbon in a sample. Because of various properties of carbon-containing compounds in liquid samples, preliminary treatment of the sample prior to analysis dictates the definition of the carbon as it is measured. Forms of carbon that are measured by the method are.

A) soluble, nonvolatile organic carbon; for instance, natural sugars.

B) soluble, volatile organic carbon; for instance, mercaptans.

C) insoluble, partially volatile carbon; for instance, oils.

D) insoluble, particulate carbonaceous materials, for instance, cellulose fibers.

E) soluble or insoluble carbonaceous materials adsorbed or entrapped on insoluble inorganic suspended matter, for instance, oily matter *adsorbed on silt particles.*

3.2 The final usefulness of the carbon measurement is in assessing the potential oxygen-demanding load of organic material on a receiving stream. This statement applies whether the carbon measurement is made on a sewage plant effluent, industrial waste, or on water taken directly from the stream. In this light, carbonate and bicarbonate carbon are not a part of the oxygen demand in the stream and therefore should be discounted in the final calculation or removed prior to analysis. The manner of preliminary treatment of the sample and instrument settings defines the types of carbon which are measured. Instrument manufacturer's instructions should be followed.

4. Sample Handling and Preservation

4.1 Sampling and storage of samples in glass bottles is preferable. Sampling and storage in plastic bottles such as conventional polyethylene and cubitainers is permissible if it is established that the containers do not contribute contaminating organics to the samples.

NOTE 1: A brief study performed in the EPA Laboratory indicated that distilled water stored in new, one quart cubitainers did not show any increase in organic carbon after two weeks exposure.

4.2 Because of the possibility of oxidation or bacterial decomposition of some components of aqueous samples, the lapse of time between collection of samples and start of analysis should be kept to a minimum. Also, samples should be kept cool (4°C) and protected from sunlight and atmospheric oxygen.

4.3 In instances where analysis cannot be performed within two hours (2 hours) from time of sampling, it is recommended that the sample is acidified ($pH \leq 2$) with HCl or H_2SO_4.

5. *Interferences*

5.1 Carbonate and bicarbonate carbon represent an interference under the terms of this test and must be removed or accounted for in the final calculation.

5.2 This procedure is applicable only to homogeneous samples which can be injected into the apparatus reproducibly by means of a microliter type syringe or pipette. The openings of the syringe or pipette limit the maximum size of particles which may be included in the sample.

6. Apparatus

6.1 Apparatus for blending or homogenizing samples: Generally, a Waring-type blender is satisfactory.

6.2 Apparatus for total and dissolved organic carbon:

6.2.1 A number of companies manufacture systems for measuring carbonaceous material in liquid samples. Considerations should be made as to the types of samples to be analyzed, the expected concentration range, and forms of carbon to be measured.

6.2.2 No specific analyzer is recommended as superior. However, analyzers which have been found to be reliable are the Dow-Beckman Carbonaceous Analyzer Model No. 915, the Dohrmann Envirotech DC-50 Carbon Analyzer and the Oceanography International Total Carbon Analyzer.

7. Reagents

7.1 Distilled water used in preparation of standards and for dilution of samples should be ultra pure to reduce the size of the blank. Carbon dioxide-free, double distilled water is recommended. Ion exchanged waters are not recommended because of the possibilities of contamination with organic materials from the resins.

7.2 Potassium hydrogen phthalate, stock solution, 1000 mg carbon/liter: Dissolve 0.2128 g of potassium hydrogen phthalate (Primary Standard Grade) in distilled water and dilute to 100.0 ml.

NOTE 2: Sodium oxalate and acetic acid are not recommended as stock solutions.

7.3 Potassium hydrogen phthalate, standard solutions: Prepare standard solutions from the stock solution by dilution with distilled water.

7.4 Carbonate-bicarbonate, stock solution, 1000 mg carbon/liter: Weigh 0.3500 g of sodium bicarbonate and 0.4418 g of sodium carbonate and transfer both to the same 100 ml volumetric flask. Dissolve with distilled water.

7.5 Carbonate-bicarbonate, standard solution: Prepare a series of standards similar to step 7.3.

NOTE 3: This standard is not required by some instruments.

7.6 Blank solution: Use the same distilled water (or similar quality water) used for the preparation of the standard solutions.

8. Procedure

8.1 Follow instrument manufacturer's instructions for calibration, procedure, and calculations.

8.2 For calibration of the instrument, it is recommended that a series of standards encompassing the expected concentration range of the samples be used.

9. Precision and Accuracy

9.1 Twenty-eight analysts in twenty-one laboratories analyzed distilled water solutions containing exact increments of oxidizable organic compounds, with the following results:

Increment as TOC mg/liter	Precision as Standard Deviation TOC, mg/liter	Accuracy as	
		Bias, %	Bias, mg/liter
4.9	3.93	+15.27	+0.75
107	8.32	+ 1.01	+1.08

(FWPCA Method Study 3, Demand Analyses)

~~remove ferric chloride color. The procedure is applicable at sulfide concentrations up to 20 mg/L.~~

~~Potentiometric methods utilizing a silver electrode may be suitable. From the potential of the electrode relative to a reference electrode an estimate can be made of the sulfide concentration, but careful attention to details of procedures and frequent standardizations are needed to secure good results. The electrode is useful particularly as an end-point indicator for titration of dissolved sulfide with silver nitrate.~~

~~Figure 427:1 shows analytical flow paths for sulfide determinations under various conditions and options.~~

~~427 A. Separation of Soluble and Insoluble Sulfides~~

~~Unless the sample is entirely free from suspended solids (dissolved sulfide equals total sulfide), to measure dissolved sulfide first remove insoluble matter. This can be done by producing an aluminum hydroxide floc that is settled, leaving a clear supernatant for analysis.~~

~~1. Apparatus~~

~~*Glass bottles with stoppers.* Use 100 mL if sulfide will be determined by the methylene blue method and 500 to 1000 mL if by the iodometric method.~~

~~2. Reagents~~

~~*a. Sodium hydroxide solution,* NaOH, 6*N*.~~

~~*b. Aluminum chloride solution,* 6*N*: Because of the hygroscopic and caking tendencies of this chemical, purchase 100-g bottles of $AlCl_3 \cdot 6H_2O$. Dissolve contents of a previously unopened 100-g bottle in 144 mL distilled water.~~

~~3. Procedure~~

~~*a.* To a 100-mL glass bottle add 0.2 mL (4 drops) 6*N* NaOH. Fill bottle with sample and add 0.2 mL (4 drops) 6*N* $AlCl_3$. Stopper bottle with no air under stopper. Rotate back and forth about a transverse axis vigorously for 1 min or longer to flocculate contents. Vary volumes of these chemicals to get good clarification without using excessively large amounts and to produce a pH of 6 to 9. If a 500- or 1000-mL bottle is used, add proportionally larger amounts of reagents.~~

~~*b.* Let settle until reasonably clear supernatant can be drawn off. With proper flocculation, this may take 5 to 15 min. Do not wait longer than necessary.~~

427 B. Sample Pretreatment to Remove Interfering Substances or to Concentrate the Sulfide

The iodometric method suffers interference from reducing substances that react with iodine, including thiosulfate, sulfite, and various organic compounds, both solid and dissolved.

Strong reducing agents also interfere in the methylene blue test by preventing formation of the blue color. Thiosulfate at concentrations above 10 mg/L may retard color formation or completely prevent it.

Sulfide itself prevents the reaction if its concentration is very high, in the range of several hundred milligrams per liter. To avoid the possibility of false negative results, use the antimony method to obtain a qualitative result in industrial wastes likely to contain sulfide but showing no color by the methylene blue method. Iodide, which is likely to be present in oil-field wastewaters, may diminish color formation if its concentration exceeds 2 mg/L. Ferrocyanide produces a blue color.

Eliminate interferences due to sulfite, thiosulfate, iodide, and many other soluble substances, but not ferrocyanide, by first precipitating ZnS, removing the supernatant, and replacing it with distilled water. Use the same procedure, even when not needed for removal of interferences, to concentrate sulfide.

1. Apparatus

Glass bottles with stoppers (see Section 427A).

2. Reagents

a. Zinc acetate, 2*N*: Dissolve 220 g $Zn(C_2H_3O_2)_2 \cdot 2H_2O$ in 870 mL water; this makes 1 L solution.

b. Sodium hydroxide solution, NaOH, 6*N*.

3. Procedure

a. Put 0.15 mL (3 drops) 2*N* zinc acetate solution into a 100-mL glass bottle, fill with sample, and add 0.10 mL (2 drops) 6*N* NaOH solution. Stopper with no air bubbles under stopper and mix by rotating back and forth vigorously about a transverse axis. For the iodometric procedure, use a 500-mL bottle or other convenient size, with proportionally larger volumes of reagents. Vary volume of reagents added according to sample so that the resulting precipitate is not excessively bulky and settles readily. Add enough NaOH to produce a pH above 9. Let precipitate settle for 30 min. The treated sample is relatively stable and can be held for several hours. However, if much iron is present, oxidation may be fairly rapid.

b. If the iodometric method is to be used, filter precipitate through glass fiber filter paper and continue at once with titration according to the procedure of Section 427D. If the methylene blue method is used, let precipitate settle for 30 min and decant as much supernatant as possible without loss of precipitate. Refill bottle with distilled water, resuspend precipitate, and withdraw a sample. If interfering substances are present in high concentration, settle, decant, and refill a second time. If sulfide concentration is known to be low, add only enough water to bring volume to one-half or one-fifth of original volume. Use this technique for analyzing samples of very low sulfide concentrations. After determining the sulfide concentration colorimetrically, multiply the result by the ratio of final to initial volume.

Cadmium salts sometimes are used instead of zinc, but CdS is more susceptible to oxidation than ZnS.

~~marks on test tubes Add to mL amine-sulfuric acid reagent and 0.15 mL (3 drops) $FeCl_3$ solution. Mix immediately by inverting slowly, only once. (Excessive mixing causes low results by loss of H_2S as a gas before it has had time to react). To Tube B add 0.5 mL 1 + 1 H_2SO_4 and 0.15 mL (3 drops) $FeCl_3$ solution and mix. The presence of S^{2-} will be indicated by the appearance of blue color in Tube A. Color development usually is complete in about 1 min, but a longer time often is required for fading out of the initial pink color. Wait 3 to 5 min and add 1.6 mL $(NH_4)_2HPO_4$ solution to each tube. Wait 3 to 15 min and make color comparisons. If zinc acetate was used, wait at least 10 min before making a visual color comparison.~~

~~*b. Color determination:*~~

~~1) Visual color estimation—Add methylene blue solution I or II, depending on sulfide concentration and desired accuracy, dropwise, to the second tube, until color matches that developed in first tube. If the concentration exceeds 20 mg/L, repeat test with a portion of sample diluted to one tenth.~~

~~With methylene blue solution I, adjusted mL (1 drop) = 1.0 mg S^{2-}/L when 7.5 mL of sample are used.~~

$$\text{mg } S^{2-}/L = \text{no. drops solution I} + 0.1\ (\text{no. drops solution II})$$

~~2) Photometric color measurement—A cell with a light path of 1 cm is suitable for measuring sulfide concentrations from 0.1 to 2.0 mg/L. Use shorter or longer light paths for higher or lower concentrations. The upper limit of the method is 20 mg/L. Zero instrument with a portion of treated sample from Tube B Prepare calibration curves on basis of colorimetric tests made on Na_2S solutions simultaneously analyzed by the iodometric method, plotting concentration vs. absorbance. A straight-line relationship between concentration and absorbance can be assumed from 0 to 1.0 mg/L.~~

~~Read sulfide concentration from calibration curve.~~

~~4. Precision and Accuracy~~

~~The accuracy is about ±10%. The standard deviation has not been determined.~~

427 D. Iodometric Method

1. Reagents

a. Hydrochloric acid, HCl, 6*N*.

b. Standard iodine solution, 0.0250*N*: Dissolve 20 to 25 g KI in a little water and add 3.2 g iodine. After iodine has dissolved, dilute to 1000 mL and standardize against 0.0250*N* $Na_2S_2O_3$, using starch solution as indicator.

c. Standard sodium thiosulfate solution, 0.0250*N*: See Section 421B.2*e*.

d. Starch solution: See Section 421B.2*d*.

2. Procedure

a. Measure from a buret into a 500-mL flask an amount of iodine solution estimated to be an excess over the amount of sulfide present. Add distilled water, if necessary, to bring volume to about 20 mL. Add 2 mL 6*N* HCl. Pipet 200 mL sample into flask, discharging sample under solution surface. If iodine color disappears, add more iodine so that color remains Back-titrate with $Na_2S_2O_3$ solution, adding a few drops of starch solution as end point is approached, and continuing until blue color disappears.

b. If sulfide was precipitated with zinc and ZnS filtered out, return filter with precipitate to original bottle and add about 100 mL water. Add iodine solution and HCl and titrate as in ¶ 2*a* above.

3. Calculation

One milliliter 0.0250*N* iodine solution reacts with 0.4 mg S^{2-}:

$$\text{mg } S^{2-}/\text{L} = \frac{[(A \times B) - (C \times D)] \times 16\,000}{\text{mL sample}}$$

where:

A = mL iodine solution,
B = normality of iodine solution,
C = mL $Na_2S_2O_3$ solution, and
D = normality of $Na_2S_2O_3$ solution

4. Precision

The precision of the end point varies with the sample. In clean waters it should be determinable within 1 drop, which is equivalent to 0.1 mg/L in a 200-mL sample.

427 E. Calculation of Un-ionized Hydrogen Sulfide

Hydrogen sulfide and HS^-, which together constitute dissolved sulfide, are in equilibrium with hydrogen ions:

$$H_2S \rightleftharpoons H^+ + HS^-$$

The ionization constant of H_2S is used to calculate the distribution of dissolved sulfide between the two forms. The practical constant written in logarithmic form, pK', is used. The constant varies with temperature and ionic strength of the solution. The ionic strength effect can be estimated most easily from the conductivity. Because the effect of ionic strength is not large, values that are sufficiently dependable generally can be assumed if the nature of the sample is known. Table 427:I gives approximate pK' values for various temperatures and conductivities. The temperature effect is practically linear from 15°C to 35°C; interpolations or extrapolations can be used. The last line of Table 427:I corresponds approximately to seawater.

From sample pH and appropriate value of pK', calculate pH − pK'. From Figure 427:2 read proportions of dissolved sulfide present as H_2S (left-side scale of Figure 427:2). Let this proportion equal J,

$$J \times (\text{dissolved sulfide}) = \text{un-ionized } H_2S \text{ expressed as } S^{2-}$$

TABLE 427:I. VALUES OF pK', LOGARITHM OF PRACTICAL IONIZATION CONSTANT FOR HYDROGEN SULFIDE

Conductivity at 25°C *µmhos/cm*	pK' at Given Temperature		
	20°C	25°C	30°C
0	—	7.03*	—
100	7.08	7.01	6.94
200	7.07	7.00	6.93
400	7.06	6.99	6.92
700	7.05	6.98	6.91
1 200	7.04	6.97	6.90
2 000	7.03	6.96	6.89
3 000	7.02	6.95	6.88
4 000	7.01	6.94	6.87
5 200	7.00	6.93	6.86
7 200	6.99	6.92	6.85
10 000	6.98	6.91	6.84
14 000	6.97	6.90	6.83
22 000	6.96	6.89	6.82
50 000	6.95	6.88	6.81

*Theoretical.

LANGELIER, W F 1946 Effect of temperature on the pH of natural water. *J. Amer. Water Works Ass* 38:179

FELDMAN, I. 1956 Use and abuse of pH measurements *Anal. Chem.* 28:1859

BRITTON, H.T.S. 1956 Hydrogen Ions, 4th ed. D Van Nostrand Co., Princeton, N.J.

KOLTHOFF, I.M. & H.A. LAITINEN. 1958 pH and Electrotitrations. John Wiley & Sons, New York, N.Y.

KOLTHOFF, I M & P.J. ELVING 1959 Treatise on Analytical Chemistry Part I, Vol 1, Chapter 10. Wiley-Interscience, New York, N Y

BATES, R.G 1962. Revised standard values for pH measurements from 0 to 95°C *J. Res. Nat. Bur. Standards* 66A:179

AMERICAN WATER WORKS ASSOCIATION. 1964 Simplified Procedures for Water Examination. Manual M12, American Water Works Ass., New York, N.Y.

WINSTEAD, M. 1967. Reagent Grade Water: How, When and Why? American Soc. Medical Technologists, The Steck Company, Austin, Tex.

STAPLES, B.R. & R.G. BATES. 1969. Two new standards for the pH scale. *J. Res. Nat. Bur. Standards* 73A:37.

BATES, R.G 1973. Determination of pH. Theory and Practice, 2nd ed. John Wiley & Sons, New York, N.Y.

424 PHOSPHORUS*

Phosphorus occurs in natural waters and in wastewaters almost solely as phosphates. These are classified as orthophosphates, condensed phosphates (pyro-, meta-, and other polyphosphates), and organically bound phosphates. They occur in solution, in particles or detritus, or in the bodies of aquatic organisms.

These forms of phosphate arise from a variety of sources. Small amounts of certain condensed phosphates are added to some water supplies during treatment. Larger quantities of the same compounds may be added when the water is used for laundering or other cleaning, because these materials are major constituents of many commercial cleaning preparations. Phosphates are used extensively in the treatment of boiler waters. Orthophosphates applied to agricultural or residential cultivated land as fertilizers are carried into surface waters with storm runoff and to a lesser extent with melting snow. Organic phosphates are formed primarily by biological processes. They are contributed to sewage by body wastes and food residues, and also may be formed from orthophosphates in biological treatment processes or by receiving water biota.

Phosphorus is essential to the growth of organisms and can be the nutrient that limits the primary productivity of a body of water. In instances where phosphate is a growth-limiting nutrient, the discharge of raw or treated wastewater, agricultural drainage, or certain industrial wastes to that water may stimulate the growth of photosynthetic aquatic micro- and macroorganisms in nuisance quantities.

Phosphates also occur in bottom sediments and in biological sludges, both as precipitated inorganic forms and incorporated into organic compounds.

1 Definition of Terms

Phosphorus analyses embody two general procedural steps: (*a*) conversion of the phosphorus form of interest to dissolved orthophosphate, and (*b*) colorimetric determination of dissolved orthophosphate. The separation of phosphorus into its various forms is defined analytically but the analytical differentiations have been selected so that they may be used for interpretive purposes.

Filtration through a 0.45-µm-pore-diam

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*Approved by Standard Methods Committee, 1981 membrane filter separates dissolved from suspended forms of phosphorus. No claim is made that filtration through 0.45-μm filters is a true separation of suspended and dissolved forms of phosphorus; it is merely a convenient and replicable analytical technique designed to make a gross separation.

Membrane filtration is selected over depth filtration because of the greater likelihood of obtaining a consistent separation of particle sizes. Prefiltration through a glass fiber filter may be used to increase the filtration rate.

Phosphates that respond to colorimetric tests without preliminary hydrolysis or oxidative digestion of the sample are termed "reactive phosphorus." While reactive phosphorus is largely a measure of orthophosphate, a small fraction of any condensed phosphate present usually is hydrolyzed unavoidably in the procedure. Reactive phosphorus occurs in both dissolved and suspended forms

Acid hydrolysis at boiling-water temperature converts dissolved and particulate condensed phosphates to dissolved orthophosphate. The hydrolysis unavoidably releases some phosphate from organic compounds, but this may be reduced to a minimum by judicious selection of acid strength and hydrolysis time and temperature. The term "acid-hydrolyzable phosphorus" is preferred over "condensed phosphate" for this fraction.

The phosphate fractions that are converted to orthophosphate only by oxidation destruction of the organic matter present are considered "organic" or "organically bound" phosphorus. The severity of the oxidation required for this conversion depends on the form—and to some extent on the amount—of the organic phosphorus present. Like reactive phosphorus and acid-hydrolyzable phosphorus, organic phosphorus occurs both in the dissolved and suspended fractions.

The total phosphorus as well as the dissolved and suspended phosphorus fractions each may be divided analytically into the three chemical types that have been described reactive, acid-hydrolyzable, and organic phosphorus. Figure 424:1 shows the steps for analysis of individual phosphorus fractions. As indicated, determinations usually are conducted only on the unfiltered and filtered samples. Suspended fractions generally are determined by difference.

2. Selection of Method

a. Digestion methods: Because phosphorus may occur in combination with organic matter, a digestion method to determine total phosphorus must be able to oxidize organic matter effectively to release phosphorus as orthophosphate. Three digestion methods are given. The perchloric acid method, the most drastic and time-consuming method, is recommended only for particularly difficult samples such as sediments. The nitric acid-sulfuric acid method is recommended for most samples. By far the simplest method is the persulfate oxidation technique It is recommended that this method be checked against one or more of the more drastic digestion techniques and be adopted if identical recoveries are obtained

b. Colorimetric methods: Three methods of orthophosphate determination are described. Selection depends largely on the concentration range of orthophosphate. The vanadomolybdophosphoric acid method (D) is most useful for routine analyses in the range of 1 to 20 mg P/L. The stannous chloride method (E) or the ascorbic acid method (F) is more suited for the range of 0.01 to 6 mg P/L. An extraction step is recommended for the lower levels of this range and when interferences must be overcome An automated version of the ascorbic acid method also is presented

3. Precision and Accuracy

To aid in method selection, Table 424 I presents the results of various combina-

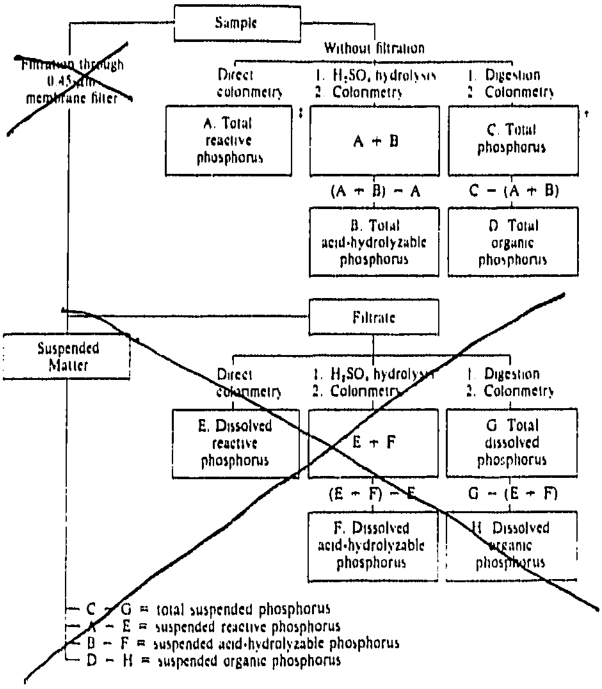

Figure 424:1. Steps for analysis of phosphate fractions.

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[*] Direct determination of phosphorus on the membrane filter containing suspended matter will be required where greater precision than that obtained by difference is desired. Digest filter with HNO_3 and follow by perchloric acid. Then perform colorimetry.

[†] Total phosphorus measurements on highly saline samples may be difficult because of precipitation of large quantities of salt as a result of digestion techniques that drastically reduce sample volume. For total phosphorus analyses on such samples, directly determine total dissolved phosphorus and total suspended phosphorus and add the results.

[‡] When determining total dissolved or total suspended reactive phosphorus, anomalous results may be obtained on samples containing large amounts of suspended sediments. Very often results depend largely on the degree of agitation and mixing to which samples are subjected during analysis because of a time-dependent desorption of orthophosphate from the suspended particles.

424 B. Preliminary Acid Hydrolysis Step for Acid-Hydrolyzable Phosphorus

1. Discussion

The acid-hydrolyzable phosphorus content of the sample is defined operationally as the difference between reactive phosphorus as measured in the untreated sample and phosphate found after mild acid hydrolysis. Generally, it includes condensed phosphates such as pyro-, tripoly-, and higher-molecular-weight species such as hexametaphosphate. In addition, some natural waters contain organic phosphate compounds that are hydrolyzed to orthophosphate under the test conditions. Polyphosphates generally do not respond to reactive phosphorus tests but can be hydrolyzed to orthophosphate by boiling with acid.

After hydrolysis, determine reactive phosphorus by a colorimetric method (D, E, or F). Interferences, precision, accuracy, and sensitivity will depend on the colorimetric method used.

2. Apparatus

Autoclave or pressure cooker, capable of operating at 98 to 137 kPa.

3. Reagents

a. Phenolphthalein indicator aqueous solution.

b. Strong acid solution: Slowly add 300 mL conc H_2SO_4 to about 600 mL distilled water. When cool, add 4.0 mL conc HNO_3 and dilute to 1 L.

c. Sodium hydroxide, NaOH, 6N.

4. Procedure

To 100-mL sample or a portion diluted to 100 mL, add 0.05 mL (1 drop) phenolphthalein indicator solution. If a red color develops, add strong acid solution dropwise, to just discharge the color. Then add 1 mL more.

Boil gently for at least 90 min, adding distilled water to keep the volume between 25 and 50 mL. Alternatively, heat for 30 min in an autoclave or pressure cooker at 98 to 137 kPa. Cool, neutralize to a faint pink color with NaOH solution, and restore to the original 100-mL volume with distilled water.

Prepare a calibration curve by carrying a series of standards containing orthophosphate (see colorimetric method D, E, or F) through the hydrolysis step. Do not use orthophosphate standards without hydrolysis, because the salts added in hydrolysis cause an increase in the color intensity in some methods.

Determine reactive phosphorus content of treated portions, using Method D, E, or F. This gives the sum of polyphosphate and orthophosphate in the sample. To calculate its content of acid-hydrolyzable phosphorus, determine reactive phosphorus in a sample portion that has not been hydrolyzed, using the same colorimetric method as for treated sample, and subtract.

424 C. Preliminary Digestion Steps for Total Phosphorus

Total phosphorus includes all orthophosphates and condensed phosphates, both dissolved and particulate, organic and inorganic. To release phosphorus from combination with organic matter, digest and oxidize. The rigor of digestion required depends on the type of sample. The three digestion techniques presented, in order of decreasing rigor, are perchloric acid digestion, sulfuric acid-nitric acid digestion, and persulfate digestion. Compare phosphorus recovery by each digestion technique for the specific type of sample being tested; if the less tedious persulfate method gives good phosphorus recovery, use this method.

After digestion, determine liberated orthophosphate by Method D, E, or F. The colorimetric method used, rather than the digestion procedure, governs in matters of interference and minimum detectable concentration.

I—PERCHLORIC ACID DIGESTION

1. Apparatus

a. Hot plate: A 30- × 50-cm heating surface is adequate.

b. Safety shield.

c. Safety goggles.

d. Erlenmeyer flasks, 125-mL, acid-washed and rinsed with distilled water.

2. Reagents

a. Nitric acid, HNO_3, conc.

b. Perchloric acid, $HClO_4 \cdot 2H_2O$, purchased as 70 to 72% $HClO_4$, reagent-grade.

c. Sodium hydroxide, NaOH, 6*N*.

d. Methyl orange indicator solution.

e. Phenolphthalein indicator aqueous solution.

3. Procedure

CAUTION—*Heated mixtures of $HClO_4$ and organic matter may explode violently. Avoid this hazard by taking the following precautions: (a) Do not add $HClO_4$ to a hot solution that may contain organic matter. (b) Always initiate digestion of samples containing organic matter with HNO_3. Complete digestion using the mixture of HNO_3 and $HClO_4$. (c) Do not fume with $HClO_4$ in ordinary hoods. Use hoods especially constructed for $HClO_4$ fuming or a glass fume eradicator* connected to a water pump. (d) Never let samples being digested with $HClO_4$ evaporate to dryness.*

Measure sample containing the desired amount of phosphorus (this will be determined by whether Method D, E, or F is to be used) into a 125-mL erlenmeyer flask. Acidify to methyl orange with conc HNO_3, add another 5 mL conc HNO_3, and evaporate on a steam bath or hot plate to 15 to 20 mL.

Add 10 mL each of conc HNO_3 and $HClO_4$ to the 125-mL conical flask, cooling the flask between additions. Add a few boiling chips, heat on a hot plate, and evaporate gently until dense white fumes of $HClO_4$ just appear. If solution is not clear, cover neck of flask with a watch glass and keep solution barely boiling until it clears. If necessary, add 10 mL more HNO_3 to aid oxidation.

Cool digested solution and add 1 drop aqueous phenolphthalein solution. Add 6*N* NaOH solution until the solution just turns pink. If necessary, filter neutralized solution and wash filter liberally with distilled water. Make up to 100 mL with distilled water.

Determine the PO_4^{3-}-P content of the treated sample by Method D, E, or F.

Prepare a calibration curve by carrying a series of standards containing orthophosphate (see Method D, E, or F) through digestion step. Do not use orthophosphate standards without treatment.

II—SULFURIC ACID-NITRIC ACID DIGESTION

1. Apparatus

a. Digestion rack: An electrically or gas-heated digestion rack with provision for withdrawal of fumes is recommended.

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*G. F. Smith Chemical Co., Columbus, Ohio, or equivalent.

~~Digestion racks typical of those used for micro-kjeldahl digestions are suitable.~~

~~*b. Micro-kjeldahl flasks.*~~

~~2. Reagents~~

~~*a. Sulfuric acid,* H_2SO_4, conc.~~

~~*b. Nitric acid,* HNO_3, conc.~~

~~*c. Phenolphthalein indicator aqueous solution.*~~

~~*d. Sodium hydroxide,* NaOH, 1*N*.~~

~~3. Procedure~~

~~Into a micro-kjeldahl flask, measure a sample containing the desired amount of phosphorus (this is determined by the colorimetric method used). Add 1 mL conc H_2SO_4 and 5 mL conc HNO_3.~~

~~Digest to a volume of 1 mL and then continue until solution becomes colorless to remove HNO_3.~~

~~Cool and add approximately 20 mL distilled water, *0.05 mL (1 drop) phenolphthalein indicator, and as much* 1*N* NaOH solution as required to produce a faint pink tinge. Transfer neutralized solution, filtering if necessary to remove particulate material or turbidity, into a 100-mL volumetric flask. Add filter washings to flask and adjust sample volume to 100 mL with distilled water.~~

~~Determine phosphorus by Method D, E, or F, for which a separate calibration curve has been constructed by carrying standards through the acid digestion procedure.~~

III. PERSULFATE DIGESTION METHOD

1. Apparatus

a. Hot plate: A 30- × 50-cm heating surface is adequate.

b. Autoclave: An autoclave or pressure cooker capable of developing 98 to 137 kPa may be used in place of a hot plate.

c. Glass scoop, to hold required amounts of persulfate crystals.

2. Reagents

a. Phenolphthalein indicator aqueous solution.

b. Sulfuric acid solution: Carefully add 300 mL conc H_2SO_4 *to approximately 600* mL distilled water and dilute to 1 L with distilled water.

c. Ammonium persulfate, $(NH_4)_2S_2O_8$, solid, or potassium persulfate, $K_2S_2O_8$, solid.

d. Sodium hydroxide, NaOH, 1*N*.

3. Procedure

Use 50 mL or a suitable portion of thoroughly mixed sample. *Add 0.05 mL (1 drop) phenolphthalein indicator solution. If a red color develops,* add H_2SO_4 solution dropwise to just discharge the color. Then add 1 mL H_2SO_4 solution and either 0.4 g solid $(NH_4)_2S_2O_8$ or 0.5 g solid $K_2S_2O_8$.

Boil gently on a preheated hot plate for 30 to 40 min or until a final volume of 10 mL is reached. Cool, dilute to 30 mL with distilled water, add 0.05 mL (1 drop) phenolphthalein indicator solution, and neutralize to a faint pink color with NaOH. Alternatively, heat for 30 min in an autoclave or pressure cooker at 98 to 137 kPa. *Cool, add 0.05 mL (1 drop) phenolphthalein indicator solution,* and neutralize to a faint pink color with NaOH. Make up to 100 mL with distilled water. In some samples a precipitate may form at this stage, but do not filter. For any subsequent subdividing of the sample, shake well. The precipitate (which is possibly a calcium phosphate) redissolves under the acid conditions of the colorimetric reactive phosphorus test. Determine phosphorus by Method D, E, or F, for which a separate *calibration curve has been constructed by carrying standards through the persulfate digestion procedure*

~~velops progressively and later fades, maintain equal timing conditions for samples and standards. Prepare at least one standard with each set of samples or once each day that tests are made. The calibration curve may deviate from a straight line at the upper concentrations of the 0.3 to 2.0-mg/L range.~~

~~*d. Extraction:* When increased sensitivity is desired or interferences must be overcome, extract phosphate as follows: Pipet a 40-mL sample, or one diluted to that volume, into a 125-mL separatory funnel. Add 50.0 mL benzene-isobutanol solvent and 15.0 mL molybdate reagent II. Close funnel at once and shake vigorously for exactly 15 s. If condensed phosphate is present, any delay will increase its conversion to orthophosphate. Remove stopper and withdraw 25.0 mL of separated organic layer, using a pipet with safety bulb. Transfer to a 50-mL volumetric flask, add 15 to 16 mL alcoholic H_2SO_4 solution, swirl, add 0.50 mL (10 drops) dilute stannous chloride reagent II, swirl, and dilute to the mark with alcoholic H_2SO_4. Mix thoroughly. After 10 min, but before 30 min, read against the blank at 625 nm. Prepare blank by carrying 40 mL distilled water through the same procedure used for the sample. Read phosphate concentration from a calibration curve prepared by taking known phosphate standards through the same procedure used for samples.~~

~~5. Calculation~~

~~Calculate as follows:~~

~~*a. Direct procedure:*~~

$$\text{mg P/L} = \frac{\text{mg P (in approximately 104.5 mL final volume)} \times 1000}{\text{mL sample}}$$

~~*b. Extraction procedure:*~~

$$\text{mg P/L} = \frac{\text{mg P (in 50 mL final volume)} \times 1000}{\text{mL sample}}$$

6. Precision and Accuracy

See Table 424:I.

424 F. Ascorbic Acid Method

1. General Discussion

a. Principle: Ammonium molybdate and potassium antimonyl tartrate react in acid medium with orthophosphate to form a heteropoly acid—phosphomolybdic acid—that is reduced to intensely colored molybdenum blue by ascorbic acid.

b. Interference: Arsenates react with the molybdate reagent to produce a blue color similar to that formed with phosphate. Concentrations as low as 0.1 mg As/L interfere with the phosphate determination. Hexavalent chromium and NO_2^- interfere to give results about 3% low at concentrations of 1 mg/L and 10 to 15% low at 10 mg/L. Sulfide (Na_2S) and silicate do not interfere at concentrations of 1.0 and 10 mg/L.

c. Minimum detectable concentration: Approximately 10 µg P/L. P ranges are as follows:

Approximate P Range *mg/L*	*Light Path cm*
0.30–2.0	0.5
0.15–1.30	1.0
0.01–0.25	5.0

2. Apparatus

a. Colorimetric equipment: One of the following is required:

1) *Spectrophotometer,* with infrared phototube for use at 880 nm, providing a light path of 2.5 cm or longer.

2) *Filter photometer,* equipped with a red color filter and a light path of 0.5 cm or longer.

b. Acid-washed glassware: See Section 424D.2*b*.

3. Reagents

a. Sulfuric acid, H_2SO_4, 5*N*: Dilute 70 mL conc H_2SO_4 to 500 mL with distilled water.

b. Potassium antimonyl tartrate solution: Dissolve 1.3715 g $K(SbO)C_4H_4O_6 \cdot 1/2H_2O$ in 400 mL distilled water in a 500-mL volumetric flask and dilute to volume. Store in a glass-stoppered bottle.

c. Ammonium molybdate solution: Dissolve 20 g $(NH_4)_6Mo_7O_{24} \cdot 4H_2O$ in 500 mL distilled water. Store in a glass-stoppered bottle.

d. Ascorbic acid, 0.01*M*: Dissolve 1.76 g ascorbic acid in 100 mL distilled water. The solution is stable for about 1 week at 4°C.

e. Combined reagent: Mix the above reagents in the following proportions for 100 mL of the combined reagent: 50 mL 5*N* H_2SO_4, 5 mL potassium antimonyl tartrate solution, 15 mL ammonium molybdate solution, and 30 mL ascorbic acid solution. *Mix after addition of each reagent.* Let all reagents reach room temperature before they are mixed and mix in the order given. If turbidity forms in the combined reagent, shake and let stand for a few minutes until turbidity disappears before proceeding. The reagent is stable for 4 h.

f. Stock phosphate solution: See Section 424D.3*e*.

g. Standard phosphate solution: Dilute 50.0 mL stock phosphate solution to 1000 mL with distilled water; 1.00 mL = 2.50 μg P.

4. Procedure

a. Treatment of sample: Pipet 50.0 mL sample into a clean, dry test tube or 125-mL erlenmeyer flask. Add 0.05 mL (1 drop) phenolphthalein indicator. If a red color develops add 5*N* H_2SO_4 solution dropwise to just discharge the color. Add 8.0 mL combined reagent and mix thoroughly. After at least 10 min but no more than 30 min, measure absorbance of each sample at 880 nm, using reagent blank as the reference solution.

b. Correction for turbidity or interfering color: Natural color of water generally does not interfere at the high wavelength used. For highly colored or turbid waters, prepare a blank by adding all reagents except ascorbic acid and antimonyl potassium tartrate to the sample. Subtract blank absorbance from absorbance of each sample.

c. Preparation of calibration curve: Prepare individual calibration curves from a series of six standards within the phosphate ranges indicated in Section 424F.1*c*. Use a distilled water blank with the combined reagent to make photometric readings for the calibration curve. Plot absorbance vs. phosphate concentration to give a straight line passing through the origin. Test at least one phosphate standard with each set of samples.

5. Calculation

$$\text{mg P/L} = \frac{\text{mg P (in approximately 58 mL final volume)} \times 1000}{\text{mL sample}}$$

6. Precision and Accuracy

The precision and accuracy values given in Table 424:I are for a single-solution procedure given in the 13th edition. Procedure 424F differs in reagent-to-sample ratios, no addition of solvent, and acidity conditions.

It is superior in precision and accuracy to the previous technique in the analysis of both distilled water and river water at the 228-µg P/L level (Table 424:II).

TABLE 424:II. COMPARISON OF PRECISION AND ACCURACY OF ASCORBIC ACID METHODS

Ascorbic Acid Method	Phosphorus Concentration, Dissolved Orthophosphate µg/L	No of Laboratories	Relative Standard Deviation %		Relative Error %	
			Distilled Water	River Water	Distilled Water	River Water
13th Edition (Edwards, Molof, and Schneeman)	228	8	3.87	2.17	4.01	2.08
Current method (Murphy and Riley)	228	8	3.03	1.75	2.36	1.39

~~424 G. Automated Ascorbic Acid Reduction Method*~~

~~1. General Discussion~~

~~*a. Principle:* Ammonium molybdate and potassium antimonyl tartrate react with orthophosphate in an acid medium to form an antimony-phosphomolybdate complex, which, on reduction with ascorbic acid, yields an intense blue color suitable for photometric measurement.~~

~~*b. Interferences:* As much as 50 mg Fe^{3+}/L, 10 mg Cu/L, and 10 mg SiO_2/L can be tolerated. High silica concentrations cause positive interference.~~

~~In terms of phosphorus, the results are high by 0.005, 0.015, and 0.025 mg/L for silica concentrations of 20, 50, and 100 mg/L, respectively. Salt concentrations up to 20% (w/v) cause an error of less than 1%. Arsenate (AsO_4^{3-}) is a positive interference.~~

~~Eliminate interference from NO_2^- and S^{2-} by adding an excess of bromine water or a saturated potassium permanganate ($KMnO_4$) solution. Remove interfering turbidity by filtration before analysis. Filter samples for total or total hydrolyzable phosphorus only after digestion. Sample color that absorbs in the photometric range used for analysis also will interfere. See also Section 424F.1*b*.~~

~~*c. Application:* Orthophosphate can be determined in potable, surface, and saline waters as well as domestic and industrial wastewaters over a range of 0.001 to 10.0 mg P/L when photometric measurements are made at 650 to 660 or 880 nm in a 15-mm or 50-mm tubular flow cell. Determine higher concentrations by diluting sample.~~

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*Approved by Standard Methods Committee 1985

417 NITROGEN (AMMONIA)*

1. Selection of Method

The two major factors that influence selection of the method to determine ammonia are concentration and presence of interferences. In general, direct manual determination of low concentrations of ammonia is confined to drinking waters, clean surface water, and good-quality nitrified wastewater effluent. In other instances, and where interferences are present and greater precision is necessary, a preliminary distillation step (A) is required. For high ammonia concentrations a distillation and titration technique is preferred. The data presented in 417.4 below and Table 417:I should be helpful in selecting the appropriate method of analysis.

Two manual colorimetric techniques—the nesslerization (B) and phenate (C) methods—and one titration method (D) are presented. An ammonia-selective electrode method (E), which may be used either with or without prior sample distillation, and an automated version of the phenate method (G) also are included. While the stated maximum concentration ranges for the manual methods are not rigorous limits, titration is preferred at concentrations higher than the stated maximum levels for the photometric procedure.

The nessler method is sensitive to 20 µg NH_3-N/L under optimum conditions and may be used for up to 5 mg NH_3-N/L. Turbidity, color, and substances precipitated by hydroxyl ion, such as magnesium and calcium, interfere and may be removed by preliminary distillation or, less satisfactorily, by precipitation with zinc sulfate and alkali.

The manual phenate method has a sensitivity of 10 µg NH_3-N/L and is useful for up to 500 µg NH_3-N/L. Preliminary distillation is required if the alkalinity exceeds 500 mg $CaCO_3$/L, if color or turbidity is present, or if the sample has been preserved with acid.

The distillation and titration procedure is used especially for NH_3-N concentrations greater than 5 mg/L.

Distillation into sulfuric acid (H_2SO_4) absorbent is mandatory for the phenate method when interferences are present. Boric acid must be the absorbent following distillation if the distillate is to be nesslerized or titrated.

The ammonia-selective electrode method is applicable over the range from 0.03 to 1400 mg NH_3-N/L.

2. Interferences

Glycine, urea, glutamic acid, cyanates, and acetamide hydrolyze very slowly in solution on standing but, of these, only urea and cyanates will hydrolyze on distillation at pH of 9.5. Hydrolysis amounts to about 7% at this pH for urea and about 5% for cyanates. Glycine, hydrazine, and some amines will react with nessler reagent to give the characteristic yellow color in the time required for the test. Similarly, volatile alkaline compounds such as hydrazine and amines will influence titrimetric results. Some organic compounds such as ketones, aldehydes, alcohols, and some amines may cause a yellowish or greenish off-color or a turbidity on nesslerization following distillation. Some of these, such as formaldehyde, may be eliminated by boiling off at a low pH before nesslerization. Remove residual chlorine by sample pretreatment.

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*Approved by Standard Methods Committee, 1985.

3. Storage of Samples

Most reliable results are obtained on fresh samples. Destroy residual chlorine immediately after sample collection to prevent its reaction with ammonia. If prompt analysis is impossible, preserve samples with 0.8 mL conc H_2SO_4/L sample and store at 4°C. The pH of the acid-preserved samples should be between 1.5 and 2. Some wastewaters may require more conc H_2SO_4 to achieve this pH. If acid preservation is used, neutralize samples with NaOH or KOH immediately before making the determination.

4. Precision and Accuracy

Six synthetic samples containing ammonia and other constituents dissolved in distilled water were analyzed by five procedures. The first three samples were subjected to direct nesslerization alone, distillation followed by nesslerization, and distillation followed by titration. Samples through 6 were analyzed by direct ness- zation, by distillation followed by ness- ation, by the phenate method alone, by distillation followed by the phenate method. Results obtained by the participating laboratories are summarized in Table 417:I.

Sample 1 contained the following additional constituents: 10 mg Cl^-/L, 1.0 mg NO_3^--N/L, 1.5 mg organic N/L, 10.0 mg PO_4^{3-}/L, and 5.0 mg silica/L.

Sample 2 contained the following constituents: 200 mg Cl^-/L, 1.0 mg NO_3^--N/L, 0.8 mg organic N/L, 5.0 mg PO_4^{3-}/L, and 15.0 mg silica/L.

Sample 3 contained the following additional constituents: 400 mg Cl^-/L, 1.0 mg NO_3^--N/L, 0.2 mg organic N/L, 0.5 mg PO_4^{3-}/L, and 30.0 mg silica/L.

Sample 4 contained the following additional constituents: 400 mg Cl^-/L, 0.05 mg NO_3^--N/L, 0.23 mg organic phosphorus/L added in the form of adenylic acid, 7.00 mg orthophosphate phosphorus/L, and 3.00 mg polyphosphate phosphorus/L added as sodium hexametaphosphate.

Sample 5 contained the following additional constituents: 400 mg Cl^-/L, 5.00 mg NO_3^--N/L, 0.09 mg organic phosphorus/L added in the form of adenylic acid, 0.6 mg orthophosphate phosphorus/L, and 0.3 mg polyphosphate phosphorus/L added as sodium hexametaphosphate.

Sample 6 contained the following additional constituents: 400 mg Cl^-/L, 0.4 mg NO_3^--N/L, 0.03 mg organic phosphorus/L added in the form of adenylic acid, 0.1 mg orthophosphate phosphorus/L, and 0.08 mg polyphosphate phosphorus/L added as sodium hexametaphosphate.

For the ammonia-selective electrode in a single laboratory using surface water samples at concentrations of 1.00, 0.77, 0.19, and 0.13 mg NH_3-N/L, standard deviations were ±0.038, ±0.017, ±0.007, and ±0.003, respectively. In a single laboratory using surface water samples at concentrations of 0.10 and 0.13 NH_3-N/L, recoveries were 96% and 91%, respectively. The results of an interlaboratory study involving 12 laboratories using the ammonia-selective electrode† on distilled water and effluents are summarized in Table 417:II.

For an automated phenate system‡ in a single laboratory using surface water samples at concentrations of 1.41, 0.77, 0.59, and 0.43 mg NH_3-N/L, the standard deviation was ±0.005, and at concentrations of 0.16 and 1.44 mg NH_3-N/L, recoveries were 107 and 99%, respectively.

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†American Society for Testing and Materials, ASTM Method 1426-79

‡AutoAnalyzer™ I, Technicon Instrument Corp., Tarrytown, N.Y. 10591

TABLE 417:I PRECISION AND ACCURACY DATA FOR AMMONIA METHODS

Number of Laboratories	Ammonia Nitrogen Concentration µg/L	Relative Standard Deviation					Relative Error				
				Distillation Plus					Distillation Plus		
		Direct Nesslerization %	Direct Manual Phenate Method %	Nessler Method %	Manual Phenate Method %	Titrimetric Method %	Direct Nesslerization %	Direct Manual Phenate Method %	Nessler Method %	Manual Phenate Method %	Titrimetric Method %
20	200	38.1	—	—	—	—	0	—	—	—	—
44	200	—	—	46.3	—	—	—	—	10.0	—	—
21	200	—	—	—	—	69.8	—	—	—	—	20.0
20	800	11.2	—	—	—	—	0	—	—	—	—
42	800	—	—	21.2	—	—	—	—	8.7	—	—
20	800	—	—	—	—	28.6	—	—	—	—	5.0
21	1500	11.6	—	—	—	—	0.6	—	—	—	—
42	1500	—	—	18.0	—	—	—	—	4.0	—	—
21	1500	—	—		—	21.6	—	—	—	—	2.6
70	200	—	39.2		—	—	—	2.4	—	—	
3	200		—		15.1	—	—	—	—	16.7	—
9	200	22.0				—	8.3	—	—	—	—
5	200		—	15.7	—	—	—	—	2.0	—	—
66	800	—	15.8	—	—	—	—	1.5	—	—	—
3	800	—	—	—	16.6	—	—	—	—	1.7	—
9	800	16.1	—		—	—	0.3	—	—	—	—
6	800	—		16.3	—	—	—	—	3.1	—	—
71	1500	—	26.0	—	—	—	—	10.0	—	—	—
3	1500	—			7.3	—	—	—	—	0.4	—
8	1500	5.3	—	—	—	—	1.2	—	—	—	—
6	1500	—	—	7.5	—	—	—	—	3.6	—	—

TABLE 417:II. PRECISION AND BIAS OF AMMONIA-SELECTIVE ELECTRODE

Level *mg/L*	Matrix	Mean Recovery %	Precision	
			Overall, S_T	Operator, S_o
0.04	Distilled water	200	0.05	0.01
	Effluent water	100	0.03	0.00
0.10	Distilled water	180	0.05	0.01
	Effluent water	470	0.61	0.01
0.80	Distilled water	105	0.11	0.04
	Effluent water	105	0.30	0.06
20	Distilled water	95	2	1
	Effluent water	95	3	2
100	Distilled water	98	5	2
	Effluent water	97	—	—
750	Distilled water	97	78	12
	Effluent water	99	106	10

417 A. Preliminary Distillation Step

General Discussion

he sample is buffered at pH 9.5 with a te buffer to decrease hydrolysis of cy- inates and organic nitrogen compounds. It is distilled into a solution of boric acid when nesslerization or titration is to be ised or into H_2SO_4 when the phenate method is used. The ammonia in the distillate can be determined either colorimet- ically by nesslerization or the phenate nethod or titrimetrically with standard H_2SO_4 and a mixed indicator or a pH meter. The choice between the colorimetric nd the acidimetric method depends on the oncentration of ammonia. Ammonia in the distillate also can be determined by the ammonia-selective electrode method, using 04*N* H_2SO_4 to trap the distillate.

2. Apparatus

a. Distillation apparatus: Arrange a bo- silicate glass flask of 800- to 2000-mL capacity attached to a vertical condenser that the outlet tip may be submerged below the surface of the receiving acid solution. Use an all-borosilicate-glass apparatus or one with condensing units constructed of block tin or aluminum tubes.

b. pH meter.

3. Reagents

a. Ammonia-free water: Prepare by ion-exchange or distillation methods:

1) Ion exchange—Prepare ammonia-free water by passing distilled water through an ion-exchange column containing a strongly acidic cation-exchange resin mixed with a strongly basic anion-exchange resin. Select resins that will remove organic compounds that interfere with the ammonia determination. Some anion-exchange resins tend to release ammonia. If this occurs, prepare ammonia-free water with a strongly acidic cation-exchange resin. Regenerate the column according to the manufacturer's instructions. Check ammonia-free water for the possibility of a high blank value.

2) Distillation—Eliminate traces of am

monia in distilled water by adding 0.1 mL conc H_2SO_4 to 1 L distilled water and redistilling. Alternatively, treat distilled water with sufficient bromine or chlorine water to produce a free halogen residual of 2 to 5 mg/L and redistill after standing at least 1 h. Discard the first 100 mL distillate. Check redistilled water for the possibility of a high blank.

It is very difficult to store ammonia-free water in the laboratory without contamination from gaseous ammonia. However, if storage is necessary, store in a tightly stoppered glass container to which is added about 10 g ion-exchange resin (preferably a strongly acidic cation-exchange resin) per liter ammonia-free water. For use, let resin settle and decant ammonia-free water. If a high blank value is produced, replace the resin or prepare fresh ammonia-free water.

Use ammonia-free distilled water for preparing all reagents, rinsing, and sample dilution.

b. Borate buffer solution: Add 88 mL 0.1*N* NaOH solution to 500 mL approximately 0.025*M* sodium tetraborate ($Na_2B_4O_7$) solution (9.5 g $Na_2B_4O_7 \cdot 10\,H_2O$/L) and dilute to 1 L.

c. Sodium hydroxide, 6*N*: Dissolve 240 g NaOH in water and dilute to 1 L.

d. Dechlorinating agent: Use 1 mL of any of the following reagents to remove 1 mg/L residual chlorine in 500 mL sample.

1) *Sodium sulfite:* Dissolve 0.9 g Na_2SO_3 in water and dilute to 1 L. Prepare fresh daily.

2) *Sodium thiosulfate:* Dissolve 3.5 g $Na_2S_2O_3 \cdot 5H_2O$ in water and dilute to 1 L. Prepare fresh weekly.

3) *Phenylarsine oxide:* Dissolve 1.2 g C_6H_5AsO in 200 mL 0.3*N* NaOH solution, filter if necessary, and dilute to 1 L with water. (CAUTION *Toxic—take care to avoid ingestion.*)

4) *Sodium arsenite:* Dissolve 0.93 g $NaAsO_2$ in water and dilute to 1 L. (CAUTION: *Toxic—take care to avoid ingestion.*) Prepare fresh weekly.

e. Neutralization agent: Prepare with ammonia-free water.

1) *Sodium hydroxide,* NaOH, 1*N*.

2) *Sulfuric acid,* H_2SO_4, 1*N*.

f. Absorbent solution, plain boric acid: Dissolve 20 g H_3BO_3 in water and dilute to 1 L.

g. Indicating boric acid solution: See Section 417D.3*a* and *b*.

h. Sulfuric acid, 0.04*N*: Dilute 1.0 mL conc H_2SO_4 to 1 L.

4. Procedure

a. Preparation of equipment: Add 500 mL water and 20 mL borate buffer to a distillation flask and adjust pH to 9.5 with 6*N* NaOH solution. Add a few glass beads or boiling chips and use this mixture to steam out the distillation apparatus until distillate shows no traces of ammonia.

b. Sample preparation: Use 500 mL dechlorinated sample or a portion diluted to 500 mL with water. When NH_3-N concentration is less than 100 µg/L, use a sample volume of 1000 mL. Remove residual chlorine by adding, at the time of collection, dechlorinating agent equivalent to the chlorine residual. If necessary, neutralize to approximately pH 7 with dilute acid or base, using a pH meter.

Add 25 mL borate buffer solution and adjust to pH 9.5 with 6*N* NaOH using a pH meter.

c. Distillation: To minimize contamination, leave distillation apparatus assembled after steaming out and until just before starting sample distillation. Disconnect steaming-out flask and immediately transfer sample flask to distillation apparatus. Distill at a rate of 6 to 10 mL/min with the tip of the delivery tube below the surface of acid receiving solution. Collect distillate in a 500-mL erlenmeyer flask containing 50 mL plain boric acid solution for nesslerization method. Use 50 mL indicating boric acid solution for titrimetric method: Distill ammonia into 50 mL 0.04*N* H$_2$SO$_4$ for the phenate method and for the ammonia-selective electrode method. Collect at least 200 mL distillate. Lower collected distillate free of contact with delivery tube and continue distillation during the last minute or two to cleanse condenser and delivery tube. Dilute to 500 mL with water.

When the phenate method is used for determining NH$_3$-N, neutralize distillate with 1*N* NaOH solution.

d. Ammonia determination: Determine ammonia by the nesslerization method (*B*), the phenate method (C), the titrimetric method (D) or the ammonia-selective electrode method (E).

~~417 B. Nesslerization Method (Direct and Following Distillation)~~

~~1. General Discussion~~

~~Use direct nesslerization only for purified drinking waters, natural water, and highly purified wastewater effluents, all of which should be low in color and have NH$_3$-N concentrations exceeding 20 µg/L. Apply the direct nesslerization method to domestic wastewaters only when errors of to 2 mg/L are acceptable. Use this thod only after it has been established at it yields results comparable to those obtained after distillation. Check validity of direct nesslerization measurements periodically.~~

~~Pretreatment before direct nesslerization with zinc sulfate and alkali precipitates calcium, iron, magnesium, and sulfide, which form turbidity when treated with nessler reagent. The floc also removes suspended matter and sometimes colored matter. Addition of EDTA or Rochelle salt solution inhibits precipitation of residual calcium and magnesium ions in the presence of the alkaline nessler reagent. However, use of EDTA demands an extra amount of nessler reagent to insure a sufficient nessler reagent excess for reaction with the ammonia.~~

~~The graduated yellow to brown colors produced by the nessler-ammonia reaction absorb strongly over a wide wavelength range. The yellow color characteristic of low ammonia nitrogen concentration (0.4 o 5 mg/L) can be measured with acceptable sensitivity in the wavelength region from 400 to 425 nm when a 1-cm light path is available. A light path of 5 cm extends measurements into the nitrogen concentration range of 5 to 60 µg/L. The reddish brown hues typical of ammonia nitrogen levels approaching 10 mg/L may be measured in the wavelength region of 450 to 500 nm. A judicious selection of light path and wavelength thus permits the photometric determination of ammonia nitrogen concentrations over a considerable range.~~

~~Departures from Beer's law may be evident when photometers equipped with broad-band color filters are used. For this reason, prepare the calibration curve under conditions identical with those adopted for the samples.~~

~~A carefully prepared nessler reagent may respond under optimum conditions to as little as 1 µg NH$_3$-N/50 mL. In direct nesslerization, this represents 20 µg/L. However, reproducibility below 100 µg/L may be erratic.~~

~~2. Apparatus~~

~~*a. Colorimetric equipment:* One of the following is required:~~

~~1) *Spectrophotometer,* for use at 400 to 500 nm and providing a light path of 1 cm or longer.~~

Ammonia Nitrogen in Sample *mg/L*	Sample Volume *mL*
5–10	250
10–20	100
20–50	50.0
50–100	25.0

2. Apparatus

Distillation apparatus: See Section 417A.2*a* and *b*.

3. Reagents

Use ammonia-free water in making all reagents and dilutions.

a. Mixed indicator solution: Dissolve 200 mg methyl red indicator in 100 mL 95% ethyl or isopropyl alcohol. Dissolve 100 mg methylene blue in 50 mL 95% ethyl or isopropyl alcohol. Combine solutions. Prepare monthly.

b. Indicating boric acid solution: Dissolve 20 g H_3BO_3 in ammonia-free distilled water, add 10 mL mixed indicator solution, and dilute to 1 L. Prepare monthly.

c. Standard sulfuric acid titrant, 0.02*N*: Prepare and standardize as directed in Alkalinity, Section 403.3*c*. For greatest accuracy, standardize titrant against an amount of Na_2CO_3 that has been incorporated in the indicating boric acid solution to reproduce the actual conditions of sample titration; 1.00 mL = 280 μg N.

4. Procedure

a. Proceed as described in Section 417A using indicating boric acid solution as absorbent for the distillate.

b. Sludge or sediment samples: Rapidly weigh to within ±1% an amount of wet sample, equivalent to approximately 1 g dry weight, in a weighing bottle or crucible. Wash sample into a 500-mL kjeldahl flask with water and dilute to 250 mL. Proceed as in ¶ 4*a* but add a piece of paraffin wax to distillation flask and collect only 100 mL distillate.

c. Titrate ammonia in distillate with standard 0.02*N* H_2SO_4 titrant until indicator turns a pale lavender.

d. Blank: Carry a blank through all steps of the procedure and apply the necessary correction to the results.

5. Calculation

a. Liquid samples:

$$\text{mg } NH_3\text{-N/L} = \frac{(A - B) \times 280}{\text{mL sample}}$$

b. Sludge or sediment samples:

$$\text{mg } NH_3\text{-N/kg} = \frac{(A - B) \times 280}{\text{g dry wt sample}}$$

where:

A = volume of H_2SO_4 titrated for sample, mL, and

B = volume of H_2SO_4 titrated for blank, mL

6. Precision and Accuracy

See Section 417.4 and Table 417.I.

417 E. Ammonia-Selective Electrode Method

1. General Discussion

a. Principle: The ammonia-selective electrode uses a hydrophobic gas-permeable membrane to separate the sample solution from an electrode internal solution of ammonium chloride. Dissolved ammonia ($NH_{3(aq)}$ and NH_4^+) is converted to $NH_{3(aq)}$

by raising pH to above 11 with a strong base. $NH_{3(aq)}$ diffuses through the membrane and changes the internal solution pH that is sensed by a pH electrode. The fixed level of chloride in the internal solution is sensed by a chloride ion-selective electrode that serves as the reference electrode. Potentiometric measurements are made with a pH meter having an expanded millivolt scale or with a specific ion meter.

b. Scope and application: This method is applicable to the measurement of 0.03 to 1400 mg NH_3-N/L in potable and surface waters and domestic and industrial wastes. High concentrations of dissolved ions affect the measurement, but color and turbidity do not. Sample distillation is unnecessary. Use standard solutions and samples that have the same temperature and contain about the same total level of dissolved species. The ammonia-selective electrode responds slowly below 1 mg NH_3-N/L; hence, use longer times of electrode immersion (5 to 10 min) to obtain stable readings.

c. Interference: Amines are a positive interference. Mercury and silver interfere by complexing with ammonia.

d. Sample preservation: Do not use $HgCl_2$ as a sample preservative. Refrigerate at 4°C for samples to be analyzed within 24 h. Preserve samples high in organic and nitrogenous matter, and any other samples for a prolonged period, by lowering pH to 2 or less with conc H_2SO_4.

2. Apparatus

a. Electrometer: A pH meter with expanded millivolt scale capable of 0.1 mV resolution between −700 mV and +700 mV or a specific ion meter.

*b. Ammonia-selective electrode.**

c. Magnetic stirrer, thermally insulated, with TFE-coated stirring bar.

3. Reagents

a. Ammonia-free water: See Section 417A.3*a*. Use for making all reagents.

b. Sodium hydroxide, 10*N*: Dissolve 400 g NaOH in 800 mL water. Cool and dilute to 1000 mL with water.

c. Stock ammonium chloride solution: See Section 417B.3*d*.

d. Standard ammonium chloride solutions: See ¶ 4*a* below.

4. Procedure

a. Preparation of standards: Prepare a series of standard solutions covering the concentrations of 1000, 100, 10, 1, and 0.1 mg NH_3-N/L by making decimal dilutions of stock NH_4Cl solution with water.

b. Electrometer calibration: Place 100 mL of each standard solution in a 150-mL beaker. Immerse electrode in standard of lowest concentration and mix with a magnetic stirrer. Do not stir so rapidly that air bubbles are sucked into the solution because they will become trapped on the electrode membrane. Maintain the same stirring rate and a temperature of about 25°C throughout calibration and testing procedures. Add a sufficient volume of 10*N* NaOH solution (1 mL usually is sufficient) to raise pH above 11. Keep electrode in solution until a stable millivolt reading is obtained. CAUTION: Check electrode sensing element performance according to manufacturer's instructions to make sure that electrode is operating properly. Do not add NaOH solution before immersing electrode, because ammonia may be lost from a basic solution. Repeat procedure with remaining standards, proceeding from lowest to highest concentration. Wait for at least 5 min before recording millivolts for standards and samples containing ≤ 1 mg NH_3-N/L.

c. Preparation of standard curve: Using semilogarithmic graph paper, plot ammonia concentration in milligrams NH_3-N per liter on the log axis vs. potential in milli-

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*Orion Model 95-10 or 95-12, EIL Model 8002-2, Beckman Model 39565, or equivalent volts on the linear axis starting with the lowest concentration at the bottom of the scale. If the electrode is functioning properly a tenfold change of NH_3-N concentration produces a potential change of 59 mV.

d. Calibration of specific ion meter: Refer to manufacturer's instructions and proceed as in ¶s 4*a* and *b*.

e. Measurement of samples: Dilute if necessary to bring NH_3-N concentration to within calibration curve range. Place 100 mL sample in 150-mL beaker and follow procedure in ¶ 4*b* above. Record volume of 10*N* NaOH added in excess of 1 mL. Read NH_3-N concentration from standard curve.

5. Calculation

$$\text{mg NH}_3\text{-N/L} = A \times B \times \left[\frac{101 - C}{101}\right]$$

where:

A = dilution factor,
B = concentration of NH_3-N/L, mg/L, from calibration curve, and
C = volume of added 10*N* NaOH in excess of 1 mL, mL.

6. Precision and Accuracy

See Section 417.4.

~~417 F. Ammonia-Selective Electrode Method Using Known Addition~~

~~1. General Discussion~~

~~*a. Principle:* When a linear relationship exists between concentration and response, known addition is convenient for measuring occasional samples because no calibration is needed. Because an accurate measurement requires that the concentration approximately double as a result of the addition, sample concentration must be known within a factor of three. Total concentration of ammonia can be measured in the absence of complexing agents down to 0.8 mg NH_3-N/L or in the presence of a large excess (50 to 100 times) of complexing agent. Known addition is a convenient check on the results of direct measurement.~~

~~*b.* See Section 417E.1 for further discussion.~~

~~2. Apparatus~~

~~Use apparatus specified in Section 417E.2.~~

~~3. Reagents~~

~~*Use reagents specified in Section 417E.3.*~~

~~Add standard ammonium chloride solution approximately 10 times as concentrated as samples being measured.~~

~~4. Procedure~~

~~*a.* Dilute 1000 mg/L stock solution to make a standard solution about 10 times as concentrated as the sample concentrate.~~

~~*b.* Add 1 mL 10*N* NaOH to each 100 mL sample and immediately immerse electrode. When checking a direct measurement, leave electrode in 100 mL of sample solution. Use magnetic stirring throughout. Measure mV reading and record as E_1.~~

~~*c.* Pipet 10 mL of standard solution into sample. Thoroughly stir and immediately record new mV reading as E_2.~~

~~5. Calculation~~

~~*a.* $\Delta E = E_1 - E_2$.~~

~~*b.* From Table 417:IV find the concen-~~

water; 1.00 mL = 50.0 µg N. Prepare daily.

i. Standard nitrite solution: Dilute 10.00 mL intermediate NO_2^- solution to 1000 mL with water; 1.00 mL = 0.500 µg N. Prepare daily.

4. Procedure

a. Removal of turbidity: If sample contains suspended solids, filter through a 0.45-µm pore diam membrane filter.

b. Color development: To 50.0 mL clear sample neutralized to pH 7, or to a portion diluted to 50.0 mL, add 1 mL sulfanilamide solution. Let reagent react for 2 to 8 min. Add 1.0 mL NED dihydrochloride solution and mix immediately. Let stand at least 10 min but not more than 2 h.

c. Photometric measurement: Measure absorbance at 543 nm. As a guide use the following light paths for the indicated NO_2^--N concentrations:

Light Path Length *cm*	NO_2^--N *µg/L*
1	2–25
5	2–6
10	<2

Run parallel checks frequently against NO_2^- standards, preferably in the concentration range of the sample. Redetermine complete calibration curves after preparing new reagents.

d. Color standards for visual comparison: Prepare a suitable series of visual color standards in nessler tubes by adding the following volumes of standard NO_2^- solution and diluting to 50 mL with water: 0, 0.1, 0.2, 0.4, 0.7, 1.0, 1.4, 1.7, 2.0, and 2.5 mL, corresponding, respectively, to 0, 1.0, 2.0, 4.0, 7.0, 10, 14, 17, 20, and 25 µg NO_2^--N/L. Develop color as described in ¶ 4*b*. Compare samples to visual standards in matched nessler tubes between 10 and 120 min after adding NED dihydrochloride reagent. Select the concentration where the sample tube color matches the standard tube color.

5. Calculation

$$\text{mg } NO_2^-\text{-N/L} = \frac{\mu g\ NO_2^-\text{-N (in 52 mL final volume)}}{\text{mL sample}}$$

6. Bibliography

RIDER, B.F. & M.G. MELLON. 1946. Colorimetric determination of nitrites. *Ind. Eng. Chem.*, Anal. Ed. 18:96.

BARNES, H. & A.R. FOLKARD. 1951. The determination of nitrites. *Analyst* 76:599.

BOLTZ, D.F., ed. 1958. Colorimetric Determination of Nonmetals. Interscience Publishers, New York, N.Y.

420 NITROGEN (ORGANIC)*

1. Selection of Method

The major factor that influences the selection of a macro- or semi-micro-kjeldahl method to determine organic nitrogen is the concentration of organic nitrogen. The macro-kjeldahl method is applicable for samples containing either low or high concentrations of organic nitrogen but requires a relatively large sample volume for low concentrations. In the semi-micro-kjeldahl method, which is applicable to samples containing high concentrations of organic nitrogen, the sample volume should be chosen to contain organic plus ammonia ni-

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*Approved by Standard Methods Committee, 1985.

trogen (kjeldahl nitrogen) in the range of 0.2 to 2 mg.

2. Storage of Samples

The most reliable results are obtained on fresh samples. If an immediate analysis is not possible, preserve samples by acidifying to pH 1.5 to 2.0 with concentrated H_2SO_4 and storing at 4°C. Do not use $HgCl_2$ because it will interfere with ammonia removal.

3. Interferences

a. Nitrate: During digestion, nitrate in excess of 10 mg/L can oxidize a portion of the ammonia released from the digested organic nitrogen, producing N_2O and resulting in a negative interference. When sufficient organic matter in a low state of oxidation is present, nitrate can be reduced to ammonia, resulting in a positive interference. The conditions under which significant interferences occur are not well defined and there is no proven way to eliminate the interference in conjunction with the methods described herein.

b. Inorganic salts and solids: The acid and salt content of the digestion reagent is intended to produce a digestion temperature of about 360 to 370°C. If the sample contains a very large quantity of salt or inorganic solids that dissolve during digestion, the temperature may rise above 400°C, at which point pyrolytic loss of nitrogen begins to occur. To prevent an excessive digestion temperature, add more H_2SO_4 to maintain the acid-salt balance. Not all salts cause precisely the same temperature rise, but adding of 1 mL H_2SO_4/g salt in the sample gives reasonable results. Add the extra acid and the digestion reagent to both sample and reagent blank. Too much acid will lower the digestion temperature below 360°C and result in incomplete digestion and recovery. If necessary, add more sodium hydroxide-sodium thiosulfate before the final distillation step to neutralize the excess acid.

Large amounts of salt or solids also may cause bumping during distillation. If this occurs, add more dilution water to the samples after digestion.

c. Organic matter: During digestion, H_2SO_4 oxidizes organic matter to CO_2 and H_2O. If a large amount of organic matter is present, a large amount of acid will be consumed, the ratio of salt to acid will increase, and the digestion temperature will increase. If enough organic matter is present, the temperature will rise above 400°C, resulting in pyrolytic loss of nitrogen. To prevent this, add to the digestion flask 10 mL conc H_2SO_4/3 g COD. (For most organic substances, 3 g COD equals about 1 g organic matter). Alternately, add 50 mL more of digestion reagent/g COD. Additional sodium hydroxide-sodium thiosulfate reagent may be necessary to keep the distillation pH high. Because reagents may contain traces of ammonia, treat the reagent blank identically with the samples.

4. Use of a Catalyst

Although it generally is desirable to avoid using mercury because of its toxicity and the problems associated with disposal of residues, mercury is the catalyst of choice. Only selenium is as effective as mercury, but selenium is highly toxic and there are potential interferences associated with its use. Digestion of some samples may be complete or nearly complete without the use of a catalyst or with the use of a less toxic catalyst, such as copper. If copper is substituted for mercury, add 10 mL of a solution containing 25.115 g $CuSO_4$/L to each macro-kjeldahl digestion flask with 50 mL digestion reagent from which the HgO has been omitted. Use 2 mL of $CuSO_4$ solution for the semi-micro method. If the mercury catalyst is omitted, report this deviation and indicate, if possible, the percentage recovery relative to the results for similar samples analyzed using the mercury catalyst.

420 A. Macro-Kjeldahl Method

1. General Discussion

The kjeldahl method determines nitrogen in the trinegative state. It fails to account for nitrogen in the form of azide, azine, azo, hydrazone, nitrate, nitrite, nitrile, nitro, nitroso, oxime, and semi-carbazone. If ammonia nitrogen is not removed in the initial phase (¶ 4*b* below) of the procedure, the term "kjeldahl nitrogen" is applied to the result. Should kjeldahl nitrogen and ammonia nitrogen be determined individually, "organic nitrogen" can be obtained by difference.

a. Principle: In the presence of H_2SO_4, potassium sulfate (K_2SO_4), and mercuric sulfate ($HgSO_4$) catalyst, amino nitrogen of many organic materials is converted to ammonium sulfate [$(NH_4)_2SO_4$]. Free ammonia and ammonium-nitrogen also are converted to $(NH_4)_2SO_4$. During sample digestion, a mercury ammonium complex is formed and then decomposed by sodium thiosulfate ($Na_2S_2O_3$). After decomposition the ammonia is distilled from an alkaline medium and absorbed in boric or sulfuric acid. The ammonia is determined colorimetrically or by titration with a standard mineral acid.

b. Selection of ammonia measurement method: The sensitivity of colorimetric methods makes them particularly useful for determining organic nitrogen levels below 5 mg/L. The titrimetric and selective electrode methods of measuring ammonia in the distillate are suitable for determining a wide range of organic nitrogen concentrations.

2. Apparatus

a. Digestion apparatus: Kjeldahl flasks with a total capacity of 800 mL yield the best results. Digest over a heating device adjusted so that 250 mL water at an initial temperature of 25°C can be heated to a rolling boil in approximately 5 min. For testing, preheat heaters for 10 min if gas or 30 min if electric. A heating device meeting this specification should provide the temperature range of 365 to 370°C for effective digestion.

b. Distillation apparatus: See Section 417A.2*a*.

c. Apparatus for ammonia determination: See Sections 417B.2, 417C.2, 417D.2, or 417E.2.

3. Reagents

Prepare all reagents and dilutions in ammonia-free water.

All of the reagents listed for the determination of Nitrogen (Ammonia), Sections 417B.3, 417C.3, 417D.3, or 417E.3, are required, plus the following:

a. Mercuric sulfate solution: Dissolve 8 g red mercuric oxide, HgO, in 100 mL 6*N* H_2SO_4.

b. Digestion reagent: Dissolve 134 g K_2SO_4 in 650 mL water and 200 mL conc H_2SO_4. Add, with stirring, 25 mL mercuric sulfate solution. Dilute the combined solution to 1 L with water. Keep at a temperature close to 20°C to prevent crystallization.

c. Sodium hydroxide-sodium thiosulfate reagent: Dissolve 500 g NaOH and 25 g $Na_2S_2O_3 \cdot 5H_2O$ in water and dilute to 1 L.

d. Borate buffer solution: See Section 417A.3*b*.

e. Sodium hydroxide, NaOH, 6*N*.

4. Procedure

a. Selection of sample volume and sample preparation: Place a measured volume of sample in an 800-mL kjeldahl flask. Select sample size from the following tabulation:

Organic Nitrogen in Sample *mg/L*	Sample Size *mL*
0–1	500
1–10	250
10–20	100
20–50	50.0
50–100	25.0

If necessary, dilute sample to 300 mL, neutralize to pH 7, and dechlorinate as described in Section 417A.4*b*.

b. Ammonia removal: Add 25 mL borate buffer and then $6N$ NaOH until pH 9.5 is reached. Add a few glass beads or boiling chips and boil off 300 mL. If desired, distill this fraction and determine ammonia nitrogen. Alternately, if ammonia has been determined by the distillation method, use residue in distilling flask for organic nitrogen determination.

For sludge and sediment samples, weigh wet sample in a crucible or weighing bottle, transfer contents to a kjeldahl flask, and determine kjeldahl nitrogen. Follow a similar procedure for ammonia nitrogen and organic nitrogen determined by difference. Determinations of organic and kjeldahl nitrogen on dried sludge and sediment samples are not accurate because drying results in loss of ammonium salts. Measure dry weight of sample on a separate portion.

c. Digestion: Cool and add carefully 50 mL digestion reagent (or substitute 10 mL conc H_2SO_4, 6.7 g K_2SO_4, and 1.25 mL $HgSO_4$ solution) to distillation flask. Add a few glass beads and, after mixing, heat under a hood or with suitable ejection equipment to remove acid fumes. Boil briskly until the volume is greatly reduced (to about 25 to 50 mL) and copious white fumes are observed (fumes may be dark for samples high in organic matter). Then continue to digest for an additional 30 min. As digestion continues, colored or turbid samples will turn clear or straw-colored. After digestion, let flask and contents cool, dilute to 300 mL with water, and mix. Tilt flask and carefully add 50 mL hydroxide-thiosulfate reagent to form an alkaline layer at flask bottom. Connect flask to steamed-out distillation apparatus and shake flask to insure complete mixing. A black precipitate, HgS, will form, and the pH should exceed 11.0.

d. Distillation: Distill and collect 200 mL distillate below surface of 50 mL absorbent solution. Use plain boric acid solution when ammonia is to be determined by nesslerization and use indicating boric acid for a titrimetric finish. Use 50 mL $0.04N$ H_2SO_4 solution for collecting distillate for manual phenate, nesslerization, or electrode methods. Extend tip of condenser well below level of absorbent solution and do not let temperature in condenser rise above 29°C. Lower collected distillate free of contact with delivery tube and continue distillation during last 1 or 2 min to cleanse condenser.

e. Final ammonia measurement: Use the nesslerization (417B), manual phenate (417C), titration (417D), or ammonia-selective electrode (417E) method.

f. Blank: Carry a reagent blank through all steps of the procedure and apply necessary corrections to the results.

5. Calculation

See Section 417B.5, 417C.5, 417D.5, or 417E.5.

6. Precision and Accuracy

Three synthetic samples containing various organic nitrogen concentrations and other constituents were analyzed by three procedural modifications of the macro-kjeldahl method: kjeldahl-nessler finish, kjeldahl-titrimetric finish, and calculation of the difference between kjeldahl nitrogen and ammonia nitrogen, both determined by a nessler finish. The results obtained by participating laboratories are summarized in Table 420:I.

TABLE 420:I. PRECISION AND ACCURACY DATA FOR ORGANIC NITROGEN, MACRO-KJELDAHL PROCEDURE

			Relative Standard Deviation			Relative Error		
Sample	No of Laboratories	Organic *Nitrogen* Concentration $\mu g/L$	Nessler Finish %	Titrimetric Finish %	Calculation of *Total Kjeldahl* N Minus NH_3-N %	Nessler Finish %	Titrimetric Finish %	Calculation of Total *Kjeldahl* N Minus NH_3-N %
1	26	200	94.8	—	—	55.0	—	—
	29	200	—	104.4	—	—	70.0	—
	15	200	—	—	68.8	—	—	70.0
2	26	800	52.1	—	—	12.5	—	—
	31	800	—	44.8	—	—	3.7	—
	16	800	—	—	52.6	—	—	8.7
3	26	1500	43.1	—	—	9.3	—	—
	30	1500	—	54.7	—	—	22.6	—
	16	1500	—	—	45.9	—	—	4.0

No data on the precision of the macro-kjeldahl-phenate method are available.

Sample 1 *contained the following* additional constituents: 400 mg Cl^-/L, 1.50 mg NH_3-N/L, 1.0 mg NO_3^--N/L, 0.5 mg PO_4^{3-}/L, and 30.0 mg SiO_2/L.

Sample 2 contained the following additional constituents: 200 mg Cl^-/L, 0.8 mg NH_3-N/L, 1.0 mg NO_3^--N/L, 5.0 mg PO_4^{3-}/L, and 15.0 mg SiO_2/L.

Sample 3 contained the following additional constituents: 10 mg Cl^-/L, 0.2 mg NH_3-N/L, 1.0 mg NO_3^--N/L, 10.0 mg PO_4^{3-}/L, and 5.0 mg SiO_2/L.

7. Bibliography

KJELDAHL, J. 1883. A new method for the determination of nitrogen in organic matter. *Z. Anal. Chem.* 22:366.

PHELPS, E.B. 1905. The determination of organic nitrogen in sewage by the Kjeldahl process. *J. Infect. Dis.* (Suppl.) 1:225.

MEEKER, E.W. & E.C. WAGNER. 1933. Titration of ammonia in the presence of boric acid. *Ind. Eng. Chem.*, Anal. Ed. 5:396.

WAGNER, E.C. 1940. Titration of ammonia in the presence of boric acid. *Ind. Eng. Chem.*, Anal. Ed. 12:771.

MCKENZIE, H.A. & H.S. WALLACE. 1954. The Kjeldahl determination of nitrogen: A critical study of digestion conditions. *Aust. J. Chem.* 7:55.

MORGAN, G.B., J.B. LACKEY & F.W. GILCREAS. 1957. Quantitative determination of organic nitrogen in water, sewage, and industrial wastes. *Anal. Chem.* 29:833.

BOLTZ, D.F., ed. 1978. Colorimetric Determination of Nonmetals. Interscience Publishers, New York, N.Y.

NITROGEN, NITRATE-NITRITE

Method 353.2 (Colorimetric, Automated, Cadmium Reduction)

STORET NO. Total 00630

1. Scope and Application
   1.1 This method pertains to the determination of nitrite singly, or nitrite and nitrate combined in surface and saline waters, and domestic and industrial wastes. The applicable range of this method is 0.05 to 10.0 mg/l nitrate-nitrite nitrogen. The range may be extended with sample dilution.
2. Summary of Method
   2.1 A filtered sample is passed through a column containing granulated copper-cadmium to reduce nitrate to nitrite. The nitrite (that originally present plus reduced nitrate) is determined by diazotizing with sulfanilamide and coupling with N-(1-naphthyl)-ethylenediamine dihydrochloride to form a highly colored azo dye which is measured colorimetrically. Separate, rather than combined nitrate-nitrite, values are readily obtained by carrying out the procedure first with, and then without, the Cu-Cd reduction step.
3. Sample Handling and Preservation
   3.1 Analysis should be made as soon as possible. If analysis can be made within 24 hours, the sample should be preserved by refrigeration at 4°C. When samples must be stored for more than 24 hours, they should be preserved with sulfuric acid (2 ml conc. H_2SO_4 per liter) and refrigeration.
   Caution: Samples for reduction column must not be preserved with mercuric chloride.
4. Interferences
   4.1 Build up of suspended matter in the reduction column will restrict sample flow. Since nitrate-nitrogen is found in a soluble state, the sample may be pre-filtered.
   4.2 Low results might be obtained for samples that contain high concentrations of iron, copper or other metals. EDTA is added to the samples to eliminate this interference.
   4.3 Samples that contain large concentrations of oil and grease will coat the surface of the cadmium. This interference is eliminated by pre-extracting the sample with an organic solvent.
5. Apparatus
   5.1 Technicon AutoAnalyzer (AAI or AAII) consisting of the following components:
      5.1.1 Sampler.
      5.1.2 Manifold (AAI) or analytical cartridge (AAII).
      5.1.3 Proportioning Pump
      5.1.4 Colorimeter equipped with a 15 mm or 50 mm tubular flow cell and 540 nm filters.
      5.1.5 Recorder.

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5.1.6 Digital printer for AAII (Optional)

6. Reagents

6.1 Granulated cadmium 40–60 mesh (E M Laboratories, Inc., 500 Exec. Blvd., Elmsford, NY 10523, Cat. 2001 Cadmium, Coarse Powder)

6.2 Copper-cadmium The cadmium granules (new or used) are cleaned with dilute HCl (6.7) and copperized with 2% solution of copper sulfate (6.8) in the following manner

6.2.1 *Wash the cadmium with HCl (6.7) and rinse with distilled water The color of the* cadmium so treated should be silver.

6.2.2 Swirl 10 g cadmium in 100 ml portions of 2% solution of copper sulfate (6.8) for five minutes or until blue color partially fades, decant and repeat with fresh copper sulfate until a brown colloidal precipitate forms.

6.2.3 *Wash the cadmium-copper with distilled water (at least 10 times) to remove all the* precipitated copper. The color of the cadmium so treated should be black.

6.3 Preparation of reduction column AAI: The reduction column is an 8 by 50 mm glass tube with the ends reduced in diameter to permit insertion into the system Copper-cadmium granules (6.2) are placed in the column between glass wool plugs The packed reduction column is placed in an up-flow 20° incline to minimize channeling. *See Figure 1*

6.4 *Preparation of reduction column AAII*: The reduction column is a U-shaped, 35 cm length, 2 mm I.D. glass tube (Note 1). Fill the reduction column with distilled water to prevent entrapment of air bubbles during the filling operations. Transfer the copper-cadmium granules (6.2) to the reduction column and place a glass wool plug in each end To prevent entrapment of air bubbles in the reduction column be sure that all pump tubes are filled with *reagents before putting the column into the analytical system*

NOTE 1: A 0.081 I.D. pump tube (purple) can be used in place of the 2 mm glass tube

6.5 Distilled water: Because of possible contamination, this should be prepared by passage through an ion exchange column comprised of a mixture of both strongly acidic-cation and strongly basic-anion exchange resins The regeneration of the ion exchange column *should be carried out according to the manufacturer's instructions*

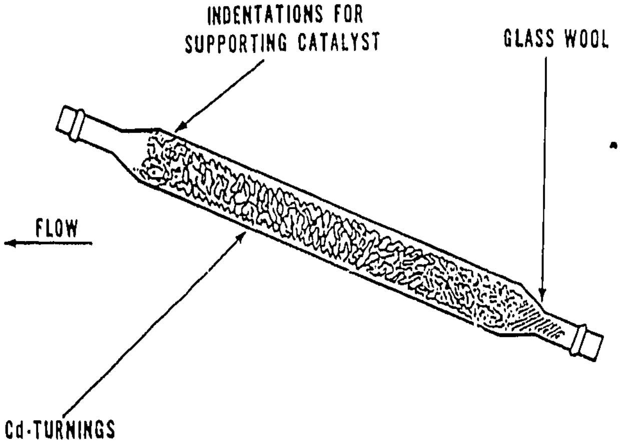
6.6 Color reagent: To approximately 800 ml of distilled water, add, while stirring, 100 ml conc. phosphoric acid, 40 g sulfanilamide, and 2 g N-1-naphthylethylenediamine dihydrochloride. Stir until dissolved and dilute to 1 liter. Store in brown bottle and keep in the dark when not in use. This solution is stable for several months.

6.7 *Dilute hydrochloric acid, 6N: Dilute 50 ml of conc. HCl to 100 ml with distilled water.*

6.8 Copper sulfate solution, 2%: Dissolve 20 g of $CuSO_4 \cdot 5H_2O$ in 500 ml of distilled water and dilute to 1 liter.

6.9 Wash solution: Use distilled water for unpreserved samples. For samples preserved with H_2SO_4, use 2 ml H_2SO_4 per liter of wash water.

6.10 Ammonium chloride-EDTA solution: *Dissolve 85 g of reagent grade ammonium chloride and 0.1 g of disodium ethylenediamine tetracetate in 900 ml of distilled water.* Adjust the pH to 8.5 with conc. ammonium hydroxide and dilute to 1 liter. Add 1/2 ml Brij-35 (available from Technicon Corporation)



TILT COLUMN TO 20° POSTION

FIGURE 1. COPPER CADMIUM REDUCTION COLUMN
(1 1/2 ACTUAL SIZE)

6.11 Stock nitrate solution: Dissolve 7.218 g KNO_3 and dilute to 1 liter in a volumetric flask with distilled water. Preserve with 2 ml of chloroform per liter. Solution is stable for 6 months. 1 ml = 1.0 mg NO_3-N.

6.12 Stock nitrite solution: Dissolve 6.072 g KNO_2 in 500 ml of distilled water and dilute to 1 liter in a volumetric flask. Preserve with 2 ml of chloroform and keep under refrigeration. 1.0 ml = 1.0 mg NO_2-N.

6.13 Standard nitrate solution: Dilute 10.0 ml of stock nitrate solution (6.11) to 1000 ml. 1.0 ml = 0.01 mg NO_3-N. Preserve with 2 ml of chloroform per liter. Solution is stable for 6 months.

6.14 Standard nitrite solution: Dilute 10.0 ml of stock nitrite (6.12) solution to 1000 ml. 1.0 ml = 0.01 mg NO_2-N. Solution is unstable; prepare as required.

6.15 Using standard nitrate solution (6.13), prepare the following standards in 100.0 ml volumetric flasks. At least one nitrite standard should be compared to a nitrate standard at the same concentration to verify the efficiency of the reduction column.

Conc., mgNO_2-N or NO_3-N/l	ml Standard Solution/100 ml
0.0	0
0.05	0.5
0.10	1.0
0.20	2.0
0.50	5.0
1.00	10.0
2.00	20.0
4.00	40.0
6.00	60.0

NOTE 2: When the samples to be analyzed are saline waters, Substitute Ocean Water (SOW) should be used for preparing the standards; otherwise, distilled water is used. A tabulation of SOW composition follows:

NaCl - 24.53 g/l
$CaCl_2$ - 1.16 g/l
KBr - 0.10 g/l
NaF - 0.003 g/l
$MgCl_2$ - 5.20 g/l
KCl - 0.70 g/l
H_3BO_3 - 0.03 g/l
Na_2SO_4 - 4.09 g/l
$NaHCO_3$ - 0.20 g/l
$SrCl_2$ - 0.03 g/l

7. Procedure

7.1 If the pH of the sample is below 5 or above 9, adjust to between 5 and 9 with either conc. HCl or conc. NH_4OH.

7.2 Set up the manifold as shown in Figure 2 (AAI) or Figure 3 (AAII). Note that reductant column should be in 20° incline position (AAI). Care should be taken not to introduce air into reduction column on the AAII.

7.3 Allow both colorimeter and recorder to warm up for 30 minutes. Obtain a stable baseline with all reagents, feeding distilled water through the sample line.

NOTE 3: Condition column by running 1 mg/l standard for 10 minutes if a new reduction column is being used. Subsequently wash the column with reagents for 20 minutes.

7.4 Place appropriate nitrate and/or nitrite standards in sampler in order of decreasing concentration of nitrogen. Complete loading of sampler tray with unknown samples.

7.5 For the AAI system, sample at a rate of 30/hr, 1:1. For the AAII, use a 40/hr, 4:1 cam and a common wash.

7.6 Switch sample line to sampler and start analysis.

8. Calculations

8.1 Prepare appropriate standard curve or curves derived from processing NO$_3$ and/or NO$_2$ standards through manifold. Compute concentration of samples by comparing sample peak heights with standard curve.

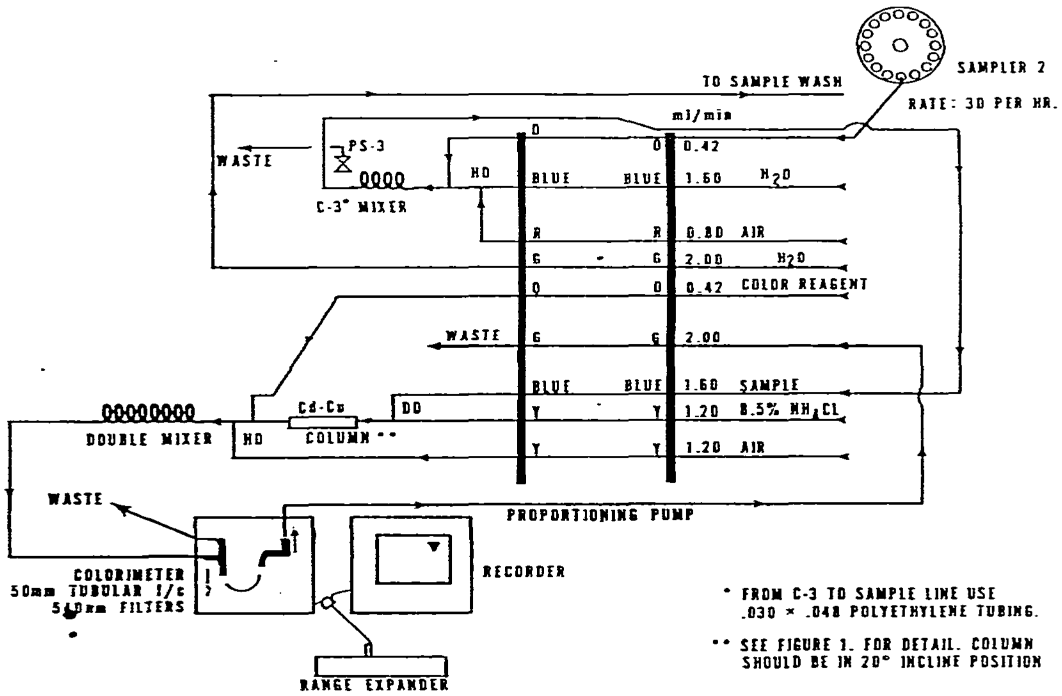
9. Precision and Accuracy

9.1 Three laboratories participating in an EPA Method Study, analyzed four natural water samples containing exact increments of inorganic nitrate, with the following results:

Increment as Nitrate Nitrogen mg N/liter	Precision as Standard Deviation mg N/liter	Accuracy as	
		Bias, %	Bias, mg N/liter
0.29	0.012	+ 5.75	+0.017
0.35	0.092	+18.10	+0.063
2.31	0.318	+ 4.47	+0.103
2.48	0.176	− 2.69	−0.067

Bibliography

1. Fior., J., and O'Brien, J. E., "Automation in Sanitary Chemistry – parts 1 & 2 Determination of Nitrates and Nitrites", Wastes Engineering 33, 128 & 238 (1962).
2. Armstrong, F. A., Stearns, C. R., and Strickland, J. D., "The Measurement of Upwelling and Subsequent Biological Processes by Means of the Technicon AutoAnalyzer and Associated Equipment", Deep Sea Research 14, p 381–389 (1967).
3. Annual Book of ASTM Standards, Part 31, "Water", Standard D1254, p 366 (1976).
4. Chemical Analyses for Water Quality Manual, Department of the Interior, FWPCA, R. A. Taft Sanitary Engineering Center Training Program, Cincinnati, Ohio 45226 (January, 1966).
5. Annual Book of ASTM Standards, Part 31, "Water", Standard D 1141–75, Substitute Ocean Water, p 48 (1976).



* FROM C-3 TO SAMPLE LINE USE .030 × .048 POLYETHYLENE TUBING.

** SEE FIGURE 1. FOR DETAIL. COLUMN SHOULD BE IN 20° INCLINE POSITION

FIGURE 2. NITRATE - NITRITE MANIFOLD AA-I

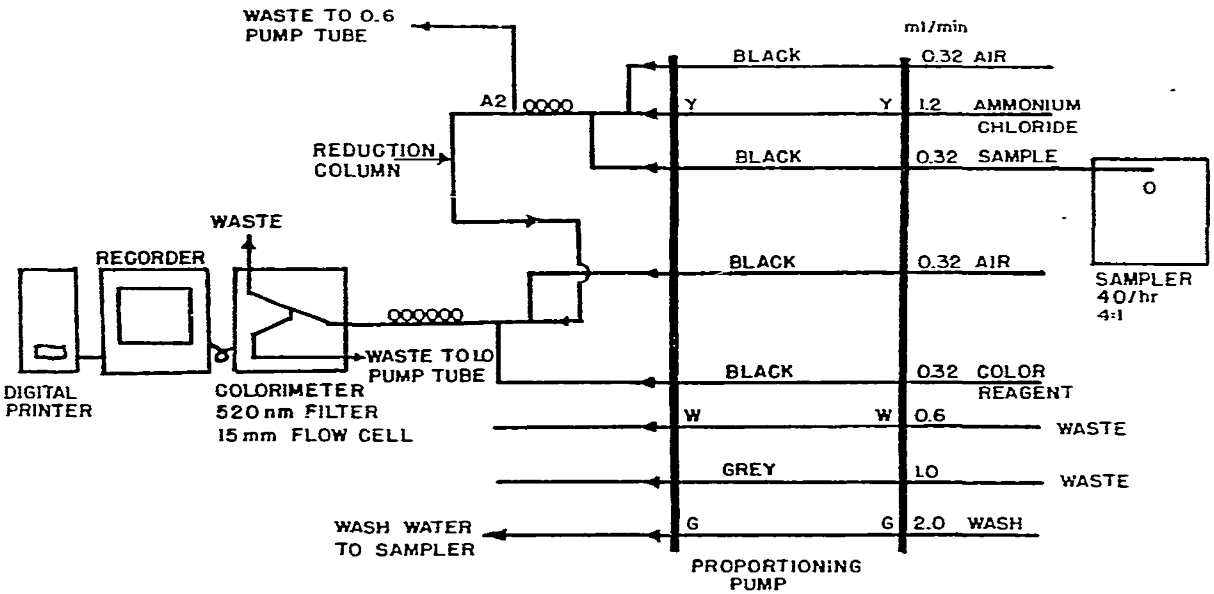


FIGURE 3 NITRATE-NITRITE MANIFOLD AA II

SULFATE

Method 375.4 (Turbidimetric)

STORET NO. Total 00945

1. Scope and Application
   1.1 This method is applicable to drinking and surface waters, domestic and industrial wastes.
   1.2 The method is suitable for all concentration ranges of sulfate; however, in order to obtain reliable readings, use a sample aliquot containing not more than 40 mg SO_4/l.
   1.3 The minimum detectable limit is approximately 1 mg/l sulfate.
2. Summary of Method
   2.1 Sulfate ion is converted to a barium sulfate suspension under controlled conditions. The resulting turbidity is determined by a nephelometer, filter photometer or spectrophotometer and compared to a curve prepared from standard sulfate solutions.
   2.2 Suspended matter and color interfere. Correct by running blanks from which the barium chloride has been omitted.
   2.3 Silica in concentrations over 500 mg/l will interfere.
3. Comments
   3.1 Proprietary reagents, such as Hach Sulfaver or equivalent, are acceptable.
   3.2 Preserve by refrigeration at 4°C.
4. Apparatus
   4.1 Magnetic stirrer, variable speed so that it can be held constant just below splashing. Use identical shape and size magnetic stirring bars.
   4.2 *Photometer: one of the following which are given in order of preference.*
      4.2.1 Nephelometer
      4.2.2 Spectrophotometer for use at 420 nm with light path of 4 to 5 cm.
      4.2.3 Filter photometer with a violet filter having a maximum near 420 nm and a light path of 4 to 5 cm.
   4.3 Stopwatch, if the magnetic stirrer is not equipped with an accurate timer.
   4.4 Measuring spoon, capacity 0.2 to 0.3 ml.
5. Reagents
   5.1 *Conditioning reagent:* Place 30 ml conc. HCl, 300 ml distilled water, 100 ml 95% ethanol or isopropanol and 75 g NaCl in solution in a container. Add 50 ml glycerol and mix.
   5.2 Barium chloride, $BaCl_2$, crystals, 20 to 30 mesh.
   5.3 Sodium carbonate solution (approximately 0.05N): Dry 3 to 5 g primary standard Na_2CO_3 at 250°C for 4 hours and cool in a desiccator. Weigh 2.5 ±0.2 g (to the nearest mg), transfer to a 1 liter volumetric flask and fill to the mark with distilled water.

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5.4 Standard sulfate solution (1.00 ml = 100 *ug* SO_4): Prepare by either 5.4.1 or 5.4.2.

5.4.1 Standard sulfate solution from H_2SO_4

5.4.1.1 Standard sulfuric acid, 0.1 N: dilute 3.0 ml conc. H_2SO_4 to 1 liter with distilled water. Standardize versus 40.00 ml of 0.05 N Na_2CO_3 solution (5.3) with about 60 ml distilled water by titrating potentiometrically to pH about 5. Lift electrodes and rinse into beaker. Boil gently for 3–5 minutes under a watch glass cover. Cool to room temperature. Rinse cover glass into beaker. Continue titration to the pH inflection point. Calculate normality using

$$N = \frac{A \times B}{53.00 \times C}$$

where:

A = g Na_2CO_3 weighed into 1 liter
B = ml Na_2CO_3 solution
C = ml acid used to inflection point

5.4.1.2 Standard acid, 0.02 N: Dilute appropriate amount of standard acid, 0.1 N (5.4.1.1) to 1 liter (200.00 ml if 0.1000 N). Check by standardization versus 15 ml of 0.05 N Na_2CO_3 solution (5.3).

5.4.1.3 Place 10.41 ml standard sulfuric acid, 0.02 N (5.4.1.2) in a 100 ml volumetric and dilute to the mark.

5.4.2 Standard sulfate solution from Na_2SO_4: Dissolve 147.9 mg anhydrous Na_2SO_4 in distilled water in a 1 liter volumetric flask and dilute to the mark with distilled water.

6. Procedure

6.1 Formation of barium sulfate turbidity

6.1.1 Place 100 ml sample, or a suitable portion diluted to 100 ml, into a 250 Erlenmeyer flask.

6.1.2 Add exactly 5.0 ml conditioning reagent (5.1).

6.1.3 Mix in the stirring apparatus.

6.1.4 While the solution is being stirred, add a measuring spoonful of $BaCl_2$ crystals (5.2) and begin timing immediately.

6.1.5 Stir exactly 1.0 minutes at constant speed.

6.2 Measurement of barium sulfate turbidity

6.2.1 Immediately after the stirring period has ended, pour solution into absorbance cell.

6.2.2 Measure turbidity at 30 second intervals for 4 minutes.

6.2.3 Record the maximum reading obtained in the 4 minute period.

6.3 Preparation of calibration curve.

6.3.1 Prepare calibration curve using standard sulfate solution (5.4).

6.3.2 Space standards at 5 mg/l increments in the 0-40 mg/l sulfate range.

6.3.3 Above 50 mg/l the accuracy decreases and the suspensions lose stability.

6.3.4 Check reliability of calibration curve by running a standard with every 3 or 4 samples.

6.4 Correction for sample color and turbidity.

6.4.1 Run a sample blank using the procedure 6.1 and 6.2 without the addition of barium chloride (6.1.4).

7. Calculations

7.1 Read mg SO_4 from calibration curve

$$\text{mg } SO_4/l = \frac{\text{mg } SO_4 \times 1{,}000}{\text{ml sample}}$$

8. Precision and Accuracy

8.1 Thirty-four analysts in 16 laboratories analyzed six synthetic water samples containing exact increments of inorganic sulfate with the following results:

Increment as Sulfate mg/liter	Precision as Standard Deviation mg/liter	Accuracy as	
		Bias, %	Bias mg/liter
8.6	2.30	-3.72	-0.3
9.2	1.78	-8.26	-0.8
110	7.86	-3.01	-3.3
122	7.50	-3.37	-4.1
188	9.58	+0.04	+0.1
199	11.8	-1.70	-3.4

(FWPCA Method Study 1, Mineral and Physical Analyses).

8.2 A synthetic unknown sample containing 259 mg/l sulfate, 108 mg/l Ca, 82 mg/l Mg, 3.1 mg/l K, 19.9 mg/l Na, 241 mg/l chloride, 0.250 mg/l nitrite N, 1.1 mg/l nitrate N, and 42.5 mg/l total alkalinity (contributed by $NaHCO_3$) was analyzed in 19 laboratories by the turbidimetric method, with a relative standard deviation of 9.1% and a relative error of 1.2%.

Bibliography

1. Annual Book of ASTM Standards, Part 31, "Water", Standard D516-68, Method B, p 430 (1976).
2. Standard Methods for the Examination of Water and Wastewater, 14th Edition, p 496, Method 427C, (1975).

CHLORIDE

Method 325.3 (Titrimetric, Mercuric Nitrate)

STORET NO. 00940

1. Scope and Application
    - 1.1 This method is applicable to drinking, surface, and saline waters, domestic and industrial wastes.
    - 1.2 The method is suitable for all concentration ranges of chloride content; however, in order to avoid large titration volume, a sample aliquot containing not more than 10 to 20 mg Cl per 50 ml is used.
    - 1.3 Automated titration may be used.
2. Summary of Method
    - 2.1 An acidified sample is titrated with mercuric nitrate in the presence of mixed diphenylcarbazone-bromophenol blue indicator. The end point of the titration is the formation of the blue-violet mercury diphenylcarbazone complex.
3. Comments
    - 3.1 Anions and cations at concentrations normally found in surface waters do not interfere.
    - 3.2 Sulfite interference can be eliminated by oxidizing the 50 ml of sample solution with 0.5 to 1 ml of H_2O_2.
4. Apparatus
    - 4.1 Standard laboratory titrimetric equipment including a 1 ml or 5 ml microburet with 0.01 ml graduations.
5. Reagents
    - 5.1 Standard sodium chloride, 0.025 N: Dissolve 1.4613 ±0.0002 g (dried at 600°C for 1 hour) in chloride-free water in a 1 liter volumetric flask and dilute to the mark.
    - 5.2 Nitric acid, HNO_3 solution (3 + 997)
    - 5.3 Sodium hydroxide solution, NaOH, (10 g/l)
    - 5.4 Hydrogen peroxide (30%), H_2O_2
    - 5.5 Hydroquinone solution (10 g/liter): Dissolve 1 g of purified hydroquinone in water in a 100 ml volumetric and dilute to the mark.
    - 5.6 Mercuric nitrate titrant (0.141 N): Dissolve 25 g $Hg(NO_3)_2 \cdot H_2O$ in 900 ml of distilled water acidified with 5.0 ml conc. HNO_3 in a 1 liter volumetric flask and dilute to the mark with distilled water. Filter if necessary. Standardize against standard sodium chloride solution (5.1) using procedure 6. Adjust to exactly 0.141 N and check. Store in a dark bottle. A 1.00 ml aliquot is equivalent to 5.00 mg of chloride.
    - 5.7 Mercuric nitrate titrant (0.025 N): Dissolve 4.2830 g $Hg(NO_3)_2 \cdot H_2O$ in 50 ml of distilled water acidified with 0.5 ml conc. HNO_3 (sp. gr. 1.42) in a 1 liter volumetric flask and dilute to the mark with distilled water. Filter if necessary. Standardize against standard

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sodium chloride solution (5.1) using procedure 6. Adjust to exactly 0.025 N and check. Store in a dark bottle.

5.8 Mercuric nitrate titrant (0.0141 N): Dissolve 2.4200 g $Hg(NO_3)_2 \cdot H_2O$ in 25 ml of distilled water acidified with 0.25 ml of conc. HNO_3 (sp. gr. 1.42) in a 1 liter volumetric flask and dilute to the mark with distilled water. Filter if necessary. Standardize against standard sodium chloride solution (5.1) using procedure 6. Adjust to exactly 0.0141 N and check. Store in a dark bottle. A 1 ml aliquot is equivalent to 500 ug of chloride.

5.9 Mixed indicator reagent: Dissolve 0.5 g crystalline diphenylcarbazone and 0.05 g bromophenol blue powder in 75 ml 95% ethanol in a 100 ml volumetric flask and dilute to the mark with 95% ethanol. Store in brown bottle and discard after 6 months.

5.10 Alphazurine indicator solution: Dissolve 0.005 g of alphazurine blue-green dye in 95% ethanol or isopropanol in a 100 ml volumetric and dilute to the mark with 95% ethanol or isopropanol.

6. Procedure

6.1 Place 50 ml of sample in a vessel for titration. If the concentration is greater than 20 mg/l chloride, use 0.141 N mercuric nitrate titrant (5.6) in step 6.6 or dilute. If the concentration is less than 2.5 mg/l of chloride, use 0.0141 N mercuric nitrate titrant (5.8) in step 6.6, a 1 ml or 5 ml microburet, and determine an indicator blank on 50 ml chloride-free water using procedure 6.6. If the concentration is less than 0.1 mg/l of chloride concentrate an appropriate volume to 50 ml.

6.2 Add 5 to 10 drops of mixed indicator reagent (5.9), shake or swirl solution.

6.3 If a blue-violet or red color appears add HNO_3 solution (5.2) dropwise until the color changes to yellow.

6.4 If a yellow or orange color forms immediately on addition of the mixed indicator, add NaOH solution (5.3) dropwise until the color changes to blue-violet; then add HNO_3 solution (5.2) dropwise until the color changes to yellow.

6.5 Add 1 ml excess HNO_3 solution (5.2).

6.6 Titrate with 0.025 N mercuric nitrate titrant (5.7) until a blue-violet color persists throughout the solution. See 6.1 for choice of titrant normality. Alphazurine indicator solution (5.10) may be added with the indicator to sharpen the end point. This will change color shades. Practice runs should be made.

6.7 Additional steps to eliminate particular interferences:

6.7.1 If chromate is present at < 100 mg/l and iron is not present, add some alphazurine indicator solution (5.10) and acidify to pH 3 (indicating paper). End point will then be an olive-purple color.

6.7.2 If chromate is present at > 100 mg/l and iron is not present, add 2 ml of fresh hydroquinone solution (5.5).

6.7.3 If ferric ion is present use volume containing no more than 2.5 mg of ferric ion or ferric ion plus chromate ion. Add 2 ml fresh hydroquinone solution (5.5).

6.7.4 If sulfite ion is present, add 0.5 ml of H_2O_2 solution (5.4) to 50 ml sample and mix for 1 minute.

7. Calculation

$$\text{mg chloride/l} = \frac{(A - B)N \times 35,450}{\text{ml of sample}}$$

where:
A = ml titrant for sample
B = ml titrant for blank
N = normality mercuric nitrate titrant

mg NaCl/l = mg chloride/l x 1.65

8. Precision and Accuracy

8.1 Forty two analysts in eighteen laboratories analyzed synthetic water samples containing exact increments of chloride, with the following results:

Increment as Chloride mg/liter	Precision as Standard Deviation mg/liter	Accuracy as Bias, %	Bias, mg/liter
17	1.54	+2.16	+0.4
18	1.32	+3.50	+0.6
91	2.92	+0.11	+0.1
97	3.16	-0.51	-0.5
382	11.70	-0.61	-2.3
398	11.80	-1.19	-4.7

(FWPCA Method Study 1, Mineral and Physical Analyses)

8.2 In a single laboratory (EMSL), using surface water samples at an average concentration of 34 mg Cl/l, the standard deviation was ±1.0.

8.3 A synthetic unknown sample containing 241 mg/l chloride, 108 mg/l Ca, 82 mg/l Mg, 3.1 mg/l K, 19.9 mg/l Na, 1.1 mg/l nitrate N, 0.25 mg/l nitrite N, 259 mg/l sulfate and 42.5 mg/l total alkalinity (contributed by $NaHCO_3$) in distilled water was analyzed in 10 laboratories by the mercurimetric method, with a relative standard deviation of 3.3% and a relative error of 2.9%.

Bibliography

1. Annual Book of ASTM Standards, Part 31, "Water", Standard D512-67, Method A, p 270 (1976).

F

APPENDIX F

RESUMES

ARMY CREEK LANDFILL SITE

GANNETT FLEMING

BIOGRAPHICAL SKETCH

LOUIS C. FIORUCCI

TECHNICAL SPECIALITIES:

Environmental Engineering
Hazardous Waste Management
Water/Wastewater Treatment
Air Pollution
Project Management

EDUCATION:

M.H.S.; The Johns Hopkins University; Environmental Health Engineering; In progress
B.S.; University of Maryland; Chemical Engineering; 1974
B.A.; University of Maryland; Chemistry; 1971

PROFESSIONAL AFFILIATIONS:

American Institute of Chemical Engineers
American Chemical Society
American Water Works Association
American Industrial Hygiene Association

EXPERIENCE:

Hazardous Waste Management

Permitting. Participated in preparation of RCRA part B storage and disposal permit for a hazardous waste incinerator facility located at an organic chemical complex. This effort addressed receipt, storage, and disposal of onsite and transported organic wastes, incinerator off gas scrubber effluent treatment, redundant control system safeguards, environmental impact and human health effects from accidental release, and chloro-flurocarbon test burn with six-nines (99.9999%) destruction efficiency.

Thermal Destruction. Directed & Performed feasibility and design study for a rotary kiln incinerator coupled to a cogeneration power plant. Wastes consisted of trash, wood, hazardous solvents, chemicals, paints, and waste sludges. System provided electric power during peak demands and steam to operate light manufacturing and domestic heating needs. Evaluated combustion technologies and emission impacts for thermal destruction of chemical warfare agents and numerous hazardous waste mixtures derived from organic chemical production.

Hazardous Site Investigation. Directed Remedial Investigation and Feasibility Study (RI/FS) for multicomponent organic chemical hazardous waste landfill and PCB contaminated metals dismantling/reclamation facility. Provided assessment of contamination and developed treatment options and costs for remediation. Performed similar task for 5 waste landfills identified by

NYDEC and developed economic estimates for remedial options including no action, secure and cap, remove & secure, slurry wall, leachate pump and treat, and leachate quality monitoring. Closure and monitoring costs were included.

Real Property Investigations. Performed multiplant environmental compliance audits (NPDES, CAA, TSCA, RCRA) and site assessments involving industrial real estate transactions (ECRA-type) for clients in private, government, and utility sectors, and developed risk management plans for corrective and control action.

Environmental Engineering

Landfills. Developed sanitary landfill leachate treatment process train design including several different but compatible unit operations. Leachate quality needed to achieve POTW influent limits.

Solid Wastes. Designed natural gas venting system for harborside fast land area considered for commercial use. Developed chemistry, performed bench tests, and developed process for treating varying concentration of hexavalent chromium and other heavy metal wastes found in aqueous and fill material wastes.

Air Pollution. Performed EPA (methods 1-8) stack emission tests for a large multiproduct chrome chemical process plant, and chlor-alkali plant mercury vapor and chlorine emission control equipment.

Water/Wastewater Treatment

Municipal. Assisted Plant Manager in operation and maintenance of 200 mgd municipal water treatment plant and remote chlorinator stations. Developed organization and implemented process instruments and controls service plan that kept treatment plant fully operational. Modified rapid sand filtration system with mixed media that reduced organics, increased throughput, and saved wash water. Performed chemical/biological analyses and developed instrumentation procadures for pesticide residue analyses.

Industrial. Monitored operation, and made design and operation changes for large scale sulfide precipitation based Mercury removal system discharging to POTW system. Developed and analyzed large scale multistep wastewater treatment system for a major pulp and paper manufacturer. Developed chemistry and processes for treating hexavalent chromium and other heavy metals found in process waste waters.

Chemicals Manufacturing Operations

Chemical Manufacturing. Managed multiplant quality assurance organization with full responsibilities for complete chemical process control, energy management, environmental control/monitoring/reporting, and personnel protection compliance monitoring. Facilities produced chlorine-caustic soda (Hg-cell), sodium chlorite, sodium methylate, calcium hypochlorite (HTH), sulfuric acids, aluminum sulfate, and liquid sulfur dioxide.

Energy. Participated in feasibility and design study for a 24 MWe gas turbine co-generation facility selected for a multiproduct bulk chemicals complex.

Safety/Health. Managed contracts and participated in the evaluation/selection of processes and conducted process safety-hazardous (HAZOP) review for design and operation of chemical agent plant. Prepared preliminary documents for plant environmental risk assessment for chlorine-caustic soda and liquid sulfur dioxide plant expansion projects and a co-generation power plant.

SELECTED PUBLICATIONS:

Remedial Investigation of Major U.S. Copper Refining Facility, 1986, Report No. SBS-71A

Remedial Investigation of Large Scale Passenger/Industrial Tire Manufacturing Facility, 1987. Report No. NYS-71A.

Study of Waste Recovery Incinerator with Cogeneration System for the Tobyhanna Army Depot (TOAD), Pennsylvania, 1988 Report No. USA71M

BIOGRAPHICAL SKETCH

CHEN-YU YEN

TECHNICAL SPECIALTIES	Environmental Chemistry Waste Treatment Processes
EDUCATION:	B.S., 1973, Chemistry, National Taiwan University M.S., 1977, Chemistry, University of North Carolina Ph.D., 1983, Environmental Sciences and Engineering, University of North Carolina

EXPERIENCE: Dr. Yen is a senior engineer/chemist specializing in environmental chemistry and treatment processes, including the assessment of hazardous waste compositions.

He has been working on a potential hazardous waste site project in eastern Maryland. This project involves site investigation, groundwater monitoring, and study for the preparation of a remedial action plan. The groundwater monitoring was conducted to acquire data needed for evaluation of the effect of the landfill on water quality surrounding the site. The analysis of the data indicated raised concentrations of organic contaminants. Further evaluation of the site was conducted in coordination with the Maryland OEP. Dr. Yen is also leading the current Phase II activity which involves investigative measures to mitigate the presence of contamination.

Dr. Yen is also working on the investigation of the Butler Tunnel Superfund site. The project, at this time, involves a Phase I Remedial Investigation associated with the illegal discharge of oils in a mine tunnel. Using data gathered as a result of 1979 oil pollution incident and the 1985 Hurricane Gloria discharge, Dr. Yen is leading the investigation and determination of waste quantities and characterization, the frequency and duration of pollution incidence, the effects on water quality, and potential health effects. The current effort will result in the identification and scope of additional field work and a feasibility study.

Dr. Yen developed groundwater modeling techniques and prepared contaminant plume assessments for the hydrogeologic study of the Tollgate Landfill in Harford County, Maryland.

Dr. Yen had worked on research in different water and wastewater treatment processes, including adsorption, coagulation, and filtration. He is also experienced in chemical and physical characterization of the waste and wastewater generated through coal gasification and liquification processes.

BIOGRAPHICAL SKETCH
CHEN-YU YEN

EXPERIENCE

Dr. Yen had worked on research in different water and wastewater treatment processes, including adsorption, coagulation, and filtration. He is also experienced in chemical and physical characterization of the waste and wastewater generated through coal gasification and liquification processes.

BDRCY005

GANNETT FLEMING

BIOGRAPHICAL SKETCH

THOMAS R. HUNDT

TECHNICAL SPECIALTIES:
Environmental Engineering
Project Management
Solid and Industrial Waste Engineering

EDUCATION:
B.S., 1975, Environmental Science, Rutgers University
M.S., 1977, Environmental Science, Rutgers University
Ph.D., 1985, Environmental Engineering, The John Hopkins University

REGISTRATION:
Registered Sanitarian, American Public Health Association

PROFESSIONAL AFFILIATIONS:
Water Pollution Control Federation
American Water Works Association
Air Pollution Control Association

EXPERIENCE:

Dr. Hundt has successfully managed projects at Gannett Fleming that involved solid and hazardous waste management, environmental engineering evaluations and design, computer modeling and industrial waste treatment.

Dr. Hundt evaluated the results of a groundwater monitoring program at the Tollgate landfill site (Harford County) to establish the extent and characteristics of leachate that is entering the groundwater from the landfill. He evaluated the results of analytical work to identify the pathways that leachate is following as it migrates off-site for use in identification and design of remedial measures including on-site leachate treatment.

He is the lead environmental engineer for the Butler Tunnel Superfund site. He has directed evaluation and modeling of contaminant fate and transport in the colliery and in the Susquehanna River.

Dr. Hundt analyzed groundwater monitoring data obtained as part of leachate migration studies at the Carroll County, Maryland, landfill. He evaluated the effects of the landfill on water and groundwater quality in the vicinity. Study tasks included locating groundwater monitoring wells, evaluation of groundwater quality and flow regime, and projection of quality and quantity factors related to leachate.

BIOGRAPHICAL SKETCH
THOMAS R. HUNDT

EXPERIENCE:

Dr. Hundt, as project manger, is currently preparing a closure plan for a hazardous waste disposal lagoon in Georgia. The lagoon sludge is predominantly creosote with other similar materials resulting from wood preserving operations. The recommended plan called for solidification of the sludge and lined landfill disposal. Dr. Hundt studied the hydrology of this lagoon to determine the groundwater table characteristics. Additionally, he developed criteria for use in design of the fill area, including the selection of clay materials for use as a liner. He designed a leachate collection system that includes connection to sump pumps to deliver leachate to a treatment plant.

He designed elements of the proposed landfill cap for an EPA CERCLA landfill site, and evaluated caps that used clay and synthetic membranes to prevent surface water infiltration into the landfill. Dr. Hundt computed volumes of leachate that would be produced under several design scenarios using water balance techniques, and the leachate volumes were used as a basis for design of leachate collection system components. He investigated a leachate recirculation system to determine the effect of the attenuating capacity of the landfill cap on leachate quality and the "peak shaving" effect that the recirculation would have on the rate of leachate discharge from the landfill. He designed a recirculation system for the landfill.

Additionally, he reviewed the existing data base including RAMP and RI/FS, evaluated data adequacy and reliability, and developed recommendations for additional data collection. He also evaluated the economic and technical feasibility of EPA remedial alternatives.

Dr. Hundt managed a site investigation dealing with chlorinated solvent contamination of groundwater by a municipal landfill. He directed subsurface investigation, contaminant plume modeling, and remedial plan preparation.

Dr. Hundt recently completed an EPA approved course called Health Safety Training for Hazardous Waste Sites. This course covered appropriate techniques for inspection and sampling (air, waste, soil) of sites containing hazardous substances/wastes.

BDRTH009

BIOGRAPHICAL SKETCH

WILLIS W. WHITLOCK, JR.

TECHNICAL SPECIALTIES:	Environmental Planning Environmental Risk Assessment Environmental Impact Analysis Management
EDUCATION:	Master of Public Health, The Johns Hopkins University, School of Hygiene and Public Health, 1984 B.S. Geography and Environmental Planning, Towson State University, 1972
REGISTRATION:	Registered Environmental Sanitarian, Maryland Registration Number 304
PROFESSIONAL AFFILIATIONS:	American Association for the Advance of Science Governmental Refuse Collection and Disposal Association National Environmental Health Association Maryland Association of Sanitarians
EXPERIENCE:	Mr. Whitlock is an author of the 1989 Queen Anne's County, Maryland, Comprehensive Water and Sewer Plan Update. He is contributing to the Gwynns Falls Sewerage System Study conducted for Baltimore County in the area of alternative analysis, selection, and environmental impact. He is participating in planning studies and assessments of hazard and waste sites. Prior to joining Gannett Fleming Environmental Engineers, Mr. Whitlock served the Maryland Department of Health and Mental Hygiene as an Environmental Sanitarian from December 1972 to November 1988 in Harford County. As a member of the Harford County Health Department, Mr. Whitlock was responsible at various times for individual water supply sampling and problem solution; well permitting; mobile home parks; percolation testing; sewage system sizing; permitting and inspection; air pollution; solid waste; hazardous waste; radiation; and noise.