TREATABILITY STUDY REPORT FOR CONTAMINATED SOILS AND SEDIMENTS
The Halby Chemical Site
Wilmington, Delaware

by

PEI Associates, Inc.
11499 Chester Road
Cincinnati, Ohio 45246

Contract No. 68-03-3413
Work Assignment No. 2-60

Technical Project Manager

Mr. Edwin Barth, Jr.
Center for Environmental Research Information
Risk Reduction Engineering Laboratory
Cincinnati, Ohio 45268

RISK REDUCTION ENGINEERING LABORATORY
OFFICE OF RESEARCH AND DEVELOPMENT
U.S. ENVIRONMENTAL PROTECTION AGENCY
CINCINNATI, OHIO 45268

October 1990

CONTENTS

SECTION 1

INTRODUCTION

This report describes the results of treatability studies performed for the Halby Chemical site, Wilmington, Delaware. The overall approach to the treatability studies described in this report has been modeled after information contained in the "Guide for Conducting Treatability Studies Under CERCLA," Interim Final, U.S. Environmental Protection Agency, 1989.

Four treatment technologies were evaluated in the treatability studies: low-temperature thermal desorption, solidification/stabilization, xanthate flotation, and biological treatment. Each technology was evaluated as a primary treatment except low-temperature thermal desorption, which was evaluated as a pretreatment step prior to solidification/stabilization. All technologies were evaluated at the laboratory screening tier, as defined in the treatability guide.

1.1 Site Description

The Halby Chemical site covers approximately 14 acres in a highly industrialized area in Wilmington, New Castle County, Delaware. As Figure 1-1 illustrates, the site is situated in a tidal marshland that is bordered on the north and west by Interstate 495, on the east by Conrail Railroad, and on the south by Terminal Avenue. The Christina River is located east of the site.

The Halby Chemical Company and the Witco Chemical Company produced sulfur compounds from 1948 to 1977. Specific raw materials used in the manufacturing process are shown in Table 1-1, and the products and associated byproducts known to have been produced at the plant are shown in Table 1-2. The three principal

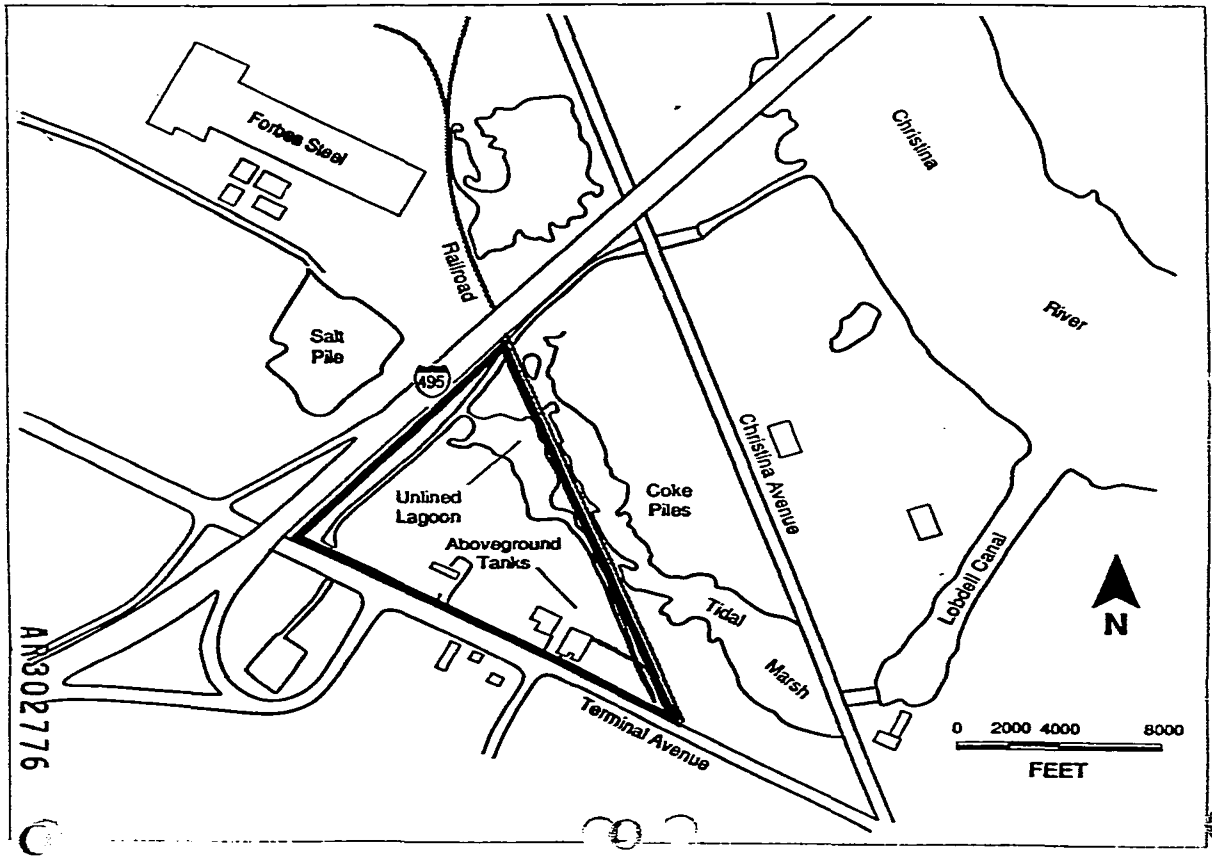


Figure 1-1. Map of Halby Chemical site.

TABLE 1-1. RAW MATERIALS USED AT THE HALBY SITE

Ammonium hydroxide
Anhydrous ammonium
Carbon disulfide
Potassium hydroxide
Sodium hydroxide
Monochloroacetic acid
Isooctyl alcohol
Isopropyl ether
Monoethanolamine
p-Toluene sulfonic acid
Solvay dense soda ash
Sulfuric acid

TABLE 1-2. PRODUCTS AND BYPRODUCTS PRODUCED AT THE HALBY SITE

Ammonium thioglycolate (ATG)
Isooctyl thioglycolate (IOTG)
Ammonium thiocyanate
Sodium sulfide
Sodium thiocyanate
Sodium hydrosulfide
Potassium thiocyanate
Monoethanolamine thioglycolate

chemicals manufactured or used at the chemical facility were ammonium thioglycolate (ATG), isooctyl thioglycolate (IOTG), and ammonium thiocyanate.

From 1948 to 1964 the wastewater, cooling water, and surface runoff were discharged into an unlined lagoon (see Figure 1.1). The lagoon waters discharged to the Christina River through a drainage ditch connected to Lobdell Canal east of the site. The lagoon presently receives runoff from the railroad tracks on the east side of the site and from the highway northwest of the site. Currently, a drainage ditch along I-495 drains the lagoon waters during tidal fluctuations into the Christina River. Although chemical production activities stopped in 1977, the site is still used for storage of carbon disulfide in aboveground tanks. Areas adjacent to the site also are used for storage of coke piles (east of the site) and for truck washing (west of the site).

According to a 1983 Delaware Department of Natural Resources and Environmental Control (DNREC) preliminary assessment, nearby residents had complained about lagoon overflow, sulfurous odors, and numerous spills. In late 1985 or early 1986, the EPA FIT III Team conducted a site inspection of the facility, and detected sulfurous odors from stained sediments, drums, and tanks found on site. In 1987, EPA began a Remedial Investigation/Feasibility Study (RI/FS) at the site. Analyses of sediment, surface and subsurface soils, and surface and ground water indicated contamination by volatile organic compounds (VOCs), semivolatile organic compounds, and inorganic compounds (metals, ammonia, and cyanide compounds). Migration from the site was evident in aqueous and sediment samples from the lagoon outfall, which contain elevated levels of various pollutants. At the time of the inspection, the one remaining on-site well showed high levels of arsenic, mercury, cyanide, and trichloroethylene. The presence of these substances indicated that the ground water underlying the site was probably contaminated.

1.2 Waste Stream Description

Tables 1-3 through 1-5 list the detected VOCs, semivolatiles, and inorganic compounds, respectively, their concentration ranges, and the frequency of detection in water and soil samples. This information was compiled from preliminary data received from the Phase I Remedial Investigation conducted by Ebasco Services, Incorporated. As these tables show, the site soils and sediments are contaminated by a complex mixture of VOCs (including carbon disulfide, chlorinated ethylenes, and benzene compounds), semivolatiles (including pyrene, benzo[a]pyrene, phenanthrene, fluorene, chrysene, fluoranthene, and acenaphthene), and inorganic compounds (ammonium thiocyanate, arsenic, copper, cobalt, lead, manganese, mercury, vanadium, and zinc) at widely varying concentrations ranging from approximately 100 ppb to 1 percent. In the sediments and surface soils, the highest concentrations of these compounds appear to be located at the southern end of the site near the tanks and chemical plant building, with lower levels in the northern and eastern portions. Subsurface soils are also contaminated with similar VOCs, semivolatiles, and inorganic compounds to a depth of approximately 10 feet.

The lateral and vertical extent of contamination at the Halby Chemical site is complex and variable. The coexistence of various classes of compounds will heavily influence the remediation activities chosen for the site. Remediation may require more than one treatment technology in a treatme[illegible] train process. In addition, in situ

TABLE 1-3. VOLATILE CONTAMINANTS AT THE HALBY SITE*

	Sediment		Subsurface		Surface Soil		Groundwater		Surface Water	
Parameters	(ug/kg)	%	(ug/kg)	%	(ug/kg)	%	(ug/L)	%	(ug/L)	%
Chloromethane	ND	0	0-1330	89	ND	0	0-0.4	4	ND	0
Vinyl Chloride	0-720	3	ND	0	ND	0	0-130	4	ND	0
Methylene Chloride	71-1500	100	0-2760	78	0-27000	86	0-780	84	0-230	92
Acetone	29-12000	100	21.9-1420	100	0-100000	95	0-860	84	0-170	85
Carbon Disulfide	320-9400000	100	0-43200	56	0-1800000	66	0-2400	16	0-2600	46
1,1,- Dichloroethane	0-14	6	ND	0	ND	0	ND	0	ND	0
1,1,- Dichloroethene	ND	0	0-.5	11	ND	0	ND	0	ND	0
Trans 1,2 Dichloroethene	0-2000	3	ND	0	ND	0	ND	0	ND	0
Chloroform	0-20	3	ND	0	ND	0	0-24	12	0-9	8
2-Butanone	0-1900	71	10.1-4700	100	0-13000	71	0-3	4	0-20	69
1,1,1- Trichloroethane	0-530	6	ND	0	0-7	14	ND	0	ND	0
Carbon Tetrachloride	ND	0	ND	0	0-6	5	ND	0	ND	0
Vinyl Acetate	ND	0	ND	0	0-12	5	ND	0	ND	0
Bromodichloromethane	ND	0	ND	0	0-6	5	ND	0	0-2	8
Trichloroethene	0-5200	12	ND	0	ND	0	0-21	8	0-3	8
Benzene	0-470	9	ND	0	0-190	10	0-0.2	4	ND	0
CIS 1,3 - Dichloropropene	ND	0	0-3.4	11	ND	0	ND	0	ND	0
4-Methyl-2-Pentanone	0-630	15	ND	0	0-770	33	ND	0	ND	0
2-Hexanone	0-210	3	ND	0	0-1200	19	0-2	4	ND	0
Tetrachloroethene	0-5100	15	0-21200	33	0-16000	24	0-18	4	ND	0
1,1,2,2-Tetrachloroethane	ND	0	ND	0	0-28	5	ND	0	ND	0
Toluene	0-19000	29	0-399	44	0-480	43	ND	0	0-1	15
Chlorobenzene	ND	0	ND	0	0-1	5	ND	0	ND	0
Ethyl Benzene	0-430	15	0-170	22	0-56	10	ND	0	ND	0
Styrene	ND	0	ND	0	0-15	10	ND	0	ND	0
Total Xylenes	0-3800	21	0-1167	22	0-490	19	ND	0	0-1	8

*Information compiled from Phase I Remedial Investigation preliminary data, Ebasco-Services, Incorporated.

TABLE 1-4. SEMIVOLATILE CONTAMINANTS AT THE HALBY SITE*

	Sediment		Subsurface		Surface		Groundwater		Surface Water	
Parameters	(ug/kg)	%	(ug/kg)	%	(ug/kg)	%	(ug/L)	%	(ug/L)	%
Phenol	0-1600	9	0-1600	44	ND	0	ND	0	ND	0
1,3-Dichlorobenzene	0-94	3	ND	0	ND	0	ND	0	ND	0
1,4-Dichlorobenzene	ND	0	ND	0	0-54	5	ND	0	ND	0
Benzyl Alcohol	ND	0	ND	0	ND	0	ND	0	0-1	14
2-Methylphenol	ND	0	ND	0	ND	0	ND	0	0-3	12
4-Methylphenol	0-5300	29	0-3200	11	0-54	5	ND	0	0-6	19
Benzoic Acid	0-4500	15	0-2700	44	0-1100	5	0-4	4	ND	0
Naphthalene	0-3000	29	0-1420	33	0-1300	29	ND	0	ND	0
2-Methylnaphthalene	0-6400	35	0-280	22	0-2800	29	ND	0	ND	0
Fluorene	0-2000	26	0-470	33	0-20000	14	ND	0	ND	0
Acenaphthylene	0-600	18	0-130	22	ND	0	ND	0	ND	0
Acenaphthene	0-2800	18	0-480	22	0-27000	5	ND	0	ND	0
N-Nitrosodiphenylamine	ND	0	0-140	11	ND	0	ND	0	ND	0
Hexachlorobenzene	ND	0	0-180	11	ND	0	ND	0	ND	0
Dibenzofuran	0-130	6	0-190	22	0-14000	10	ND	0	ND	0
Pentachlorophenol	ND	0	0-410	11	ND	0	ND	0	ND	0
Diethylphthalate	0-170	3	ND	0	ND	0	ND	0	0-1	4
Phenanthrene	0-6800	50	0-2600	78	0-110000	33	ND	0	ND	0
Anthracene	0-1400	29	0-630	33	0-19000	19	ND	0	ND	0
Di-N-Butylphthalate	0-4100	26	0-150	44	0-2000	43	ND	0	0-2	35
Fluoranthene	0-4900	71	0-1800	78	0-85000	43	ND	0	ND	0
Pyrene	0-3800	71	0-2000	78	0-66000	62	0-1	4	ND	0
Butylbenzylphthalate	0-2800	12	0-2000	33	0-900	5	ND	0	ND	0
Benzo(a)Anthracene	0-1800	38	0-1100	33	0-19000	24	ND	0	ND	0
Chrysene	0-2600	44	0-1300	56	0-22000	33	ND	0	ND	0
Bis(2-Ethylhexyl)Phthalate	0-4400	65	0-1900	78	0-24000	76	0-160	36	0-26	73
Benzo(b)Fluoranthene	0-2200	32	0-1800	66	0-12000	24	ND	0	ND	0
Benzo(k)Fluoranthene	0-1900	15	0-510	22	0-11000	10	ND	0	ND	0
Benzo(a)Pyrene	0-1900	32	0-1100	89	0-7900	14	ND	0	ND	0
Indeno(1,2,3-cd)Pyrene	0-170	9	0-460	22	0-4200	5	ND	0	ND	0
Dibenzo(a,h)Anthracene	ND	3	0-370	22	ND	0	ND	0	ND	0
Benzo(g,h,i)Perylene	0-260	3	0-580	22	0-5000	5	ND	0	ND	0

*Information compiled from Phase I Remedial Investigation preliminary data, Ebasco Services, Incorporated.

TABLE 1-5. INORGANIC CONTAMINANTS AT THE HALBY SITE*

	Sediment		Subsurface		Surface		Groundwater		Surface Water	
Parameters	(mg/kg)	%	(mg/kg)	%	(mg/kg)	%	(ug/L)	%	(ug/L)	%
Arsenic	4.9-3110	100	10.4-1670	100	1.5-4430	100	0-2100	49	0-84	62
Barium	13-843	100	52.3-309.3	100	0.82-393	100	0-2210	88	56.4-179	100
Cadmium	0-128	91	0-13.5	56	0-15	62	0-87	22	0-44.3	19
Chromium	7.6-243	100	18.2-125.6	100	0-343	95	0-689	35	0-370	35
Cobalt	18.3-327	100	9.4-269	100	0-71.6	95	0-45500	53	0-58.2	92
Copper	27.4-26800	100	14.1-4950	100	1.6-3870	100	0-423	41	0-56.8	58
Lead	10.6-2930	100	8.7-1100	100	0.3-443	100	0-278	37	0-167	58
Manganese	47.7-1350	100	98.3-300	100	4.1-2680	100	0-185000	98	43-7590	100
Mercury	0-7.7	76	0.6-80.6	100	0-43	90	0-0.6	10	0-0.3	8
Vanadium	9.1-627	100	16.3-66.2	100	9.6-135	100	0-1290	49	0-42.2	23
Zinc	38.8-16400	100	56-4940	100	3.7-6690	100	0-416000	86	9.3-2320	100
TOC	407-7220	100	ND	0	ND	0	ND	0	ND	0
Total Cyanide	0-22	37	0-1.29	17	0-40	67	0-884	32	0-11800	46
Thiocyanate	0-940	17	0-700	17	0-410	19	0-1070000	40	0-19500	23

---

*Information compiled from Phase I Remedial Investigation preliminary data, Ebasco Services, Incorporated.

technologies may be more appropriate than aboveground technologies for treating and water.

1.3 Technology Descriptions

Since a complex chemical mixture of volatile, semivolatile, and inorganic compounds is present in soils and sediments at the Halby Chemical site, several treatment technologies may be applicable for remediation of the soil. In the treatability studies, described herein, solidification/stabilization, xanthate flotation, and biological treatment were each evaluated for applicability as a primary treatment. Low-temperature thermal desorption was tested as a pretreatment step prior to solidification/stabilization. These studies were designed to evaluate the applicability of these technologies to treat the contaminated soils and sediment.

Figure 1-2 presents the overall treatability scheme for this project, and Table 1-6 presents the experimental plan. Table 1-7 summarizes the analytical testing program for the soil, water, and treatment residues.

TABLE 1-6. EXPERIMENTAL PLAN

	Matrix	Test conditions	No. of replicates	Total No. of test runs
Low-temperature thermal desorption	Surface soil/sediment	3 temperatures x 2 residences	2	8
Solidification/stabilization	Thermal residues	2 binders x 2 mix + ratios + 4 blanks	2	12
Solidification/stabilization	Surface soil/sediment	1 binder x 2 mix ratios	2	4
Xanthate flotation	Surface soil/sediment	1 reagent/frother	1	2
Biodegradation	Sediment/ground water composite	2 treatments + 2 controls	1	4

Soil from the Halby site was subjected to physical, chemical, and biological characterization tests to delineate the soil characteristics that may influence treatment effectiveness. Each separate soil sample (surface soil and sediment) was homogenized prior to chemical analysis and testing to ensure that representative samples were tested for each technology and that the results from those technologies with similar starting matrices could be compared. The soil was analyzed for a select list of

ORIGINAL (Red)

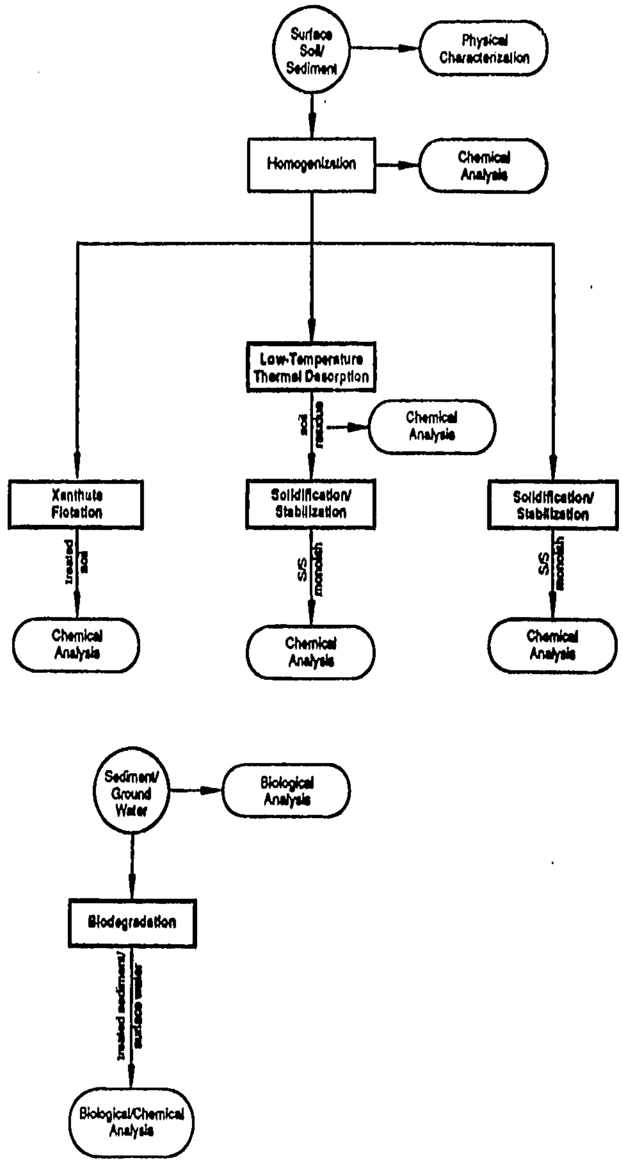


Figure 1-2. Overview of treatability scheme.

TABLE 1-7. SUMMARY OF ANALYTICAL TESTING

Parameter	Low-temperature thermal desorption		Solidification/ stabilization		Xanthate flotation		Biological studies	
	Untreated soil	Treated soil	Untreated soil	Treated soil	Untreated soil	Treated soil	Water	Composite
VOC	2	8						
Semivolatiles	2	8						
Metals	2	8	4[a]	16[a]	1[b]	2[b]		
Other inorganics	2	8						
Biological parameters							1	4
Other parameters	2		4	8			1	4

VOCs

Carbon disulfide
Tetrachloroethene
Methylene chloride
Toluene
2-Butanone

Semivolatiles

Chrysene
Pyrene
Fluoranthene
Benzo(b)fluoranthene
Benzo(k)fluoranthene
Benzo(a)pyrene

Metals

Arsenic
Chromium
Cobalt
Copper
Mercury
Zinc

Inorganics

Ammonia
Cyanide (total)

Biological Parameters

TOC (total organic carbon)
Oxygen consumption
Microbial density
Nitrogen (as ammonia)
Orthophosphate
pH

Other parameters

UCS (s/s)
Moisture content (LTTD)

---

[a] TCLP extracts analysis.

[b] Because no visible separation occurred, these analyses were not performed.

indicator compounds (Table 1-7) to provide initial concentration data for determining the effectiveness of the technologies. The surface soil and sediment also were analyzed for microbial activity to assess the applicability of biodegradation.

The low-temperature thermal desorption studies were performed at two temperatures and two residence times. The VOC, semivolatile, and metal indicator compounds were measured in the soil residues from all tests. Duplicate test runs were conducted on soil samples with high concentrations of VOC and semivolatile contaminants (i.e., a worst-case scenario).

The solidification/stabilization studies of residue from low-temperature thermal desorption involved two binders [asphalt and portland cement (Type II)] at two different mix ratios. Solidification/stabilization of untreated surface soils and sediments involved one binder (cement) at two different mix ratios. Duplicate test runs were conducted. The unstabilized soil and the stabilized products (including four blanks) were subjected to leaching by TCLP, and the extracts were analyzed for the metal indicator compounds.

Xanthates are the reaction products of carbon disulfide, alcohol and an alkali-metal hydroxide. The initial alcohol/hydroxide reaction forms an alkoxide, which then reacts with carbon disulfide to give the alkali-metal xanthate. Alkali metal xanthate salts are soluble in water and readily decompose in acidic environments to liberate carbon disulfide and the corresponding alcohol. Xanthates are used extensively in the minerals processing industry as collectors in the selective separation of nonferrous metal sulfide ores from gangue (mixtures of undesirable ores, silicates, and non-ore material). This process exploits a surface chemistry phenomenon, where the xanthate compound selectively coats the metal sulfide particle, increasing its hydrophobicity and affinity to gas bubbles. The bubbles lift the metal sulfide particles to the surface, where they can be skimmed off and collected in a separate vessel. The degree of flotation accomplished is dependent upon the particular xanthate chosen and the presence of activators, such as cupric sulfate, or depressants, such as cyanide salts. Frothing agents can be added to enhance the life of the bubbles and allow for a more efficient separations. Although this process has been extensively used in the mining industry, it has not been previously demonstrated on contaminated soils. Nevertheless, the presence of high concentrations of carbon disulfide in the surface soils and in aboveground tanks onsite made this mining process a candidate for testing at the laboratory screening level using a preformulated xanthate reagent (potassium amyl xanthate) with a frothing agent (2-ethylhexanol). The soil and the recovered froth were analyzed for the metal indicator compounds.

The biological studies involved an initial evaluation of the existing microbial population in sediment and ground water, followed by a series of treatments evaluating the effects of oxygen and nutrients on the indigenous population. Microbial growth and oxygen consumption were measured to evaluate the potential for biological treatment of carbon disulfide and thiocyanate compounds in sediment and ground water.

1.4 Project Objectives

The objective of these treatability studies was to determine the applicability of low-temperature thermal desorption, solidification/stabilization, xanthate flotation, and biodegradation for treatment of surface soils and sediments at the Halby Chemical site. The laboratory screening studies were designed to determine the applicability of these technologies quickly and relatively inexpensively; they were not designed to evaluate process parameters or to develop cost data.

Table 1-8 summarizes the treatment objectives for the various indicator compounds, which are the primary contaminants of concern at the site. These objectives are based on human health and environmental risks (soil), and previous experience for the tests (TCLP extract and UCS). The testing and characterization program for each technology is discussed in more detail in Sections 4 through 7.

Data generated under this work assignment will be used by Region III to identify those technologies that are potentially applicable for remediation of the Halby Chemical Superfund site. The data will be used to screen out technologies that do not demonstrate effective removal of contaminants from the soil, and to retain those technologies that either result in a significant reduction in contamination or, in the case of biological treatment, indicate the potential for significant reduction of contaminant

ORIGINAL
(Red)

TABLE 1-8. TREATMENT OBJECTIVES FOR TREATABILITY STUDIES

Indicator compound	Soil[a]	TCLP extract[b], mg/L
Semivolatiles (low-temperature thermal desorption)		
Benzo(a)pyrene	8 mg/kg[c]	NA[d]
Chrysene	8 mg/kg[c]	NA
Fluoranthene	8 mg/kg	NA
Benzo(b)fluoranthene	8 mg/kg	NA
Benzo(k)fluoranthene	8 mg/kg	NA
Metals (solidification/stabilization and xanthate flotation)		
Arsenic	50 mg/kg	1
Cobalt	1,000 mg/kg[e]	1
Copper	300 mg/kg	1
Chromium	NS[f]	1
Zinc	260 mg/kg	1
Mercury	1 mg/kg	1
Physical parameters (solidification/stabilization)		
Unconfined compressive strength	50 psi[b]	NA

[a] Based on NOAA-recommended levels for sediments in the marsh area next to the Halby site, except as noted.

[b] Arbitrary values set for study.

[c] Based on 10^{-6} human health risk levels.

[d] NA = Not applicable.

[e] Based on preliminary target cleanup levels for human health (EBASCO 2/90).

[f] NS = Not specified.

levels. Depending on results of the laboratory testing, the data may be used to select a remedy for the site to be included in the record of decision (ROD) due in December 1990, or to identify further studies necessary for more in-depth evaluation of these technologies. The less-stringent data quality objectives and statistical designs associated with laboratory screening could result in a potentially applicable alternative being rejected or a nonapplicable alternative being retained for further testing. The risk of this occurring, however, is acceptable in light of the cost and time savings associated with laboratory screening treatability studies.

ORIGINAL
(Red)

SECTION 2

CONCLUSIONS AND RECOMMENDATIONS

Based on these preliminary studies, the following overall conclusions are drawn:

- Bacteria capable of degrading carbon disulfide and thiocyanate compounds are present in the soils under aerobic conditions with sufficient nutrient supply. The presence, growth and metabolism of aerobic carbon disulfide and thiocyanate degraders suggest the possibility of using an aboveground bioreactor treatment system. Further studies are needed to more fully evaluate the performance of bioremediation.
- Xanthate flotation/separation of heavy metals from soils was not successful under the limited experimental conditions tested.
- While low-temperature thermal desorption at temperatures between 300° and 500°F and between 15 and 30 minutes residence time can successfully remove VOCs and semivolatile organic compounds from soils, it may not be needed as a pretreatment step prior to solidification/stabilization.
- The soils, themselves, do not leach metals at appreciable levels under TCLP test conditions. However, among the two binders tested, asphalt binder appears to be the better binder material for reducing leachate concentrations of the metal indicator compounds, although dilution of soils by the binder was not taken into account. Additional feasibility and treatability studies are needed if solidification/stabilization is deemed necessary to reduce metal leaching from soils to surface and ground water.

The following additional studies are recommended prior to selecting a final remedy for soils at the Halby Chemical site:

- Further xanthate flotation laboratory studies examining the effects of (1) *mixing speeds, (2) particles size, (3) water to solids mix ration, (4)* xanthate flotation agent and concentration, and (5) frother concentration/bubbler flow speeds.

- Additional TCLP or other leach testing (preferably a multiple waste extraction test or dynamic, long-term leach study) of soils to adequately determine the need for solidification/stabilization to reduce the leaching of metals from the soils to the surface and ground waters.

- Upon determining the need for solidification/stabilization of soils, perform feasibility analysis comparing costs for cement-based and asphalt-based solidification/stabilization processes taking into account the need for dewatering prior to using asphalt. Upon determining the more feasible solidification/stabilization process, perform additional bench-scale studies evaluating leachate levels of semivolatile and metal indicator compounds, and VOC emissions during mixing, at binder to soil mix ratios between 0.5 and 0.25 (for asphalt) or between 0.24 and 0.40 (for cement).

These additional studies could not be performed under the existing scope and budget, but they are needed to build upon data presented in this paper and to help further refine the design, cost and performance for soil treatment alternatives.

SECTION 3

FIELD SAMPLE COLLECTION AND SAMPLE PREPARATION

On April 10 and 11, 1990, PEI, together with EPA/ERT, collected surface soils/sediments, subsurface soils, and ground water for the treatability studies. Because of uncertainties in the available analytical data, soils were analyzed for selected metals in the field by EPA/ERT's contractor, Roy F. Weston, using an X-ray fluorescence (XRF) spectrometer. This field analytical procedure was used to select soil sampling locations and to assure that the treatability study samples had metal concentrations representing general site contamination.

This section presents the approach and procedures that were used to collect samples in the field and to prepare samples for the treatability studies. Subsection 3.1 summarizes the overall sampling program and objectives. Subsection 3.2 covers the basis for selecting field sampling sites. Subsection 3.3 discusses procedures for collecting samples, sampling equipment, equipment decontamination, sample quantities, sample logging and documentation, sample preservation, and shipping procedures. Subsection 3.4 describes the manner in which field-activity-generated wastes were managed. Subsection 3.5 discusses procedures used to homogenize the samples to the extent practical prior to instituting treatability experiments.

3.1 Objectives of Field Sample Collection Program

The general objective of the field sample collection program was to collect soil and water samples from the Halby site necessary for the completion of treatability studies. Sufficient quantities of material were collected to conduct treatability studies and physical-chemical characterization tests on materials ranging from average to worst-case concentrations of volatile and semivolatile organic and inorganic indicator compounds. The sample collection procedures were conducted to minimize losses of volatile constituents so that samples would be representatives of general conditions present on site.

Table 3-1 gives an overview of the field sampling program for each technology. Surface soil/sediment samples were collected for the low-temperature thermal desorption, solidification/stabilization, xanthate flotation, and biological treatment studies. Subsurface soil samples were also collected in case EPA wished to conduct additional treatability studies (e.g., soil washing). Groundwater samples were collected for biological treatment studies.

TABLE 3-1. OVERVIEW OF FIELD SAMPLING PROGRAM FOR EACH TECHNOLOGY

Treatment technology	Sample matrices[a]	Sample quantity[b]	Sample location[c]
Low-temperature thermal desorption and solidification/stabilization	SSS	17.2 kg	SSS-L
Xanthate flotation	SSS	2 kg	SSS-X
Other treatability studies	SUBS	8.5 kg	SUBS-S
Biological treatment	SSS GW	1.5 kg 4 liters	SSS-B GW-B

[a] SSS = Surface soil/sediments (0 to 2 ft); SUBS = subsurface soil (2 to 4 ft); GW = ground water.

[b] Excess quantity of sample was collected to allow for contingencies.

[c] See Figure 3-1 for sample locations. -X = xanthate; -L = low-temperature thermal desorption; -S = soil washing; -B = biological.

3.2 Selection of Sampling Sites for Treatability Testing

In selecting sampling sites at Halby for the treatability studies, the following issues were considered:

- Distribution of site contaminants and contaminant concentrations.
- Site characteristics limiting access to sample locations.

These issues were discussed in the Revised Quality Assurance Project Plan (QAPjP) for this project (April 1990) and are not repeated here.

Figure 3-1 shows the locations of the selected sampling sites. Groundwater samples for the biological treatment studies (GW-B) were collected from the on-site

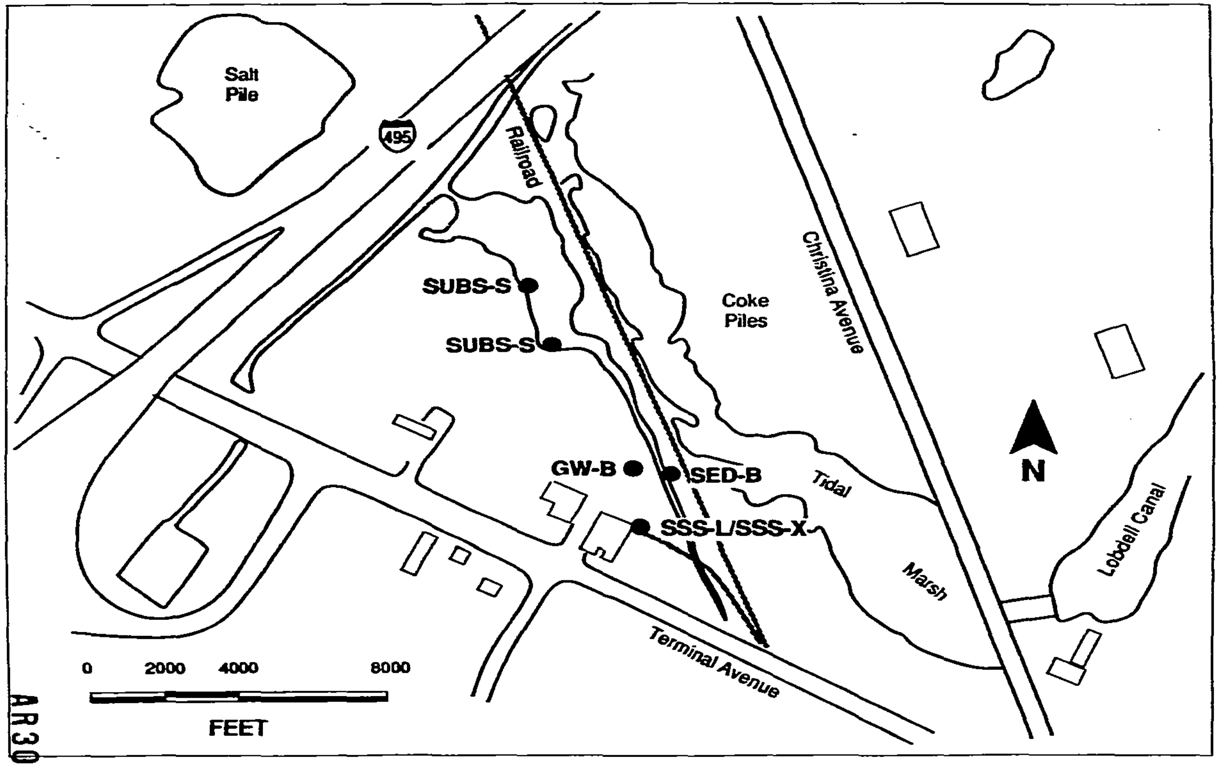


Figure 3-1. Location of sampling sites

ORIGINAL (Red)

monitoring well (SMW-01). Surface soil/sediment samples for the low-temperature thermal desorption, xanthate flotation, and biological treatment studies (SSS-L, SSS-X, and SSS-B) were collected from the drainage ditch and in the plant area near the aboveground storage tanks. The subsurface soil samples for other treatability studies (SUB-S) were collected from two points in the lagoon north of the plant area. Because of the uncertainty in the actual chemical concentrations at these selected locations, field analytical screening techniques (X-ray fluorescence spectroscopy and organic vapor monitoring) were used to verify the presence of metals and volatiles in the collected samples.

3.3 Field Sampling and Analysis Procedures

The following topics associated with field sampling activities are discussed separately below:

- Sample quantities required, sample containers, and preservation or (Subsection 3.3.1)
- Sampling methods (subsection 3.3.2)
- Field analytical screening methods (Subsection 3.3.3)
- Equipment decontamination (Subsection 3.3.4)

3.3.1 Sample Quantities Required, Sample Containers, and Preservation

Table 3-2 lists the sample quantities required for individual digestion procedures and those requested by the laboratory for test material characterization. The amounts requested by the laboratory are greater to accommodate the necessary quality assurance/quality control (QA/QC) procedures. Table 3-3 presents the total amounts of soil and ground water that were collected for the initial soil characterization and for evaluation of each treatability technology.

The surface soil/sediment samples for the low-temperature thermal desorption, solidification/stabilization, and xanthate flotation studies (SSS-L and SSS-X) were collected in a single 30-gallon steel drum. The subsurface soil samples for the additional treatability studies (SUB-S) were collected in two 5-gallon steel containers.

TABLE 3-2. QUANTITY OF MATERIAL REQUIRED FOR INDIVIDUAL SAMPLE ANALYSES

Parameter	Matrix	Quantity needed for digestion	Quantity requested by laboratory
Volatiles	Solid	5 g	Two 4-oz jars
Semivolatiles	Solid	30 g	8 oz total for semivolatiles, metals, and other inorganics
Metals	Solid	10 g	8 oz total for semivolatiles, metals, and other inorganics
Other inorganics	Solid		
Cyanide		5-10 g	8 oz total for semivolatiles, metals, and other inorganics
Ammonia		5-10 g	
Physical	Solid	NA[a]	1 kg
Leach testing (TCLP)	Solid	100g	0.5 kg
Biological	Solid	NA	0.5 kg
Volatiles	Liquid	5 ml	80 ml
Semivolatiles	Liquid	1000 ml	2000 ml
Metals	Liquid		250 ml
Arsenic		30 ml	
Others		150 ml	
Other inorganics	Liquid		
Cyanide		500 ml	1000 ml
Ammonia		50 ml	50 ml
Biological	Liquid	NA	1000 ml

[a] NA = Not applicable.

TABLE 3-3. TOTAL QUANTITY OF MATERIAL REQUIRED FOR ANALYSIS AND TESTING

Technology	Surface soil/sediment, kg	Subsurface soil, kg	Ground water, liters
Low-temperature thermal desorption			
Initial physical characterization	1.0		
Initial chemical characterization (including TCLP)	0.9		
Treatability tests, 8 runs at 0.8 kg/run			
	6.4		
Solidification/stabilization (thermal residues)			
Characterization of low-temperature thermal desorption residue (including TCLP)	0.9		
Treatability tests, 16 samples (UCS and TCLP)			
	5.8		
Solidification/stabilization (untreated soil)			
Initial physical characterization			
Initial chemical characterization (including TCLP)	1.0		
Treatability tests, 8 samples (UCS and TCLP)	0.9		
Xanthate flotation	2.9		
Initial chemical characterization (including TCLP)			
Initial physical characterization			
Treatability tests, 4 runs at 0.2 kg/run			
	0.7		
	1.0		
	0.8		
Other treatability studies (soil washing)			
Initial chemical characterization (including TCLP)		1.2	
Initial physical characterization			
Soil sieving		1.0	
Treatability tests, 12 runs at 0.4 kg/run		2.0	
		4.8	
Biodegradation			
Biological characterization	0.5		
Treatability tests 8 runs	1.0		4.0
Total	23.8	9.0	4.0

ORIGINAL
(Red)

All sample containers were filled and sealed in a manner which minimizes headspace. All samples were kept cool prior to and during shipment to the testing facilities.

3.3.2 Sampling Methods

The sampling and field screening equipment used for obtaining samples from Halby site are listed in Table 3-4. The Standard Operating Procedure for collecting solid and aqueous samples at the Halby Chemical site is provided in Appendix B-5.

TABLE 3-4. FIELD SAMPLING AND ANALYSIS EQUIPMENT

- Sampling/analysis equipment
  - Steel round-point shovel or other trenching shovel
  - Steel hand-coring device and scoops
  - Steel hand auger or post-hole digger (Iwan-type)[a]
  - Teflon bailers
  - Portable photoionization detector (11.7eV lamp)[a]
  - Plastic tarp
  - Wood planks
  - X-ray fluorescence detector, sieves, microwave oven[a]
- Sample containers
  - Steel buckets (5 gallons) with locking lids
  - Precleaned presterilized sample containers
    - 1-gallon brown glass jugs
    - 3-gallon wide-mouth glass jars
  - DOT 17H open-head steel drum
- Decontamination equipment[a]
  - Alconox
  - Methanol
  - Distilled or deionized water
  - Tap water
  - Brushes/scrubbers
  - Pails
  - Plastic tarp
  - Trash bags
  - Kem-wipes
  - Plastic tubs
- Sample packing equipment
  - Sample cooler (shock resistant)
  - Blue ice/dry ice
  - Packing tape
  - Packing materials (vermiculite/wood chips)
  - Plastic bags
  - Appropriate manifest, shipping forms, and labels, custody seals
- Personal protection equipment
  - As required by Site Health and Safety Plan

[a] Provided by EPA Emergency Response Team (ERT) contractor.

Surface Soil/Sediment Sampling--

Three surface soil/sediment samples were collected and placed into a 30-gallon DOT 17H open-head steel drum--one each for low-temperature thermal desorption and solidification/stabilization of residue (15.0 kg), solidification/stabilization of untreated soils (4.8 kg), and xanthate flotation treatment (2.5 kg). The surface soil/sediment sample collected for biological treatment (1.5 kg) was placed into a 3-gallon, wide-mouth, sterilized jar. The location of these surface soil/sediment samples was in the southeastern corner of the site near the tanks and chemical plant (Figure 3-1) and in the drainage ditch. Excess sample was collected in each case in order to fill the sample containers and to provide excess sample for additional analyses or tests. Surface soil/sediment samples were taken at depths of 0 to 2 feet using steel shovels and stainless steel scoops or spoons. The 30-gallon drum was completely filled with surface soil/sediment and sealed. Samples collected in the field for the biological treatment studies (SSS-B, GW-B) were stored in precleaned, sterilized sample containers fitted with Teflon-lined lids. Special care was taken to minimize sample container contamination and exposure of the samples to light, air, and dust during sampling.

Subsurface Soil Sampling--

One 9.0-kg subsurface soil sample was collected for other treatability studies and placed in two 5-gallon steel containers. This sample was collected from the lagoon area indicated in Figure 3-1. The sample was collected with an auger at a depth of 2 to 4 feet below the ground surface. Samples quantities collected were in excess of 9 kg, in order to completely fill the two 5-gallon steel drums.

Ground-Water Sampling--

A 4-liter ground water sample for biodegradation was collected from Monitoring Well SMW-01, which has been identified as having the highest concentrations of volatile, semivolatile, and inorganic contaminants. According to the standard operating procedure provided in Appendix B-5, a bailer was used to collect a groundwater sample after the well was purged. Four well volumes were withdrawn from the well prior

ORIGINAL (Red)

to collecting the groundwater sample.

3.3.3 Field Analytical Screening Methods

Subsequent to sample collection, the samples were delivered to EPA/ERT's contractor, Roy F. Weston, for on-site X-Ray Fluorescence (XRF) analysis. Samples were dried in a microwave oven, sieved through a 20 mesh sieve, and homogenized before analysis. XRF results were used to identify the following elements in the site soils: zinc, lead, chromium, copper, and arsenic. Analyte values were determined by using a generic hazardous waste soil calibration model standard. Since this calibration model did not utilize site specific soil standards, values were reported as estimates rather than as actual concentrations. A second calibration model was used to recalculate values for lead from the sample spectra. All results were reported in parts per million (ppm). Appendix D contains results obtained from the field XRF instrument.

3.3.4 Equipment Decontamination

All nondisposable sampling equipment used at the Halby site was decontaminated using the following method:

1) Wash and scrub with low-phosphate detergent.
2) Rinse with tap water.
3) Rinse with 10 percent nitric acid.
4) Rinse with tap water.
5) Rinse with methanol.
6) Rinse with deionized water.
7) Air dry.
8) Wrap in aluminum foil for transport to sampling locations.

These steps were followed prior to each use and after the last use. Care was taken to avoid contact of the foil-wrapped sampling equipment with the ground.

3.4 Field-Sampling-Derived Waste Management

Wastes generated during the sampling activities included:

- Disposable equipment
- Used decontamination solutions
- Sample residuals

Disposable equipment (e.g., plastic gloves, Tyvek suits, broken containers) contaminated with site sediments or water were put in sealed plastic bags and placed in a 55-gallon (DOT 17H open-top) drum designated for disposable equipment. Decontamination solutions were captured and placed in a 55-gallon (DOT 17H open-top) drum designated for decontamination fluids. The drums were sealed at the end of sampling and labeled in accordance with applicable regulations. The drums are currently stored in the process plant area.

Sample residuals generated at the site during field analysis using X-ray fluorescence spectroscopy were placed back on site after analysis. Sample residuals generated at the EPA Testing and Evaluation (T&E) facility after the treatability studies were conducted will be disposed of at an approved and permitted off site facility.

3.5 Sample Preparation Procedures

Surface soil/sediment samples collected for the low-temperature thermal desorption, solidification/stabilization, and xanthate studies were placed unmixed in a 30-gallon steel open-head drum and shipped to the EPA T&E facility in Cincinnati, Ohio, in accordance with DOT shipping and hazardous waste manifest codes. These samples were unloaded at the T&E facility and placed in an insulated drum overpack with dry ice for cooling in the drum storage area of the facility.

The treatability study samples were extracted from the open-head drum and homogenized prior to waste characterization analysis and treatability testing. An aluminum scoop was used to place samples in stainless steel pans under the laboratory hood for homogenization. Large fragments and debris were removed by hand from the pans during mixing and placed back in the steel drum. The soils were mixed until they appeared to be of uniform color and texture. The mixing time did not exceed 15 minutes so as to minimize volatile losses during this step. Stainless steel spoons were used to transfer the soils from the pans to the testing apparatus or the appropriate sample containers for analysis.

The sediment and ground water samples collected for the biological studies were packed in a sample cooler, and shipped to IT Corporation's Biotechology Center in Knoxville, Tennessee in accordance with procedures outlined in PEI's revised QAPjP (April, 1990). These samples were received by IT laboratory personnel on April 13, 1990 and stored at 4°C.

Prior to biological testing, ground water was with drawn from the container using a sterilized, pre-cleaned pipette. Soil samples were withdrawn with a stainless steel scoop and manually mixed and pulverized with a mortar and pestle. The pulverized soil was then screened to remove any large, gravel-sized particles.

SECTION 4

BIOLOGICAL STUDIES

In this treatability study, microbial activity and environmental conditions that favor biodegradation of carbon disulfide and thiocyanate compounds were evaluated to assess the applicability of biological treatment of soils and water. This section presents the overall approach in Subsection 4.1 and the results of the test in Subsection 4.2.

4.1 Treatability Study Approach

Figure 4-1 illustrates the treatability study approach. The biological studies involved an initial evaluation of the existing microbial population in soil and ground water, followed by a series of treatments evaluating the effects of oxygen and nutrients on the indigenous population. Microbial growth and oxygen consumption were measured to evaluate the potential for biological treatment of carbon disulfide and ammonium thiocyanate in soils and ground water.

The following subsections describe the test objectives and rationale (Subsection 4.1.1), experimental design and procedures (Subsection 4.1.2), equipment and materials (Subsection 4.1.3), laboratory sampling and analysis performed (Subsection 4.1.4), management of data collected (Subsection 4.1.5), and deviations from the QAPjP (Subsection 4.1.6).

4.1.1 Test Objectives and Rationale

The immediate test objective of the biological studies was to determine the absence or presence of sulfide- and thiocyanate-specific degraders in sufficient numbers to justify a full treatability assessment. The overall aim was to evaluate the potential of

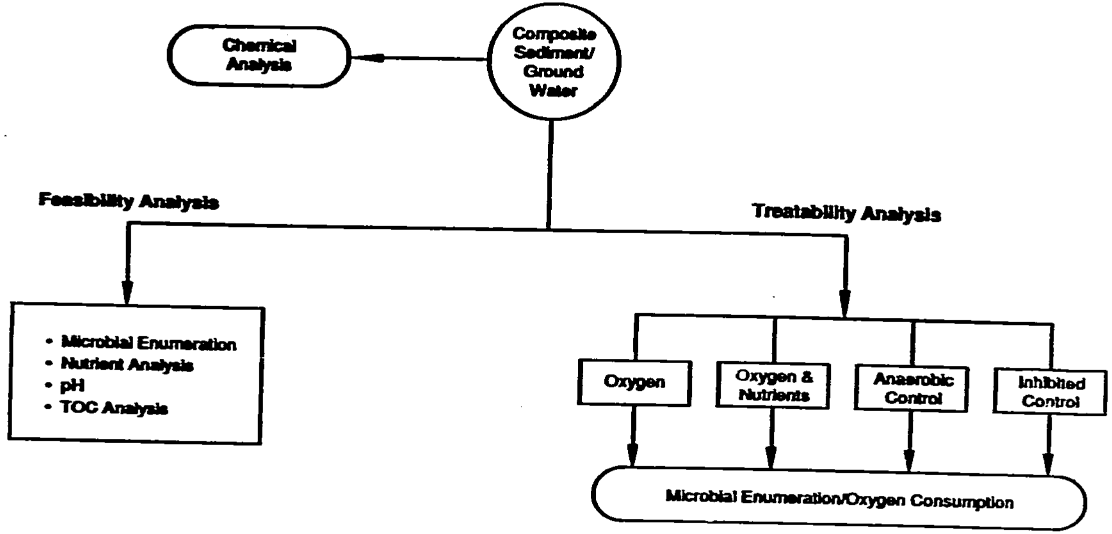


Figure 4-1. Flow diagram of biodegradation.

the site for supporting a remediation program based on the principles of microbial degradation. The purpose of the study was not to determine guidelines to implement a bioremediation program on site, but rather to determine the presence or absence of specific carbon disulfide- and thiocyanate-degrading microorganisms.

Site remediation by microbial degradation is highly site-specific, and the technology requires that important factors affecting bioremediation (e.g., contaminant-specific degraders in sufficient numbers, low concentrations of compounds highly toxic to the microbes) be identified before a complete treatability assessment can be justified.

4.1.2 Experimental Design and Procedures

Prior to testing, all samples were stored at 4°C. Water samples were taken by pipette. Soil sediment samples were prepared by mixing manually, air drying the sample in a chemical hood, pulverizing the sample with a mortar and pestle, and using a sieve to remove large gravel particles. Finally, the samples were analyzed for nitrogen (as ammonia), phosphate (as orthophosphate), and pH. Preparation of sediment samples for all other analyses was conducted in an identical manner, with the exclusion of the air-dry step. Sample quantities of sediment and ground water in the mini-treatability segments were in the ratio of 20 ml water to 2 g wet sediment (10 parts volume to 1 part weight).

Assessment Study

The assessment study consisted of the following tests:

1. Microbial enumeration
2. Nutrient analysis
3. pH
4. Total organic carbon (TOC) analysis

The test methods are included in Appendix B-1 and are briefly discussed below.

Microbial enumerations were performed according to Plate Count Method 9215 C [Standard Methods for the Examination of Water and Wastewater (17th ed.)

1989] with minor modifications for the carbon disulfide and thiocyanate degraders. Heterotrophic bacteria were enumerated under both anaerobic and aerobic conditions using dilute nutrient agar. Thiocyanate degraders were enumerated by adding potassium thiocyanate to the nutrient agar for a final solution concentration of 0.1 percent. Carbon disulfide degraders were enumerated by adding mineral salts to the nutrient agar, mixing 1 ml carbon disulfide with 9 ml deionized water in a small beaker, and placing the nutrient agar plates plus the beaker into a sealed vessel containing a carbon disulfide atmosphere. Temperature was maintained at 20°C and the experiments were run for 7 to 14 days.

The nutrient analysis consisted of an evaluation of the nitrogen and phosphate content of groundwater and lagoon sediment. Nitrogen (as ammonia) was analyzed according to Nesslerization Method 4500-NH3 C (Standard Methods 1989). Phosphate content (as orthophosphate) was determined according to Ascorbic Acid Method 4500-P E (Standard Methods 1989).

The pH of all samples was measured using a Corning pH Meter Model 150 with an Orion combination electrode. All ground-water samples were analyzed for TOC with a Dohrmann DC-80 in accordance with Method 5310 C (Standard Methods 1989).

Mini-treatability Study--

The experimental design for the mini-treatability study included four different treatments of a composite made up of 20 ml water to 2 g wet sediment (10 parts volume to 1 part weight). A total of 1.5 kg of sediment and 4 liters of ground water were obtained from the site. It was not necessary to add sulfide or thiocyanate compounds to the composite treatments because the concentrations of these contaminants in the samples taken from the Halby site were already sufficiently high to conduct the study. To determine whether native bacteria were present and metabolically active in the sediment and ground water, the following four treatments were conducted:

1) Treatment 1 (aerobic, nutrient amended)--1000 ppm Restore 375™ brand microbial nutrient was added to the vessel. The head space, which constitutes 50 percent of the total volume, was filled with air.

2) Treatment 2 (aerobic, not nutrient amended)--No nutrients were added; the head space was filled with air.

3) Treatment 3 (anaerobic, nutrient amended)--1000 ppm nutrients was added; the head space was purged with helium.

4) Treatment 4 (killed control)--100 ppm mercuric chloride was added to inhibit all biological activity. The head space consisted of air.

The vessels were sealed with Teflon™-lined silicon septa. The concentration of oxygen was measured at frequent intervals over a 2-week period. Oxygen gas samples were collected by taking 50 µl of head space with a gas-tight syringe. The oxygen content of the gas samples was determined by injecting the gas sample into a quantitative oxygen sensor. The four treatments were monitored for contaminant degradation over a period of 2 weeks, as indicated by the following parameters:

1) Microbial growth
2) Oxygen consumption (measure of microbial metabolic activity)

Microbial density was measured at the beginning and end of the study. Microbial density was evaluated using a Quebec Colony Counter with a 2-power magnifying lens and dark field light source. Oxygen consumption was monitored throughout the 2-week period by withdrawing a sample of gas from vessel headspace using a gas-tight syringe, and injecting it into a proprietary quantitative oxygen sensor instrument described in Graves and Greenbaum (1989). TOC was not measured in the mini-treatability study, because the preparation steps (i.e., soil filtering for injection of filtrate into analyzer, and soil purging with oxygen) would have driven off significant amounts of low-molecular-weight organics and invalidate the results. Complete references for the analytical methods used for the assessment and mini-treatability studies are provided in Tables 4-1 and 4-2.

4.1.3 Equipment and Materials

Testing equipment consisted of the standard microbiological materials such as flasks, test tubes, agar plates, anaerobic jars, and microscopes. Liquid samples were

ORIGINAL
(Red)

TABLE 4-1. ANALYTICAL PARAMETERS FOR CHARACTERIZATION OF GROUNDWATER AND SEDIMENT (ASSESSMENT STUDY)

Sample matrix	Sample quantity	Analytical parameter	Analytical method[a]	Number of analyses[b]
Ground-water	1 ml	Microbial density	Plate Count Method 9215 C	24[b]
	10 ml	Phosphate	Ascorbic Acid 4500-P E	4
	10 ml	Nitrogen-ammonia	Nesslerization 4500-NH3 C	4
	2 ml	TOC	Combustion-Infrared Method 5310 C	4
	10 ml	pH	Corning pH Meter Model 150 with Orion combination electrode	4
Sediment	5 g	Microbial density	Plate Count Method 9215 C	6[c]
	5 g	Phosphate	Ascorbic Acid 4500-P E	1
	5 g	Nitrogen-ammonia	Nesslerization 4500-NH3 C	1
	5 g	pH	Corning pH Meter Model 150 with Orion combination electrode	1

[a] All methods are taken from Standard Methods for the Examination of Water and Wastewater, 17th ed., 1989.

[b] Twelve tests were conducted under aerobic conditions and twelve under anaerobic conditions. Within each group of twelve, four tests each were conducted for heterotrophs, thiocyanate degraders, and carbon disul[illegible]graders.

[c] Three [illegible] were conducted under aerobic conditions and three under anaerobic conditions, with one each for heterotrophs, thiocyanate degraders, and carbon disulfide degraders.

ORIGINAL
(Red)

TABLE 4-2. ANALYTICAL PARAMETERS USED TO EVALUATE MICROBIAL GROWTH ON SITE CONTAMINANTS (MINI-TREATABILITY STUDY)

Sample matrix	Sample quantity	Analytical parameter	Analytical method[a]	Number of analyses
Initial	20 ml groundwater in 2 g sediment	Microbial density	Plate Count Method 9215 C	1
Treatment No. 1 (aerobic, nutrient-amended)	20 ml groundwater in 2 g sediment	Microbial density	Plate Count Method 9215 C	3
		Oxygen consumption	Graves and Greenbaum (1989), Plant Physiology, 90:246-250	7
Treatment No. 2 (aerobic, not nutrient-amended)	20 ml groundwater in 2 g sediment	Microbial density	Plate Count Method 9215 C	3
		Oxygen consumption	Graves and Greenbaum (1989), Plant Physiology, 90:246-250	7
Treatment No. 3 (anaerobic, nutrient-amended)	20 ml groundwater in 2 g sediment	Microbial density	Plate Count Method 9215 C	3
		Oxygen consumption	Graves and Greenbaum (1989), Plant Physiology, 90:246-250	7
Treatment No. 4 (anaerobic, not nutrient-amended)	20 ml groundwater in 2 g sediment	Microbial density	Plate Count Method 9215 C	3
		Oxygen consumption	Graves and Greenbaum (1989), Plant Physiology, 90:246-250	7

[a] Microbial density and oxygen consumption methods are taken from Standard Methods for the Examination of Water and Wastewater, 17th ed., 1989.

obtained using sterile glass and serological pipettes. Sediment samples were transferred using stainless steel spatulas. In the mini-treatability study, oxygen consumption was measured by withdrawing a sample of gas from vessel headspace using a gas-tight syringe, and injecting it into a quantitative oxygen sensor.

4.1.4 Sampling and Analysis

Table 4-1 provides information on the analytical parameters used to characterize the sediment and groundwater. Table 4-2 provides information on the treatment process sampling and analysis.

4.1.5 Data Management

The data management sheets for the biological studies are provided in Appendix A-1. For the assessment study, analyses of nitrogen (as ammonia), phosphate (as orthophosphate), TOC, and pH were conducted once for the sediment sample and once each for the four groundwater samples. Enumerations of aerobic and anaerobic microorganisms were conducted on the sediment and each of the four groundwater samples for heterotrophs, thiocyanate degraders, and carbon disulfide degraders, for a total of 30 analyses.

For the mini-treatability study, an initial enumeration was performed, and final enumerations of heterotrophs, thiocyanate degraders, and carbon disulfide degraders) were conducted on each of four treatments, for a total of 13 analyses. Oxygen consumption was monitored regularly through the 2-week period.

4.1.6 Deviations From the QAPjP

Treatment 3, containing the helium atmosphere, failed to maintain an oxygen-free atmosphere for the duration of the 14-day test period. Silicon and Teflon are highly permeable to oxygen, so the failure may have been caused by the diffusion of atmospheric gases through the silicon septum. It may also have been caused by the diffusion of oxygen from the liquid phase, or by a leak resulting from sampling through the septum. Strictly anaerobic conditions were therefore not maintained for this treatment run.

ORIGINAL
(Red)

4.2 Results and Discussion

The following discussion presents the results of the assessment and mini-treatability studies, including data analysis and interpretation, quality assurance/quality control, costs/schedule for performing the studies, and key contacts.

4.2.1 Data Analysis and Interpretation

The results of the study indicated that bacteria capable of degrading thiocyanate and carbon disulfide are present in the sediment and groundwater. Microbial growth of heterotrophs, carbon disulfide and this cyanate degraders, and respiration were also demonstrated under aerobic, nutrient-amended conditions. Only heterotrophs and carbon disulfide degrader populations grew under anaerobic conditions. Tables 4-3 through 4-6 present results from the assessment analyses. Table 4-7 and Figure 4-2 gives results from the four treatment runs. Table 4-7 provides the microbial population counts for heterotrophs, thiocyanate degraders, and carbon disulfide degraders in each of the four treatments. Figure 4-2 illustrates the cumulative consumption of oxygen in Treatments 1, 2, and 4. While oxygen levels in Treatment 3 (anaerobic test runs) were monitored, the results are not shown on Figure 4-2, because oxygen consumption is not a valid parameter for measuring metabolism of anaerobes. The variability of data points is a reflection of the variability associated with the injection and sample collection techniques.

Although the microbial density in ground water was low, indigenous microbes were shown to grow in response to the addition of nutrients and oxygen. Further investigations are necessary to confirm the initial results, to determine the feasibility of implementing bioremediation at the site, and to select the treatment scenario that will be best suited to the particular set of conditions at the site. The presence of specific compound-degrading bacteria suggests the possibility of treating site water and sediment in an above-ground bioreactor treatment system.

TABLE 4-3. INORGANIC NUTRIENT CONCENTRATION AND pH OF SITE SAMPLES

Sample	Orthophosphate, ppm	Ammonia, ppm	pH
Sediment	190	49	5.9
Water-1	<DL	201	6.5
Water-2	<DL	196	6.3
Water-3	<DL	191	6.3
Water-4	<DL	211	6.3

TABLE 4-4. TOTAL ORGANIC CARBON CONTENT OF SITE GROUND-WATER SAMPLES

Sample	Total carbon, µg/mL	Total inorganic carbon, µg/mL	Total organic carbon, µg/mL
Water-1	436	184	252
Water-2	465	210	255
Water-3	471	213	257
Water-4	468	210	260

TABLE 4-5. AEROBIC MICROBIAL ENUMERATION[a]

Sample	Heterotrophs	Thiocyanate degraders	CS_2 degraders
Sediment	290,000	28,000	26,000
Water-1	180	Not detected	40
Water-2	1,000	Not detected	60
Water-3	70	Not detected	80
Water-4	130	Not detected	170

[a] Data recorded as colony-forming units per milliliter of ground water or gram dry weight sediment.

ORIGINAL
(Red)

TABLE 4-6. ANAEROBIC MICROBIAL ENUMERATIONS[a]

Sample	Heterotrophs	Thiocyanate degraders	CS_2 degraders
Sediment	40,000	Not detected	75,000
Water-1	140	Not detected	Not detected
Water-2	1,300	Not detected	120
Water-3	430	Not detected	220
Water-4	290	Not detected	120

[a] Data recorded as colony-forming units per milliliter groundwater or gram dry weight sediment.

TABLE 4-7. MICROBIAL ENUMERATIONS FOR THE INVESTIGATION OF BIOLOGICAL *ACTIVITY IN SEDIMENT AND GROUND-WATER SLURRIES*

Sample	Heterotrophs	Thiocyanate degraders	CS_2 degraders
Initial (feasibility)	4.4×10^5	4.2×10^4	4×10^4
Nutrients + O_2 (Treatment 1)	1.8×10^8	1.6×10^6	2.5×10^7
O_2 (Treatment 2)	1.7×10^8	3.1×10^3	7.5×10^6
Anaerobic (Treatment 3)	5.5×10^7	1×10^3	5.2×10^6
Biological-inhibited (Treatment 4)	9.5×10^7	2×10^4	8.5×10^6

[a] Data are presented as colony-forming units per milliliter of slurry; treatments are described in the text.

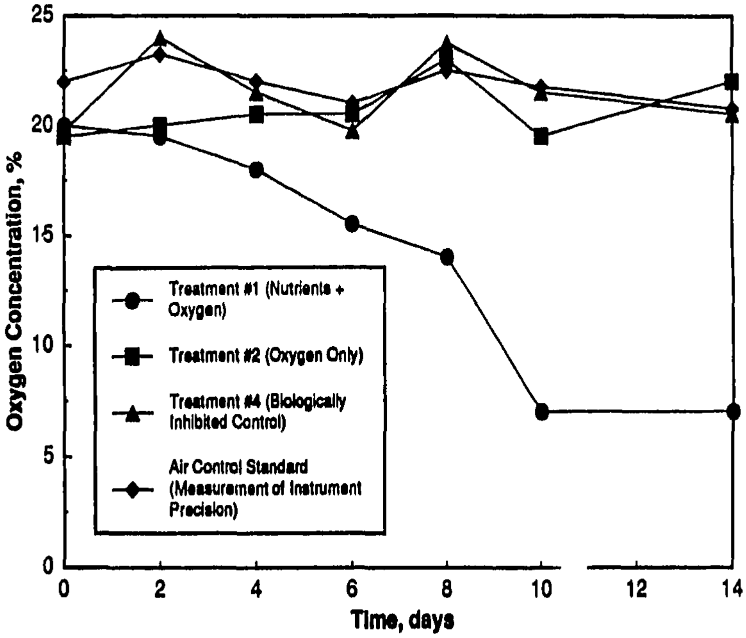


Figure 4-2. Oxygen consumption data by water-sediment slurries. Air control is a standard obtained by quantifying the oxygen content in air at each sampling point.

The following interpretations were drawn from the results of the preliminary assessment (see Table 4-3 through 4-6):

1) The pH of the site ground water was within the range generally considered suitable for bioremediation. The pH of the sediment was low but not seriously acidic.

2) Ground water was deficient in phosphate.

3) Aerobic organisms were detected in the sediment sample. The microbial population was very low in ground water samples. Thiocyanate and carbon disulfide degraders comprised approximately 10 percent of the total microbial population found in the sediment sample. Low levels of carbon disulfide degraders were found in ground water.

4) Anaerobic microbes were detected in sediment and ground water. A high concentration of carbon disulfide degraders was observed in the sediment sample. No anaerobic thiocyanate degraders were observed.

5) Metabolic activity and microbial growth were observed on the organic carbon contained in the site samples.

The data provided in Table 4-7 show an increase in microbial population size following nutrient and oxygen enrichment (Treatment 1). The heterotrophic population density increased with each treatment. Treatment 3 (anaerobic, nutrient amended) is not a valid assessment of the increase in anaerobes because of the leakage of oxygen into the treatment vessel; however, the presence of oxygen probably inhibited the growth of anaerobic microorganisms in this treatment. Microbes from this treatment were enumerated under aerobic conditions.

The increased microbial density in the biologically inhibited vessel (Treatment 4) is probably a result of the presence of mercury-resistant organisms. This phenomenon has been observed in other studies. The lack of growth of thiocyanate degraders in this treatment indicates that this subpopulation of degrading microorganisms was unable to grow in the presence of mercury. Oxygen and nutrient deficiency also affected the growth of thiocyanate degraders (Treatment 2). The results of these experiments indicate that nutrient addition stimulates the growth and metabolism of degrading microbes (Treatments 1 and 3). Based on the microbial enumerations presented in Tables 4-5 (aerobic) and 4-6 (anaerobic), oxygen and nutrients were required to support the growth of thiocyanate degraders, which appear to be obligate aerobes (obligate aerobes have metabolic pathways that require oxygen to function).

The objective of this study was to determine whether sufficient numbers of contaminant-specific microorganisms exist on site to justify additional studies. The of the analyses indicate that suitable numbers of sulfide and thiocyanate-degrading microorganisms are present in soils to justify conducting a detailed treatability assessment.

4.2.2 Quality Assurance/Quality Control

One biologically inhibited control was run for the mini-treatability study. Microbial densities for the sterilized control indicated the presence of mercury-resistant strains of microorganisms. Duplicate treatment runs were not performed at this stage.

4.2.3 Costs/Schedule for Performing Treatability Study

Samples were taken at the Halby site on April 10, 1990, and received at the IT Biological Center (located in Knoxville, Tennessee) on April 12, 1990. Analyses were completed and a preliminary report submitted on May 17, 1990. Total costs for performing the assessment and mini-treatability studies were $7300.

4.2.4 Key Contacts

The following key personnel may be contacted for additional information concerning the biological studies:

Dr. Duane Graves
IT Corporation
Biotechnology Center
312 Directors Drive
Knoxville, TN 37923
(615) 690-3211

Ms. Roxanne B. Sukol
PEI Associates, Inc.
15 Elmwood Place
Athens, OH 45701
(614) 592-2580

SECTION 5

XANTHATE FLOTATION

In this laboratory screening study, xanthate flotation was investigated as a chemical treatment for metals in soils. This section presents a detailed discussion of the approach (Subsection 5.1) and results (Subsection 5.2) of the xanthate flotation/ separation study.

5.1 Treatability Study Approach

Figure 5-1 illustrates the treatability study approach. Deionized water containing an alkyl xanthate salt was added to homogenized soil, along with a frothing agent. The mixture was stirred and air was bubbled through the solution to facilitate flotation and separation of the insoluble metal sulfide particles from the soils. The froth was then skimmed from the surface and collected in a flask.

The following subsections describe the test objectives (Subsection 5.1.1), experimental design and procedures (Subsection 5.1.2), equipment and materials (Subsection 5.1.3), and sampling and analysis (Subsection 5.1.4).

5.1.1 Test Objectives and Rationale

No reference to this procedure being investigated as a treatment for hazardous waste was identified in the literature. Consequently, the only test objective set for this study was to demonstrate a reduction in the concentrations of metals in the soil/sediment. Any reduction would signify that the process does separate metal contaminants from the soil and that further bench-scale investigations would be appropriate.

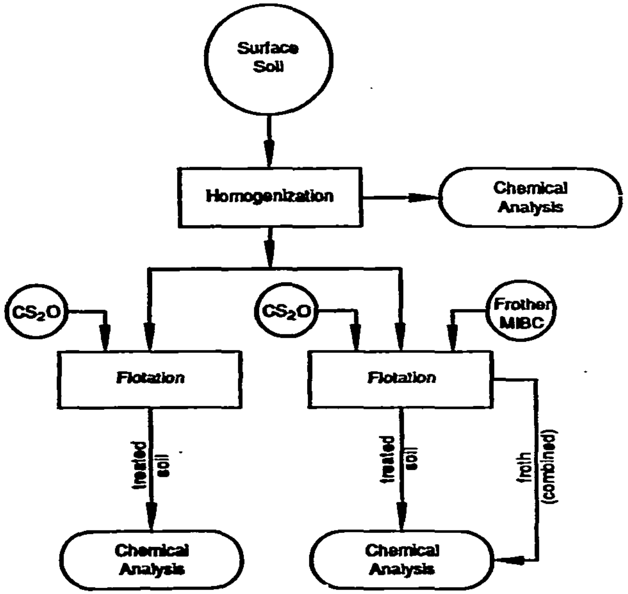


Figure 5-1. Flow diagram of xanthate flotation.

5.1.2 Experimental Design and Procedures

The design of this experiment was very simple as illustrated in Figure 5-2. Untreated soil was mixed with deionized water and potassium amyl xanthate in a 4-liter heavy-duty glass beaker. A frothing agent, 2-ethylhexanol, was also added to the beaker. The mixture was stirred and air was bubbled through to facilitate flotation of the insoluble metal sulfides present. The froth was then skimmed from the surface and collected. Figure 5-2 presents a diagram of the flotation cell. The complete, detailed experimental procedure is presented in Appendix B-2.

5.1.3 Equipment and Materials

The equipment used in this study included a mechanical stirrer, a variable-volume air pump with Teflon tubing and a glass bead bubbler, and a 4-liter flask to serve as the flotation cell.

The potassium amyl xanthate was provided by American Cyanamid Company as their product AERO 350. It is supplied in pellet form. The frother, 2-ethylhexanol, was also provided by American Cyanamid as AEROFROTH 88.

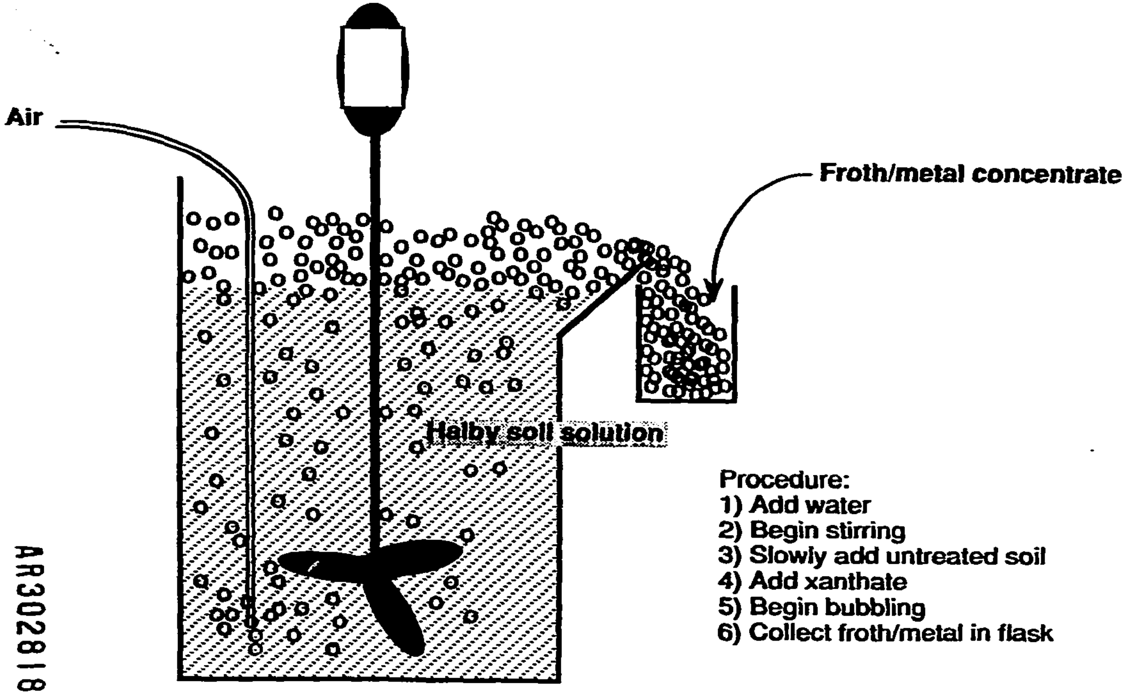
5.1.4 Sampling and Analysis

Two eight-ounce samples of the raw soil/sediment were taken for analysis of metal indicator compounds prior to treatment. After treatment, one eight-ounce sample of treated soil/sediment was collected for each of the two runs. In addition, one eight-ounce sample of froth was collected from the second run.

The samples were stored at 4°C and sent to the laboratory for analyses of metal indicator compounds.

5.1.5 Data Management

Complete notes were taken during the laboratory study and are presented in Appendix A-2.



Procedure:
1) Add water
2) Begin stirring
3) Slowly add untreated soil
4) Add xanthate
5) Begin bubbling
6) Collect froth/metal in flask

Figure 5-2. Xanthate flotation.

5.1.6 Deviations From the QAPjP

The QAPjP for this study presented the standard operating procedure (SOP) for xanthate flotation. Deviations from this SOP are summarized in Table 5-1.

TABLE 5-1. DEVIATIONS FROM SOP

	SOP		Actual	
Variable/parameter	Run #1	Run #2	Run #1	Run #2
Ratio of deionized water added to soil	2:1	2:1	3.5:1	3.5:1
Amount of xanthate added	1 pellet	1 pellet	5 pellets (.45 g)	8 pellets (.72 g)
Amount of frother added	None	1 drop	16 drops (~1 ml)	16 drops (~1 ml)
Duration of treatment	15 min	15 min	40 min	31 min

The quantities of reagents were increased due to the lack of froth appearing on the surface. The xanthate dose rate supplied by the manufacturer is 25 to 250 g/metric ton ore. To treat 500 g of soil, no more than 0.125 g (1 typical pellet) of xanthate should have been required. Up to 0.72 g was added with no appreciable increase in frothing. A large excess of frother was used; however, this too was not helpful. Attempts to mechanically increase the production of froth--increased stirring rates and durations--were also unsuccessful.

5.2 Results and Discussions

The following discussion presents the results and data interpretation (Subsection 5.2.1) and costs/schedule for performing treatability study (Section 5.2.2) as well as key contacts (Section 5.2.3). Because these were preliminary studies, no quality assurance/quality control treatment runs or analyses were performed, and therefore, no discussion is presented for these studies.

5.2.1 Data Analysis and Interpretation

Although several attempts were made to encourage long-lasting frothing action, no significant froth was observed, and therefore, no apparent separation of metal particles from the soil. Consequently, no chemical analysis was performed on the treated and untreated soils.

The negative results from the study may have occurred as a result of one or several of the following factors:

- Inadequate equipment for mixing and bubbling action;
- Too great a liquid to solids mix ratio;
- Presence of too little (or no) metal sulfide particles in the soil for flotation;
- Too small particle size or too little xanthate reagent to effect reaction between xanthate and metal particle.

Further jar studies may be necessary to determine whether the flotation process did not work as a result of the equipment and experimental design or because of a lack of chemical reaction between the xanthates and metals in soils.

5.2.2 Costs/Schedules for Performing Studies

The costs for performing the xanthate studies were $1500, including laboratory testing, equipment and materials, and report preparation. The study was performed in one week.

5.2.3 Key Contacts

The following people were involved in the testing and analysis of xanthate flotation:

Greg McNelly
PEI Associates, Inc.
11499 Chester Road
Cincinnati, OH 45246

SECTION 6

LOW-TEMPERATURE THERMAL DESORPTION STUDIES

In this treatability study, low-temperature thermal desorption (LTTD) was evaluated as a pretreatment process that removes those volatile contaminants that may interfere with the stabilization/solidification process. This section presents a detailed discussion of the approach (Subsection 6.1) and results (Subsection 6.2) of the low-temperature thermal desorption studies.

6.1 Treatability Study Approach

Figure 6-1 illustrates the treatability study approach. The LTTD studies were performed at two temperatures (300° and 500°F) and two residence times (15 and 30 minutes). These test runs were performed in duplicate. The VOC, semivolatile, and metal indicator compounds were measured in both the soils and the solid residues from all test runs. In addition, the condensate collected from the 500°F/30-minute test runs was also analyzed for VOC, semivolatile, and metal indicator compounds.

The following information describes the objectives of tests performed, experimental design and procedures, analyses performed, and management of data collected during the low-temperature thermal-desorption process of the soil samples.

6.1.1 Test Objectives and Rationale

The test objective for the LTTD studies was to reduce concentrations of some of the chemical compounds (i.e., carbon disulfide, ammonia, thiocyanate, present in site soil that had the potential to interfere with the cement or asphalt binders used in the solidification/stabilization studies. In addition, reduction of carcinogenic semivolatile organic compounds to 8 mg/kg in soils was also a treatment objective for

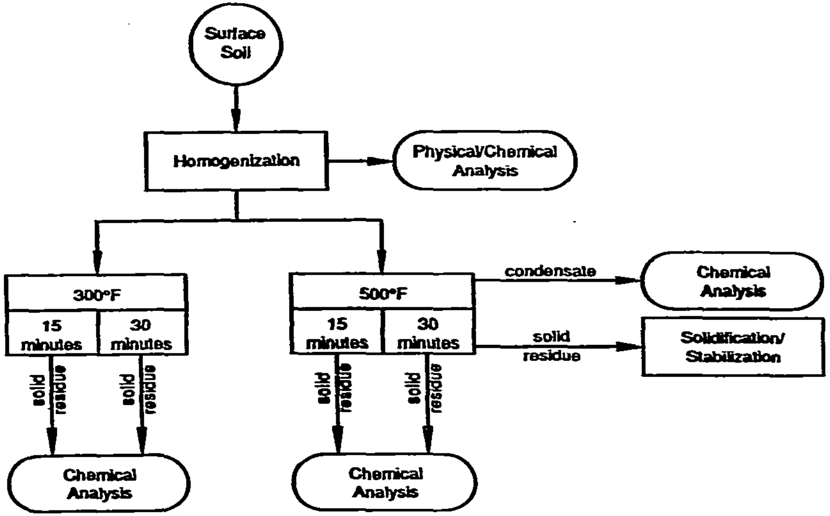


Figure 6-1. Flow diagram of low-temperature thermal desorption.

LTTD studies.

6.1.2 Experimental Design and Procedures

Detailed discussion of experimental procedures is given in Appendix B-3. The thermal treatment of the soil samples was performed by placing an approximately 800-g aliquot of homogenized soil inside a 4-liter glass reaction flask and spreading the sample in a uniform layer on the bottom of the flask at ambient temperature. The sample was then heated gradually until it reached its highest temperature (300° or 500°F). The soil was heated at that temperature for 15 or 30 minutes (reaction times). During each test run, the reactor vessel was continuously purged with nitrogen gas to reduce the possibility of fire or explosion that might occur during the heating of soil samples. The condensate generated during the process was collected in a 1-liter volumetric flask. When the prescribed residence time at the target temperature was reached, heating was terminated and the soil was allowed to cool to ambient temperature. Next, the vessel lid was removed and the soil residue was transferred into a preweighed, clean aluminum tray. All the experiments were conducted in duplicate runs to collect adequate data on test performance. A total of eight samples (2 temperatures x 2 residence times x 2 replicates) were collected during the tests. To prepare samples for the subsequent solidification/stabilization studies, addi[illegible]ns at 500°F and a 30-minute residence time were performed in order to generate sufficient quantity of soil material.

6.1.3 Equipment and Materials

The experimental apparatus used in this study is shown in Figure 6-2. Materials and reagents used are described in Appendix B-3. The apparatus consists of a 4-liter glass reaction flask mounted within a temperature-controlled heating mantle. The reaction flask is fitted with a removable glass cover, which is attached to the flask by a ground-glass joint and a Teflon™ gasket and is sealed with a metal clamp. The glass cover has four ground-glass-joint openings through which equipment is inserted. A

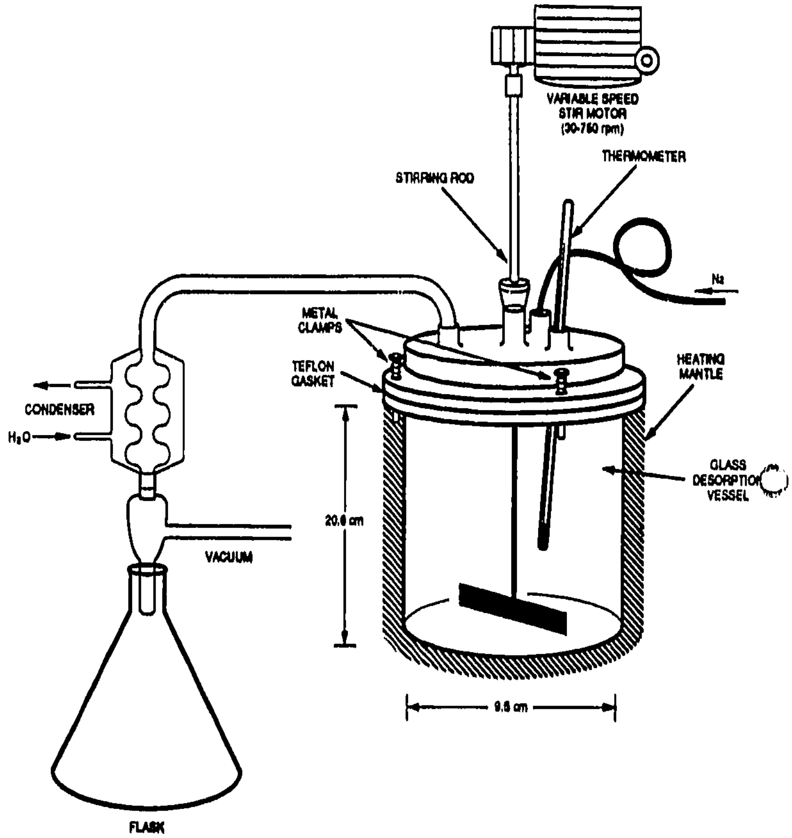


Figure 6-2. Diagram of desorption vessel.

ORIGINAL (Red)

motor-driven, Teflon™-coated, stainless steel stirring shaft is inserted through an air-tight bearing into the center opening of the vessel top. This stirrer is operated at 30 to 750 rpm during the reaction. A thermometer is inserted through another ground-glass joint in the reaction flask cover to monitor the temperature of the soil. One of the ground-glass joints in the top of the glass cover is fitted with a water-cooled condenser. A ground-glass adapter attached to Tygon™ tubing is inserted through the opening onto the top of the condenser. This allows the headspace of the reaction flask to be purged during the reaction to reduce the possibility of explosion of any unstable organic products that might evolve from the reaction mixture.

6.1.4 Sampling and Analysis

To evaluate the soil characteristics that may influence remedial processes and to determine the efficacy of LTTD in the treatment of the Halby Chemicals site soils, samples of the raw soil (surface/sediment), soil residues, and condensates collected during the low-temperature tests were subjected to chemical analysis and physical tests specified in Table 6-1. A total of 10 samples (two raw soils and eight thermally-treated residues) were analyzed for VOCs, semivolatiles, metals total cyanides, and ammonia. All 10 samples were tested to determine their water content. The raw soil samples were further tested for particle size analysis to provide adequate data on material handling and processing characteristics. The test is usually performed prior to stabilization/solidification to suggest the feasibility of the process (i.e., clay soils reduce the viability of the stabilization/solidification process) or the difficulties that could be encountered in processing.

6.1.5 Data Management

Appendix A-3 presents data summary sheets for LTTD test runs. When the soil sample was heated inside the reaction flask, a stirrer was used to homogenize and mix the sample at a speed of approximately 115 rpm. The temperature of the soil was monitored every 15 minutes until it reached the target temperature. About 2.5 to 4

TABLE 6-1. ANALYTICAL TESTING OF RAW SOILS, SOIL RESIDUES, AND CONDENSATE FROM LTTD EXPERIMENTS(a)

Parameter	Test Method(b)	Untreated Soil	LTTD Soil Residue	Condensate
VOCs(c)	8240(s)624(l)	2	8	1
Semivolatiles(d)	3550/8270(s)625(l)	2	2	1
Metals(e)	3050/6010(f)	2	8	1
Cyanide	9012	2	8	-
Ammonia	350.2(g)	2	8	-
Other Parameters				
Moisture Content	ASTM D3173	2	8	-
Particle Size	Sieve Analysis	2	-	-

[a] Values shown are number of analyses for a given matrix and analytical parameter.

[b] Test methods listed are those from EPA SW846, unless otherwise specified. Test method number followed by "(S)" is the method use for soil; the number followed by "(l)" is the method used for the condensate sample.

[c] VOCs include: Carbon disulfide; Toluene; Methylene Chloride; Tetrachloroethene; and 2-Butanone.

[d] Semivolatiles include: Chrysene; Pyrene; Fluoranthene; Benzo(b) fluoranthene; Benzo(k) fluoranthene; and Benzo(a) pyrene.

[e] Metals include: Arsenic; Copper; Zinc; Cobalt; and Chromium.

[f] Arsenic was analyzed by EPA Method 7060 (ICP).

[g] Soils were extracted with water and then analyzed for ammonia using EPA Method 350.2. (Standard Methods for the Examination of Wat and Wastewater, 17th Ed., 1989)

hours was required for the 800-g aliquots of soil to reach 300° or 500°F, respectively. Approaching the target temperatures, soil temperature was recorded at every 5-minute interval.

Because most of the water was evaporated from the mixture at temperatures around 230°F, the soil hardened and part of the sample was attached to the wall of the reaction vessel. This problem was corrected, however, by changing the position of the stirrer and folding in the soil back to the bottom of the reaction flask.

6.1.6 Deviations From the QAPjP

Changes to the experimental design and testing from that described in the QAPjP were made as a result of new information on human health and environmental risks. Table 6-2 presents the changes that were made to the LTTD studies.

TABLE 6-2. DEVIATIONS FROM QAPjP IN LTTD TESTING AND ANALYSIS OF SOIL SAMPLES

No.	Parameter	Proposed in QAPjP	Actual testing condition	Rationale
1	Weight of raw soil samples in LTTD process	500 g	800 g	To more effectively mix the soil inside the reaction flask and decrease the total number of LTTD test runs required for collection of solidification/stabilization samples.
2	Target temperatures for heating of soil samples	100°, 300°, and 500°F were determined as target temperatures for LTTD treatment of soils (total of 12 samples were proposed to be treated)	300° and 500°F temperatures were used to treat soil samples (total No. of soil samples was reduced from 12 to 8)	Since most volatile metals and organics are removed from soils at temperatures above 100°F, this temperature was of least importance in LTTD testing. Due to project budgetary limitations and time constraints, this target temperature was removed from testing program.
3	Indicator compounds for chemical tests	A list of indicator compounds is presented in Table 1-7 of QAPjP	Some of indicator compounds were being replaced with new compounds (see Table 6-1)	The list of target compounds was changed because higher risk potential that some of the contaminants pose to human health and safety.
4	TOC	Raw soil samples were supposed to be analyzed for TOC	TOC analyses were not performed	The analyses were not appropriate in LTTD testing of soils.

[a] NP = Not proposed in testing program.

6.2 Results and Discussions

The following discussion presents the analytical results and data interpretation for the low-temperature thermal desorption, including discussion of quality assurance/ quality control test runs and analyses, costs/schedule for completion of the studies, and key contacts.

6.2.1 Data Analysis and Interpretation

Table 6-3 and Figures 6-3 through 6-5 give a comparison of contaminant concentrations in untreated and thermally-treated soils. Together, these table and figures illustrate several findings:

- The concentration of most volatile and semivolatile organic compounds and total cyanides decreased significantly after thermal treatment at 500°F for 30 minutes.
- The concentration of metals and inorganic compounds remained fairly constant (concentrations varied within normal range of variability expected for analyses) in the soils throughout the thermal studies.

The apparent increase of certain VOCs (i.e., methylene chloride, 2-butanone, carbon disulfide) after thermal treatment at 300°F may be the result of high moisture content of the raw soils, which reduced the analytical recovery and quantitation of these soluble compounds. The low percent recoveries (30 to 40%) of these VOCs in the matrix spike samples of the raw soil gives support to this theory. Other possible explanations for the apparent increase in these VOC concentrations may be degradation of other compounds during thermal treatment.

In addition to analyses for soils, condensate collected in the flask from the 500°F/30 minute test runs was composited and analyzed for the organic and inorganic indicator compounds. The analytical data shown in Table 6-4 indicated that semivolatiles and metals are present at or below their solubility limits and that most VOCs are not present above detectable levels. The absence of VOCs in the collected condensate may have been due to the fact that non-condensable vapors and some

TABLE 6-3. ANALYTICAL RESULTS FOR UNTREATED (RAW) AND LTTD TREATED SOIL SAMPLES

	Metals and inorganics, µg/g							Volatile organics, µg/kg					Semivolatiles, µg/kg						Percent mois-ture
	NH_3	As	Cr	Co	Cu	CN	Zn	2-But	CS_2	MeCl	TCE	Tol	Chry	Pyr	Fluor	B(b)F	B(k)F	B(a)P	
Untreated (raw) soil samples																			
A	116	217	NA[c]	11.8	285	41.0	1380	ND[d] (10)[e]	ND (5)	21	180	370	4800	7100	7800	4500	1600	1900	20.1
B	115	248	NA	11.7	328	32.6	1230	ND	ND	24	150	150	3500	8300	7900	3800	1900	1700	20.8
Average	115	232	NA	11.3	314	36.8	1305	ND	ND	22	165	260	4150	7700	7850	4150	1750	1800	20.4
LTTD treated soil samples (300°F, 15 min)																			
A	196	288	317	19	377	16.0	1410	190	17	180	5	160	2200	1400	3700	2500	1300	ND (660)	0.05
B	195	283	310	19	406	22.8	1580	220	22	190	7	140	2400	2400	4600	3000	1500	ND	0.21
Average	195	285	313	19	391	19.4	1495	205	19	185	6	150	2300	1900	4150	2750	1400	ND	0.13
LTTD treated soil samples (300°F, 30 min)																			
A	196	344	297	17.1	351	37.4	1130	300	23	110	8	130	3200	5000	6400	4000	1800	910	0.24
B	185	322	308	17.5	398	43.5	1490	220	7	46	6	60	4900	ND (660)	ND (660)	ND (660)	ND (660)	2700	0.45
Average	190	333	302	17.3	374	40.4	1310	260	15	78	7	95	4050	<2830	<3530	<2330	<1230	1805	0.34
LTTD treated soil samples (500°F, 15 min)																			
A	275	349	272	17.9	390	6.26	1480	29	10	310	6	170	ND (660)	ND (660)	ND (660)	ND (660)	ND (660)	ND (660)	0.09
B	287	300	234	16.0	379	6.19	1600	89	6	200	5	140	ND	ND	ND	ND	ND	ND	0.05
Average	281	324	253	16.9	384	6.22	1540	59	8	255	5	155	ND	ND	ND	ND	ND	ND	0.07
LTTD treated soil samples (500°F, 30 min)																			
A	224	316	255	17.8	343	6.71	1180	15	ND (5)	79[f]	5	5	ND (660)	ND (660)	ND (660)	ND (660)	ND (660)	ND (660)	0.25
B	286	313	274	17.7	378	9.30	1330	43	10	680	5	62	ND	ND	ND	ND	ND	ND	0.10
Average	255	314	264	17.7	366	8.00	1255	29	<7	379	5	33	ND	ND	ND	ND	ND	ND	0.17

[a] 2-But = 2-Butanone, CS_2 = Carbon disulfide
MeCl = Methylene chloride
TCE = Tetrachloroethene
Tol = Toluene

[b] Chry = Chrysene, Pyr = Pyrene
Fluor = Fluoranthene
B(b)P = Benzo(b)fluoranthene
B(k)P = Benzo(k)fluoranthene
B(a)P = Benzo(a)fluoranthene

[c] NA = not analyzed.

[d] ND = not detected.

[e] Numbers in parentheses are the method detection limits.

[f] Number is considered as an outlier.

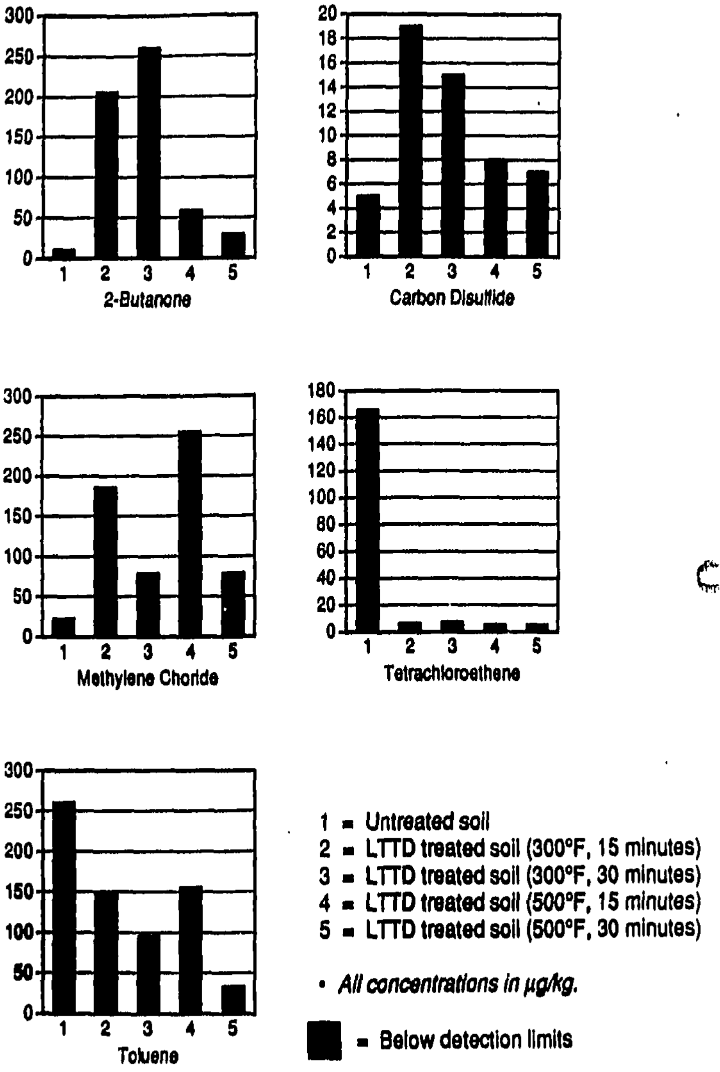
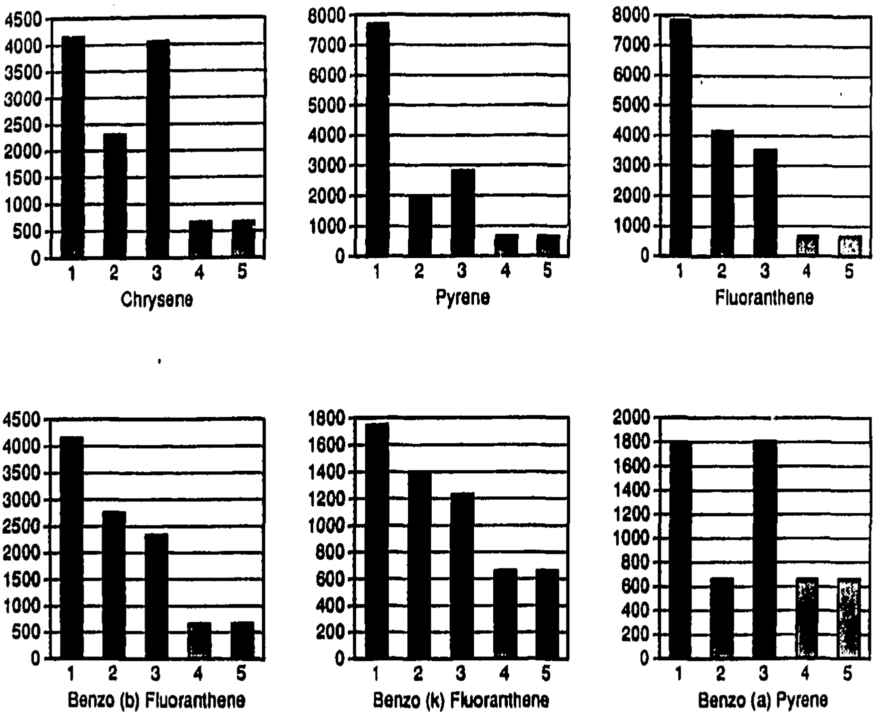


Figure 6-3. Effectiveness of LTTD Process in removal of volatiles from soil samples.

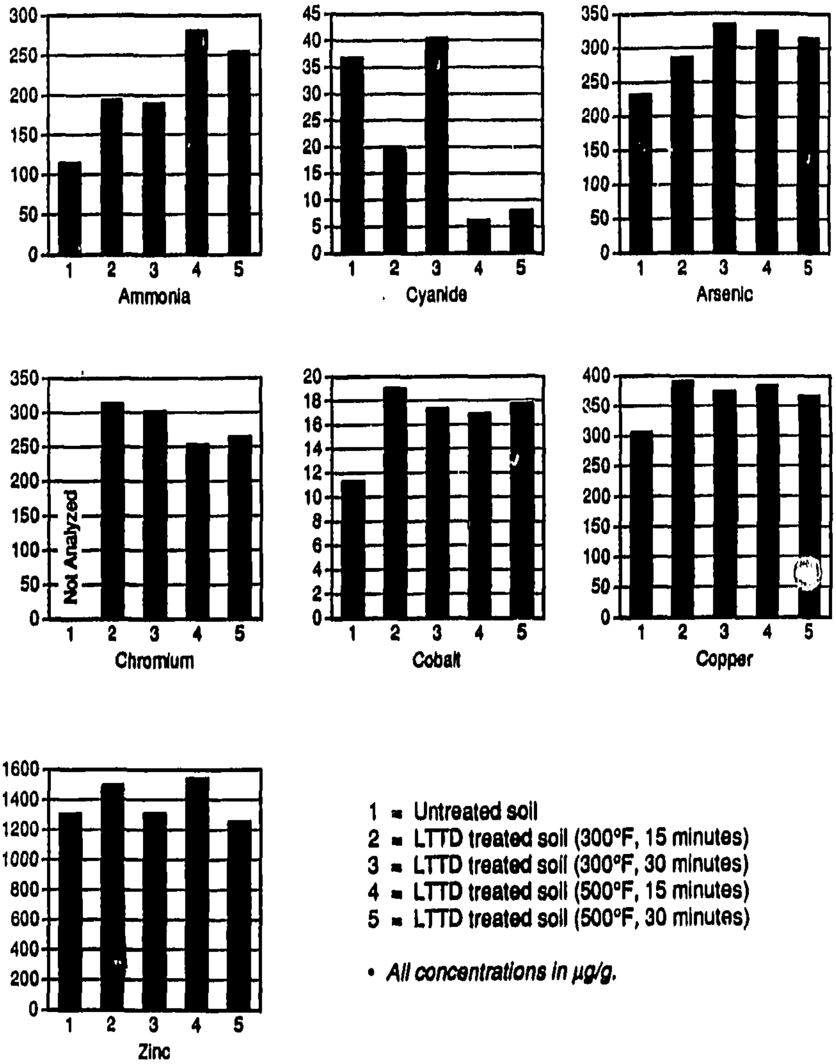


1 = *Untreated soil*
2 = LTTD treated soil (300°F, 15 minutes)
3 = LTTD treated soil (300°F, 30 minutes)
4 = LTTD treated soil (500°F, 15 minutes)
5 = LTTD treated soil (500°F, 30 minutes)

- *All concentrations in µg/kg.*

▨ = Below detection limits

Figure 6-4. Effectiveness of LTTD Process in removal of semi-volatiles from soil samples.



1 = Untreated soil
2 = LTTD treated soil (300°F, 15 minutes)
3 = LTTD treated soil (300°F, 30 minutes)
4 = LTTD treated soil (500°F, 15 minutes)
5 = LTTD treated soil (500°F, 30 minutes)

- *All concentrations in µg/g.*

Figure 6-5. Effectiveness of LTTD Process in removal of various inorganics from soil samples.

TABLE 6-4. ANALYTICAL RESULTS FOR CONDENSATE FROM LTTD TEST RUNS
AT 500°F FOR 30 MINUTES

Indicator Compound	Concentration (μg/l)
Metals	
Arsenic	165
Chromium	33
Cobalt	ND (30)
Copper	142
Zinc	533
VOCs[a]	
2-Butanone	170
Carbon disulfide	ND (5)
Methylene chloride	ND (5)
Tetrachloroethene	ND (5)
Toluene	ND (5)
Semivolatiles[b]	
Chrysene	14
Pyrene	140
Fluoranthene	190
Benzo(b)fluoranthene	ND (10)
Benzo(k)fluoranthene	ND (10)
Benzo(a)pyrene	ND (10)

[a] Low values for VOCs may have resulted from losses through the vacuum line below the condenser.

[b] Low values for semivolatiles may have resulted from poor recoveries.

of the condensate were drawn through a vacuum line that was located below the condenser.

Therefore, it is expected that the VOC concentrations in the off-gases from low-temperature thermal desorption will actually be much higher than indicated in these analyses.

6.2.2 Quality Assurance/Quality Control

As shown in Table 6-3, the analytical results for most of the indicator compounds were similar between replicate test runs. There were some inconsistencies between replicates observed for the semivolatiles (except chrysene) under the 300°F/30 minute test run. In addition, the analytical result for methylene chloride in 500°F/30 minutes test run appears to be an erroneous value. The reason for inconsistent or erroneous results are not known, but matrix effects may have hampered the extraction and analysis for these volatile and semivolatile indicator compounds.

The analytical QA/QC data from matrix surrogate spike and method blank samples indicated good recoveries of metals and inorganic compounds. For volatile and semivolatile compounds, however, some of the recoveries for surrogate compounds and in thermally treated soil samples fell outside the acceptance criteria specified in the [illegible]P. These outlier recoveries appear to be the result of matrix effects, which often occur in analysis of soils with high organic carbon and moisture content, like the Halby site soils.

6.2.3 Costs/Schedule for Studies

The costs for performing the low-temperature thermal desorption studies was $22,000, including chemical analysis and physical tests, labor for the treatability studies, and reporting. The time period needed to complete the studies was 6 weeks, include 1 week for testing and 5 weeks for laboratory analysis.

6.2.4 Key Contacts

The people involved in this study were:

Steve Giti-Pour
PEI Associates, Inc.
11499 Chester Road
Cincinnati, OH 45246

Sarah Hokanson
PEI Associates, Inc.
1233 20th St. N.W.
Washington, DC 20036

SECTION 7

SOLIDIFICATION/STABILIZATION STUDIES

Solidification/stabilization studies were performed on untreated surface soils/sediment and on thermally-pretreated soils (500°F/30 minutes). The solidification/stabilization studies of the solid residues involved two binders [asphalt and portland cement (Type II)] at two different mix ratios. For the untreated surface soils and sediments, only one binder [portland cement (Type II)] was evaluated at two different ratios. The starting materials (i.e., soils and residues) and the stabilized products were subjected to leach testing using TCLP, and the extracts were analyzed for the metal indicator compounds.

The following subsections present the treatability study approach (Subsection 7.1) and the results and discussion (Subsection 7.2).

7.1 Treatability Study Approach

The treatability study approach is shown in Figure 7-1. In this study, solidification/stabilization was evaluated for its effectiveness in reducing leachate concentrations of certain metals in untreated surface soils/sediments and thermal-pretreated soils. The untreated soil and residue from the LTTD were expected to contain high levels of arsenic, cobalt, chromium, copper, mercury, and zinc. Solidification/ stabilization, therefore, was evaluated for its ability to reduce the leaching of these metals remaining in the soil and thermal residues.

Although many different types of binders can be used to solidify/stabilize wastes, these binders can generally be placed into two main categories: inorganic binders and organic binders. Cements and pozzolans (e.g., lime, fly ash, cement kiln dust) are examples of inorganic binders; synthetic organic polymers (e.g., urea formal

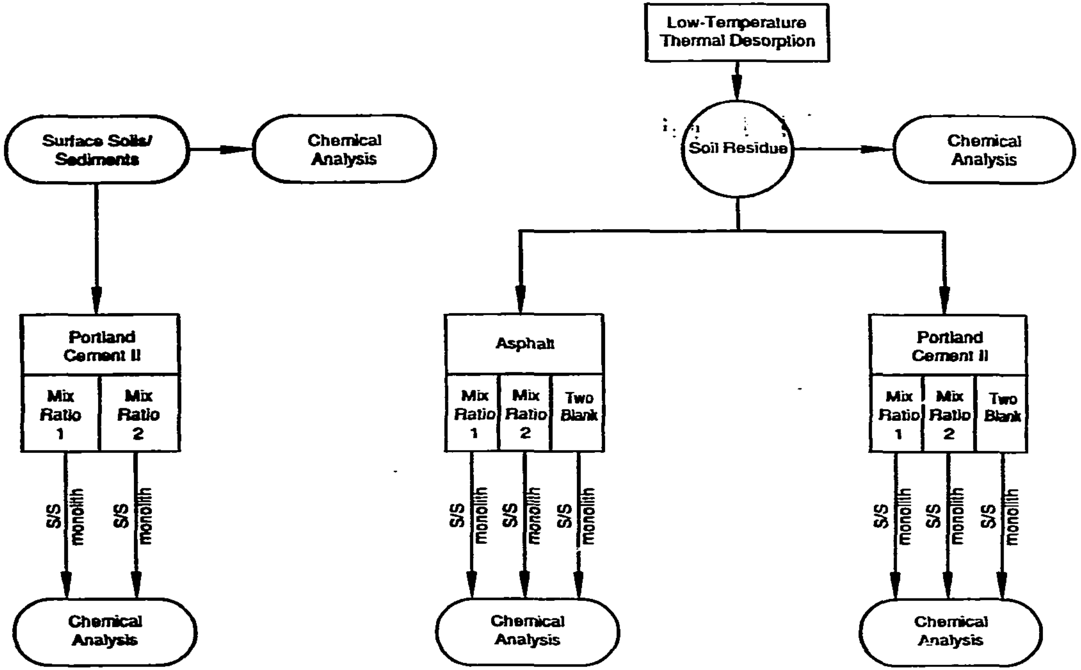


Figure 7-1. Flow diagram of solidification/stabilization.

dehyde) and asphalt are examples of organic binders. In addition, hybrid forms of organic and inorganic binders, such as organophilic clays, are available. Among these different binders, cement is the most commonly used and available binder for metal-bearing wastes. However, an asphalt plant located adjacent to the Halby site may also provide a suitable binder material. Therefore, both cement and asphalt were evaluated in this study.

7.1.1 Test Objectives and Rationale

The performance of this technology was largely based on comparing leach test results for metals in leachate generated from the stabilized products with that from the starting materials. The test objective for this study was to achieve leachate concentrations of 1 ppm for the metal indicator compounds (i.e., arsenic, cobalt, chromium, lead, mercury, and zinc). This test objective of 1 ppm in TCLP leachate was developed in the absence of risk-based levels and EPA regulatory levels for CERCLA soils and debris.

In addition to leach tests, the unconfined compressive strength of stabilized products was conducted in order to compare strengths among the different binders and mix ratios. The test objectives for the strength of stabilized materials is 50 psi, based on preliminary screening criteria currently being developed by EPA for solidification/stabilization studies.

7.1.2 Experimental Design and Procedures

The experimental design for the solidification/stabilization studies is shown in Table 7-1. Detailed discussion of experimental procedures is given in Appendix B-4.

TABLE 7-1. EXPERIMENTAL MIX RATIOS FOR SOLIDIFICATION/STABILIZATION

Binder	Binder-to-soil ratio 1	Binder-to-soil ratio 2	Water-to-soil ratio
Portland cement	0.25	0.4	0.5
Asphalt	0.5	1.0	N/A

Untreated surface soils/sediments and soil residues from low-temperature thermal desorption at the highest temperature and longest residence time (500°F/30 minutes) were homogenized separately, and a portion of the soils underwent TCLP testing and testing for moisture content. Untreated surface soils/sediments were stabilized/solidified with cement only; thermally treated residues were stabilized/solidified with cement and asphalt. The reason for using asphalt binder only on thermally-pretreated residues is because asphalt is generally applied to dry soils, and the raw soils had significant amounts of moisture (between 20 and 30% by weight). Each mix ratio was evaluated in duplicate.

For the cement-based solidification/stabilization tests, the binder was mixed with approximately 800 g of surface soil or thermal residue at ratios of 0.25 and 0.4. Sufficient water was added to the mixture to pass the slump test (ASTM D143). Because the cement binder may contain some metals, a blank run was evaluated consisting of binder mixed with clean sand at the two mix ratios. The samples were allowed to cure for a minimum of 14 days in zip-lock plastic bags in a cooler at 75°F. After curing, the solidified monoliths were leached using TCLP crushed samples. The extracts were then analyzed for the metal indicator compounds. The TCLP results for the surface soil, thermal residues and solidified samples were then compared.

A similar experimental design was used for the asphalt-based solidification/stabilization tests of the thermal residues. The asphalt was heated and mixed with approximately 500 g of thermal residue (which were preheated to 60°C) at ratios of 0.5 and 1 in duplicate and allowed to cure for a minimum of 14 days. Because the asphalt binder may contain some metals, a blank run consisting of clean sand and binder at the two mix ratios was evaluated. The solidified monoliths were leached using TCLP, and the extracts analyzed for the metal indicator compounds. Unconfined compressive strength of the solidified monoliths also was determined.

7.1.3 Equipment and Materials

The cement-based solidification/stabilization studies took place in the same glass reaction vessel used in the low-temperature thermal desorption studies (see

Figure 6-2). The same reaction vessel was also used for the asphalt-based solidification/stabilization studies, except that a thermometer was inserted through one of the openings in the lid. Plastic cylindrical molds with dimensions shown in Table 7-2 were used for curing the mixture.

TABLE 7-2. DIMENSIONS OF CYLINDRICAL MOLDS USED

S/S process	TCLP	UCS
Cement-based	4.5-cm diameter	7-cm diameter
	10 cm long	14.22 cm long
Asphalt-based	4.5-cm diameter	3.8-cm diameter
	10 cm long	8.4 cm long

Reagents used for the cement-based solidification/stabilization studies include tap water and portland cement (Type II). A petroleum-based asphalt was used for the asphalt-based solidification/stabilization studies. The decontamination solutions used for the cement and the asphalt-based solidification/stabilization studies are listed in Appendix B-4.

7.1.4 Sampling and Analysis

Waste Stream--

The untreated surface soils and sediments were homogenized thoroughly in a 5-gallon steel pail and sampled prior to treatability testing for the following analyses:

- Water content (ASTM)
- Soil pH (EPA Method 9045)
- Metals content (EPA Method 6010 and 7000 series)
- TCLP (EPA Method 1320)

The extracts from the TCLP tests were analyzed for the metal indicator compounds listed in Table 1-7.

Treatment Process--

After a curing time of at least 14 days, the solidified/stabilized monoliths were analyzed as follows:

- Unconfined compressive strength (ASTM D1633 for cement-based products and ASTM D2216 for asphalt-based products).
- TCLP (EPA Method 1320)

Two different strength tests were used for the cement- and the asphalt-based products because the physical nature of the two products was different. The cement-based product was a monolithic block, and the asphalt-based product was more soil-like. ASTM D1633 applies to monolithic forms, and ASTM D 2216 is a method that is applied to cohesive soils. For the TCLP test, the treated products were crushed in accordance with the test specifications prior to extraction. The resulting extracts were analyzed for the metal indicator compounds.

7.1.5 Data Management

Data management sheets are given in Appendix A-4. Data recorded during the solidification/stabilization studies were as follows:

- Mixing time and speed
- Reagent and starting material weight and additions
- Curing time, temperature, and humidity
- Temperature during mixing (for asphalt-based)
- Slump (per ASTM C 143)
- Samples collected and analyses performed

7.1.6 Deviations From QAPjP

The deviations in experimental procedures and sample analysis are listed in Table 7-3, along with the rationale for these changes. The changes in procedure resulted from some pretesting studies with mix ratios and from a request by EPA Region III to evaluate solidification/stabilization without the thermal pretreatment step.

7.2 Results and Discussion

The following discussions present the analytical results and data interpretation for the solidification/stabilization studies, as well as results from quality assurance/quality control test runs and analyses, cost/schedule for completing the studies, and key contacts.

TABLE 7-3. DEVIATIONS FROM THE QAPjP

No.	Parameter	Proposed for QAPjP	Actual performance	Rationale
1	Blank samples	Prepare 1 blank sample for portland cement binder and 1 blank sample for asphalt binder.	Two blank samples were prepared with B/S ratios of 0.25 and 0.40 using portland cement, and two blank samples were prepared with asphalt at B/S ratios of 1 and 0.5.	To achieve a better comparison between treated solidified samples and blank solidified samples in terms of cement leaching characteristics.
2	Cement/soil ratio	Prepare 0.2 and 0.4 cement-to-soil ratios (W/W) for cement-based solidified samples.	0.25 and 0.4 cement-to-soil ratios (W/W) were prepared for preparation of cement-based solidified samples.	To better evaluate the optimum condition under which the solidified samples achieve their maximum strength and exhibit minimum leaching characteristics.
3	Curing conditions	Cure the solidified samples in a hermetically sealed chamber at 72°F and 100% relative humidity.	The solidified samples were placed in zip-lock plastic bags and cured in a cooler at temperature of ~75°F.	No hermetically sealed chamber was available at the EPA T&E facility to keep the solidified samples under 72°F and 100% relative humidity.
4	Solidification of raw (unpretreated) soil samples	Not proposed.	Eight additional S/S samples were prepared with raw soil samples for TCLP and USC tests (4 B/S samples for each test).	To collect additional information regarding effectiveness of LTTD on S/S-treated samples and draw more valid conclusions from TCLP and UCS test results in terms of reliability of LTTD as a pretreatment step in remediation of Halby site soil samples.

7.2.1 Data Analysis and Interpretation

Table 7-4 and Figure 7-2 give the results of TCLP leachate analysis for metals from cement-based solidification/stabilization of thermal-pretreated soils (500°F/30 minute runs) and untreated soils. Table 7-5 and Figures 7-2 and 7-3 present the TCLP leachate data for metals from cement- and asphalt-based solidification/stabilization of the thermally-pretreated soils. From these tables and figures, the following conclusions can be made:

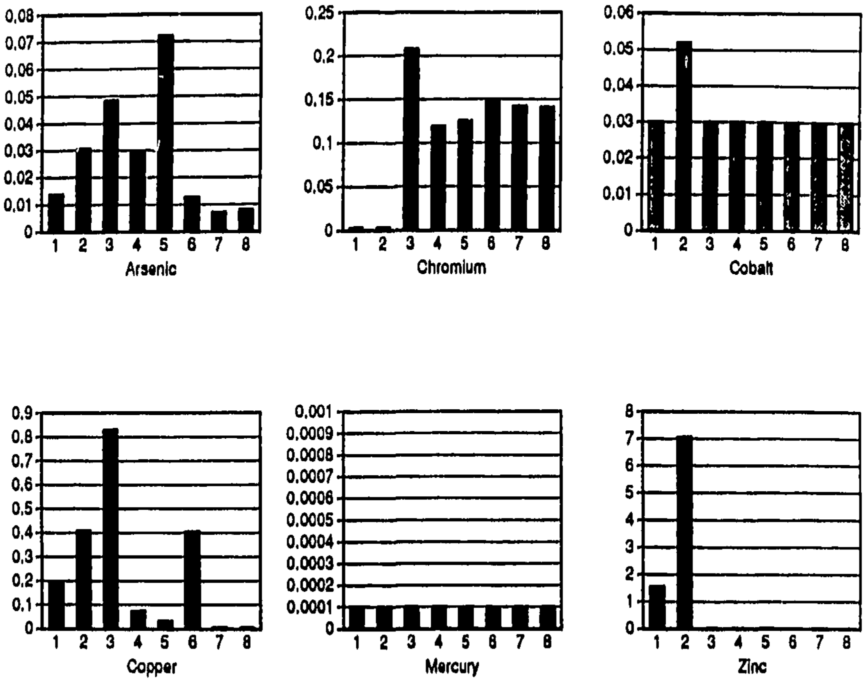
- TCLP leachate concentrations of arsenic, chromium, and mercury from the thermally-pretreated soils and untreated soils are well below the TCLP regulatory levels that are used to classify wastes as characteristically hazardous (40 CFR Part 261.24). In addition, for these unsolidified soil samples, leachate concentrations of all metal indicator compounds are at or below the treatment target level of 1 mg/l.

TABLE 7-4. RESULTS OF TCLP LEACHATE ANALYSIS FOR UNTREATED (RAW), TREATED, AND CEMENT-BASED SOLIDIFIED SOIL SAMPLES (mg/L)

Type of sample	Arsenic	Chromium	Cobalt	Copper	Mercury	Zinc
Untreated (raw) soil						
A	0.0131	ND[a] (0.003)[b]	ND (0.03)	0.201	ND (0.0001)	1.62
B	0.0141	ND	ND	0.187	ND	1.51
Average	0.0136	ND	ND	0.194	ND	1.56
LTTD treated soil (500°F, 30 min)						
A	0.0281	ND (0.003)	0.044	0.400	ND (0.0001)	6.10
B	0.0331	ND	0.060	0.419	ND	8.05
Average	0.0306	ND	0.052	0.409	ND	7.07
Solidified raw soil (B/S = 0.25)						
A	0.0515	0.213	ND (0.03)	0.846	ND (0.0001)	ND (0.02)
B	0.0455	0.204	ND	0.814	ND	ND
Average	0.0485	0.208	ND	0.830	ND	ND
Solidified raw soil (B/S = 0.40)						
A	0.0295	0.115	ND (0.03)	0.071	ND (0.0001)	ND (0.02)
B	0.0285	0.123	ND	0.077	ND	ND
Average	0.0290	0.119	ND	0.074	ND	ND
Solidified LTTD treated soil (B/S = 0.29)						
A	0.0741	0.132	ND (0.03)	0.037	ND (0.0001)	ND (0.02)
B	0.0701	0.121	ND	0.025	ND	ND
Average	0.0721	0.126	ND	0.031	ND	ND
Solidified LTTD treated soil (B/S = 0.40)						
A	0.0131	0.17	ND (0.03)	0.440	ND (0.0001)	ND (0.02)
B	0.0121	0.14	ND	0.371	ND	ND
Average	0.0126	0.15	ND	0.405	ND	ND
Solidified blank soil (B/S = 0.25)						
A	0.0071	0.142	ND (0.03)	ND (0.005)	ND (0.0001)	ND (0.02)
Average	0.0071	0.142	ND	ND	ND	ND
Solidified blank soil (B/S = 0.40)						
A	0.0081	0.141	ND (0.03)	ND (0.005)	ND (0.0001)	ND (0.02)
Average	0.0081	0.141	ND	ND	ND	ND

[a] ND = Not detected.

[b] Numbers in parentheses are the method detection limits.



1 = Untreated soil
2 = LTTD treated soil (500°F, 30 minutes)
3 = Solidified raw soil (B/S = 0.25)
4 = Solidified raw soil (B/S = 0.40)
5 = Solidified LTTD treated soil (B/S = 0.25)
6 = Solidified LTTD treated soil (B/S = 0.40)
7 = Solidified blank soil (B/S = 0.25)
8 = Solidified blank soil (B/S = 0.40)

- *All concentrations in mg/L.*

■ = Below detection limits

Figure 7-2. Results of TCLP leachate analysis for cement-based solidified samples.

TABLE 7-5. RESULTS OF TCLP LEACHATE ANALYSIS FOR LTTD-TREATED AND ASPHALT-BASED SOLIDIFIED SOIL SAMPLES (mg/L)

Type of sample	Arsenic	Chromium	Cobalt	Copper	Mercury	Zinc
LTTD treated soil (500°F, 30 min)						
A	0.0281	ND (0.003)	0.044	0.400	ND (0.0001)	6.10
B	0.0331	ND	0.060	0.419	ND	8.05
Average	0.0306	ND	0.052	0.409	ND	7.07
Solidified LTTD treated soil (B/S = 0.50)						
A	ND (0.005)	0.006	ND (0.03)	0.165	ND (0.0001)	0.553
B	ND	0.008	ND	0.017	ND	0.329
Average	ND	0.007	ND	0.091	ND	0.441
Solidified LTTD treated soil (B/S = 1.00)						
A	ND (0.005)	ND (0.003)	ND (0.03)	0.013	ND (0.0001)	0.546
B	ND	ND	ND	0.015	ND	0.276
Average	ND	ND	ND	0.014	ND	0.411
Solidified blank soil (B/S = 0.50)						
A	ND (0.005)	ND (0.003)	ND (0.025)	ND (0.005)	ND (0.0001)	0.292
Average	ND	ND	ND	ND	ND	0.29
Solidified blank soil (B/S = 1.00)						
A	ND (0.005)	ND (0.003)	ND (0.025)	ND (0.005)	ND (0.0001)	0.335
Average	ND	ND	ND	ND	ND	0.335

[a] ND = Not detected.

[b] Numbers in parentheses are the method detection limits.

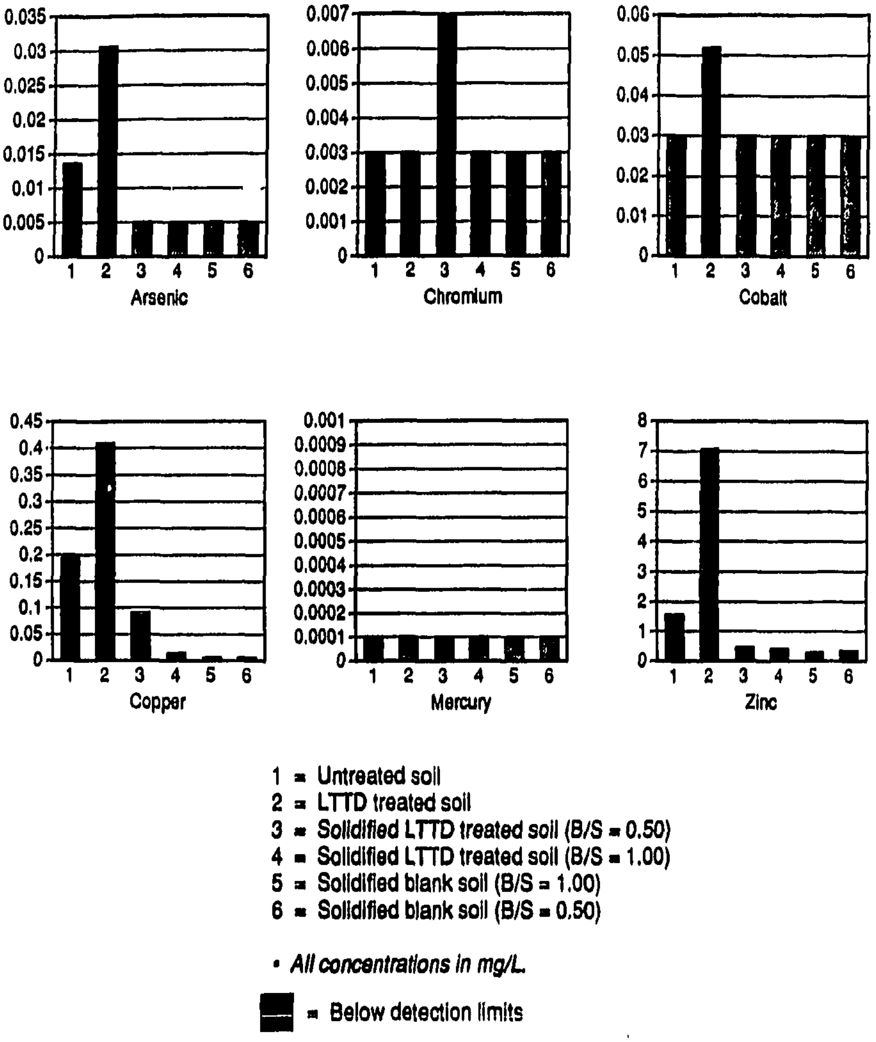


Figure 7-3. Results of TCL¯ leachate analysis for asphalt-base solidified samples.

- Although leachate concentrations are low, those for arsenic and copper are significantly reduced by asphalt binder at both mix ratios; the cement binder did not perform as well as the asphalt, and in the case of arsenic and copper, it resulted in increased concentrations.

- Both the asphalt and cement binders significantly reduced leachate concentrations of zinc.

- Increased chromium concentrations observed in leachate from cement-based products may have resulted from chromium in either the portland cement material or the tap water used during the mixing process, since the levels are comparable to that found for the two blank samples. Chromium is generally known to leach more readily under basic condi'ons such as those created by the cement process. Chromium is esent at levels near or below detection limits in the leachate from the phalt-based products.

- achate levels of the metal indicator compounds are similar for the cement-based products of thermally-pretreated soil and untreated soil.

The moderate to high concentrations of metals (between 200 and 1500 mg/kg) present in the thermally-pretreated and untreated soils (Table 6-4) apparently do not readily leach in appreciable amounts under TCLP test conditions. Leachate concentrations of these metals may be much greater, however, in multiple extraction tests or other leach tests that are designed to address long-term leaching rate over time. The need for solidification/stabilization of soils should be investigated by conducting additional leach testing of the soils.

While the asphalt appears to be the better binder of the two studied for arsenic and copper, the lower leachate levels reported for the asphalt-based products may be partially due to higher dilution by asphalt than by cement (i.e., higher binder to soil mix ratios). In addition, several issues should be evaluated further prior to selecting as the solidifying/stabilizing agent for previously untreated soils: (1) need for dewatering of the soils prior to mixing, (2) potential VOC emissions during mixing, and (3) potential leaching.

In addition to TCLP leachate levels, unconfined compressive strength tests were run on the stabilized products. Table 7-6 lists the results of the UCS tests on asphalt-

TABLE 7-6. TEST RESULTS FOR UNCONFINED COMPRESSIVE STRENGTH (UCS) OF ASPHALT-BASED[a] AND CEMENT-BASED PRODUCTS[b]

Sample type	Bearing load (lbs)	Yield stress (psi)
Asphalt-based[a]		
(B/S = 1.0)		
A	71	43
B	51	31
Average	61	37
(B/S = 0.5)		
A	98	58
B	153	88
Average	126	73
Cement-based[b] LTTD-treated soil		
(B/S = 0.25)		
A	7200	1235
B	6760	1162
Average	6980	1199
(B/S = 0.40)		
A	5910	1029
B	8660	1526
Average	7285	1278
Cement-based[b] untreated soil		
(B/S = 0.25)		
A	4630	788
B	4220	698
Average	4425	743
(B/S = 0.40)		
A	8700	1480
B	7060	1158
Average	7880	1319

[a] UCS tests performed in accordance with ASTM D2216

[b] UCS tests performed in accordance with ASTM D1633

and cement-based products. In most cases, the strengths of the asphalt and cement products exceeded the target level of 50 psi specified for these studies. Only the asphalt-based product (B/S = 1.0) recorded lower stress levels. Overall, these tests results indicate that cement-based, monolithic structures yield at a higher stress under larger bearing loads than asphalt-based products.

7.2.2 Quality Assurance/Quality Control

The data shown in Table 7-4 and 7-5 for replicate test runs indicate good reproduceability of TCLP leachate levels for metals. In addition the percent recoveries from standard reference solutions, and matrix spike and matrix spike duplicate samples were all within the acceptance criteria outlined in the QAPjP.

7.2.3 Costs/Schedule for Studies

The costs for performing the solidification/stabilization studies are $30,000, including labor for testing, laboratory costs, and report preparation. The time needed to perform the studies was 8 weeks, including 4 weeks for solidification/stabilization and 4 weeks for laboratory analysis.

7.2.4 Key Contacts

Key contacts for these studies are:

Steve Giti-Pour
PEI Associates, Inc.
11499 Chester Road
Cincinnati, OH 45246

Sarah Hokanson
PEI Associates, Inc.
1233 20th St. NW
Washington, DC 20036

APPENDIX A

TREATABILITY TESTING DATA MANAGEMENT SHEETS

CONTENTS

APPENDIX A

ORIGINAL
(Red)

APPENDIX A-1

TESTING DATA MANAGEMENT SHEETS FOR XANTHATE STUDIES

Project No. 20526?
TLE PES Halby Book No.

om Page No.

Halby Set up

1) Field samples ~~[illegible]~~ of soil from same location

2) also water samples from same well

3) Will use only one jar/bottle of soil + water no need to composite

4) Parameters to measure on 4 H_2O samples + 1 soil (fines)

A. HP
B. pH
☆ C. enumerations - special [illegible] + fuel [illegible]
D. TOC
E. Stimulation

5) Treatability -

A. Composite + fuel + O_2
B. Composite + O_2
C. Untreated
D. Bio inhibited

} days measure bugs + O_2

To Page No.

itnessed & Understood by me,
Date
Invented by
Date 4/[illegible]

Project No. 895169
Book No. ______ **TITLE** ~~Hallg~~ Halby

From Page No. __

Hall-Bog set up - used #1 water and #2 soil

	wet soil	Sta H_2O	Addition
1	2.2g	20.0	1000 ppm R375
2	1.98	20	—
3	1.96	20	He
4	1.92	20	100 ppm $HgCl_2$
5	2.03	20	1000 ppm R375
6	~~1.96~~ 2.10	20	—
7	1.96	20	He
8	2.02	20	100 ppm $HgCl_2$
B1	2.05	20	— 0^{*}
B2	2.03	20	— 0^{*}

Samples 1 & 5 received 1000 ppm Restore 375 - 0.2 ml of 10% so-

Samples 4 & 8 received 100 ppm $HgCl_2$ = 0.02 ml of saturated

Samples 3 & 7 had head space purged with He

B1 & B2 are untreated samples used for T_0 bug counts & TOC

To Pa

Witnessed & Understood by me,	Date	Invented by	Date
			01 /

Project No. [illegible]

LE Halby Book No.

m Page No.

TO Oxygen Data

Calibration values 100, 50, 25, 10 ul of AIR

samples # = vessel #

9 = pure air test injection

10: H2 test injection

```
PEI/Halby
Operator: Duane Graves      04-15-1990    15:52:00
Halby Superfund Site--TO Oxygen Data
_____________________________________________________________________

SAMPLE #       SAMPLE VOL     OXYGEN CONTENT, uM          OXYGEN, ppm
 1              50             9.44697                    188939.4 O2 + [illegible]
 2              50             9.422912                   188458.3 O2
 3              50             .8532809                   17065.62 H2
 4              50             9.818176                   196363.5 H2/CH4
 5              50             9.957497                   199149.9 O2 + [illegible]
 6              50             9.746837                   194936.7 O2
 7              50             .7146958                   14293.92 ~~[illegible]~~ [illegible]
 8              50             9.868592                   197371.9 H2/CH4
 9              50             10.77178                   215435.6 AIR
 10             50             .953272                    19065.44 H2
_____________________________________________________________________

CALIBRATION DATA
Linear Regression:    y=a+bx

   a =  0
   b =  17.41161
```

To Page No.

essed & Understood by me,	Date	Invented by	Date 4/1/

```
FEI/Halby
Operator: Duane Graves        04-15-1990     15:52:00
Halby Superfund Site--TO Oxygen Data

SAMPLE #        SAMPLE VOL     OXYGEN CONTENT, uM        OXYGEN, ppm
 1               50             9.44697                   188939.4
 2               50             9.422912                  188458.3
 3               50             .8532809                  17065.62
 4               50             9.818176                  196363.5
 5               50             9.957497                  199149.9
 6               50             9.746837                  194936.7
 7               50             .7146958                  14293.92
 8               50             9.868592                  197371.9
 9               50             10.77178                  215435.6
 10              50             .953272                   19065.44

CALIBRATION DATA
Linear Regression:    y=a+bx

  a =  0
  b =  17.41161
```

```
Halby/PEI
Operator: Duane Graves      04-17-1990    10:48:55
Halby Superfund Site -- T1 Oxygen Data

SAMPLE #      SAMPLE VOL    OXYGEN CONTENT, uM         OXYGEN, ppm
 1             50            11.10906                   222181.2
 2             50            10.50969                   210193.8
 3             50            9.092619                   181852.4
 4             50            11.29109                   225821.8
 5             50            9.777868                   195557.4
 6             50            9.997674                   199953.5
 7             50            1.490291                   29805.82
 8             50            12.10643                   242128.6
 9             50            11.62302                   232460.4
 10            50            1.334608                   26692.16

CALIBRATION DATA
Linear Regression:   y=a+bx

    a =  0
    b =  16.18677
```

```
Halby/PEI
Operator: Tim Schalk         04-19-1990     10:31:57
Halby Superfund Site T2 Oxygen Data

SAMPLE #       SAMPLE VOL     OXYGEN CONTENT, uM              OXYGEN, ppm
  1              50             10.2018                         204036
  2              50             9.858506                        197170.1
  3              50             9.836626                        196732.5
  4              50             10.66861                        213372.2
  5              50             9.337919                        186758.4
  6              50             10.12332                        202466.4
  7              50             4.781703                        95634.06
  8              50             10.53309                        210661.8
  9              50             10.76613                        215322.5
  10             50             2.383631                        47672.62

CALIBRATION DATA
Linear Regression:    y=a+bx

   a =  0
   b =  16.5508
```

[illegible]

Halby/PEI
Operator: Duane Graves 04-21-1990 15:33:59
Halby Superfund Site -- T3 Oxygen Data

SAMPLE #	SAMPLE VOL	OXYGEN CONTENT, uM	OXYGEN, ppm
1	50	7.970416	159408.3 O_2 + N_2
2	50	9.763379	195267.6 O_2
3	50	9.428318	188566.4 He
4	50	10.20665	204132.9 O_2 + H_2Cl_2
5	50	7.758327	155166.5 O_2 + [illegible]
6	50	10.0429	200857.9 O_2
7	50	8.884896	177697.9 He
8	50	9.924177	198483.6 O_2 + H_2Cl_2
9	50	9.265759	185315.2 AIR + Contamination
10	50	1.463327	29266.54 He
11	50	10.3694	207387.9 AIR
12	50	10.36718	207343.5 AIR

CALIBRATION DATA
Linear Regression: y=a+bx

a = 0
b = 15.30465

```
Halby
Operator: Keith Hague          04-23-1990     10:56:35
Oxygen sampled 4/24/90

SAMPLE #       SAMPLE VOL     OXYGEN CONTENT, uM          OXYGEN, ppm
 1              50             6.779321                    135586.4 Air+O2
 2              50             10.05521                    201104.2 He Air
 3              50             10.514                      210280  He
 4              50             10.44753                    208950.7 H2Cl2
 5              50             7.088239                    141764.8 O2 Air
 6              50             10.8848                     217696  No Air
 7              50             5.2903                      105806  He
 8              50             11.11504                    222300.7 H2Cl2
 9              50             10.96417                    219283.4 AIR
 10             50             11.32543                    226508.7 AIR
 11             50             11.32073                    226414.6 AIR
 12             50             .3466872                    6933.745 He

CALIBRATION DATA
Linear Regression:  y=a+bx

   a =  0
   b =  14.88085
```

```
Halbv/PEI
Operator: Keith Hague          04-25-1990     17:29:49
oxygen analysis 4-26-90

SAMPLE #        SAMPLE VOL     OXYGEN CONTENT, uM         OXYGEN, ppm
 1               50             6.099147                   121982.9 · C2 [illegible]
 2               50             9.939381                   198787.6 · C2
 3               50             10.62239                   212447.7 · He
 4               50             10.75047                   215009.5 · C2 + [illegible]
 5               50             3.513128                   70262.55 · C2 [illegible]
 6               50             9.870179                   197403.6 · C2
 7               50             4.477218                   89544.36 · He
 8               50             10.39076                   207815.3 · C2 + [illegible]
 9               50             10.48482                   209696.4 · Air
 10              50             10.39789                   207957.8 · Air
 11              50             10.38752                   207750.4 · Air
 12              50             7.056736E-02                        - He
 1411.347

CALIBRATION DATA
Linear Regression:    y=a+bx

  a =  0
  b =  16.01308
```

```
Halby/FEI
Operator: Keith Hague        04-29-1990     17:26:14
Oxygen analysis 4-30-90

SAMPLE #        SAMPLE VOL     OXYGEN CONTENT, uM          OXYGEN, ppm
 1                50            9.195559                    183911.2 - O2 + nutrients
 2                50            9.718788                    194375.7 - O2
 3                50            10.52768                    210553.7 - He
 4                50            10.77885                    215577   - O2 + HgCl2
 5                50            3.4924                      69847.99 - O2 + nutrients
 6                50            10.76855                    215371   - O2
 7                50            1.044334                    20886.69 - He
 8                50            9.952459                    199049.2 - O2 + HgCl2
 9                50            10.24984                    204996.8 - Air
 10               50            10.08285                    201657.1 - Air
 11               50            10.00107                    200021.5 - Air
 12               50            .2947127                    5894.253 - He

CALIBRATION DATA
Linear Regression:   y=a+bx

   a =  0
   b =  14.56334
```

MICROBIOLOGY ANALYSIS

Site Location: Halby
Site Code:
Date Rec'd:
Collected by:
Date Collected:
Sample Type:
Sample Condition:

INITIAL ENUMERATION

Set-up By: RD
Date: 5/8/98
Counted By: DL
Date: 5/-6/98

ENUMERATION METHOD:

PLATE COUNT ✓
TOTAL BACTERIA
CS2 degraders
Dilution Factor:

MPN TUBES
CONTAMINANT DEGRADERS*
Dilution Factor:

Well/Core#	Wet Wt.	Dry Wt.	%H2O	No. of Colonies						CFU/ml(gm)	No. of Colonies						CFU/ml(gm)
Dilution Factor:				1	2	3	4	5									
Treatment 1				TN	TN	TN	53 ~~[illegible]~~	~~60~~ 11 ~~200~~		2e6							
2				TN	TN	TN	212	32		3e6							
3				TN	TN	TN	TN	102		1e7							
4				TN	TN	TN	TN	82		8.2e6							
5 nut + O2				TN	TN	TN	[illegible]	250 ~~[illegible]~~		2.5e7							
6 O2				TN	TN	TN	TN	75		7.5e6							
7 He				TN	TN	TN	TN	52		5.2e6							
8 HgCl				TN	TN	TN	TN	85		8.5e6							

*Comments (Specific Conditions):

MICROBIOLOGY ANALYSIS

Site Location: Holly lab reports
Site Code:
Date Rec'd:

Collected by:
Date Collected:

Sample Type:
Sample Condition:

INITIAL ENUMERATION

Set-up By: RD
Date: 5/8

Counted By: DG
Date: 5/14 Thousands)

ENUMERATION METHOD:

PLATE COUNT
TOTAL BACTERIA
Thousands
Dilution Factor:

MPN TUBES
CONTAMINANT DEGRADERS*
Dilution Factors

				PLATE COUNT							MPN TUBES						
Dilution Factor				1	2	3	4	5									
Well/Core#	Wet Wt.	Dry Wt.	%H_2O	No. of Colonies						CFU/ml(gm)	No. of Colonies						CFU/ml(gm)
#1				TN	TN	146	17	3		1.5e5							
2				TN	TN	58	6	2		5.7e4							
3				TN	63	8	0	0		6.5e3							
4				TN	74	12	1	0		7.5e3							
5				TN	34	TN	16	15		1.6e6							
6				TN	31	3	0	0		3.1e3							
7				102	10	0	0	0		1e3							
8				TN	182	23	3	0		2e4							

*Comments (Specific Conditions):

MICROBIOLOGY ANALYSIS

Site Location: Halby
Site Code:
Date Rec'd:
Collected by:
Date Collected:
Sample Type:
Sample Condition:

INITIAL ENUMERATION

Set-up By: R. Dameron / K. Hague
Date: 5/1/90
Counted By: R. Dameron
Date: 5/7/90

ENUMERATION METHOD:

PLATE COUNT: Nut. PLATES
TOTAL BACTERIA

MPN TUBES
CONTAMINANT DEGRADERS*

Well/Core#	Wet Wt.	Dry Wt.	%H₂O	No. of Colonies (Dilution Factor 1)	2	3	4	5	6	CFU/ml(gm)	No. of Colonies (Dilution Factor)						CFU/ml(gm)
#1				TN	TN	TN	384	92	68	6.8×10^7							
#2				TN	TN	TN	TN	100	13	1.0×10^7							
#3				TN	TN	TN	TN	TN	169	1.7×10^8							
#4				TN	TN	TN	TN	222	37	3.7×10^7							
#5				TN	TN	[illegible]	TN	TN	184	1.8×10^8							
#6				TN	TN	TN	TN	TN	[illegible]	1.7×10^8							
#7				TN	TN	TN	TN	235	55	5.5×10^7							
#8				TN	TN	TN	TN	TN	95	9.5×10^7							

*Comments (Specific Conditions):

MICROBIOLOGY ANALYSIS

Site Location: Halby - Anaerobic

Site Code:

Date Rec'd:

Collected By:

Date Collected:

Sample Type:

Sample Condition:

INITIAL ENUMERATION

Set-up By:

Date: 4/12/90

Counted By: JN

Date: 4/13/90

ENUMERATION METHOD:

				PLATE COUNT TOTAL BACTERIA							MPN TUBES CONTAMINANT DEGRADERS* Thiocynate plates						
				Dilution Factor:							Dilution Factor:						
				1	2	3	4	5	6		1	2	3	4	5	6	
Well/Core#	Wet Wt.	Dry Wt.	%H₂O	No. of Colonies						CFU/ml(gm)	No. of Colonies						CFU/ml(gm)
Sed				-	TC	31	2	0	0	3e4	6	0	0	0	0	0	—
1				20	7	2	0	0		1.4e2	0	0	0	0	0	0	—
2				93	19	1	0	0		1.3e3	0	0	0	0			—
3				43	6	1	0	0		4.3e2	0	0	0	0			—
4				22	29	2	2	0		2.9e2	0	0	0	0			—

*Comments (Specific Conditions):

MICROBIOLOGY ANALYSIS

Site Location: Halby Part II
Site Code: ______ Date Rec'd: ______
Collected By: ______
Date Collected: ______
Sample Type: ______
Sample Condition: ______

INITIAL ENUMERATION

Set-up By: ______ Date: 4/13/90
Counted By: DG Date: 4/27/90

ENUMERATION METHOD:

AEROBIC

PLATE COUNT
TOTAL BACTERIA
Thiocyanate

MPN TUBES
CONTAMINANT DEGRADERS*
CS2

Well/Core#	Wet Wt.	Dry Wt.	%H2O	Dilution Factor: 1	2	3	4	5	6	CFU/ml(gm)	Dilution Factors: 1	2	3	4	5	6	CFU/ml(gm)
				No. of Colonies							No. of Colonies						
Halby Sediment				–	13#	22	2	0	0	2e4	–	45#	21	2	3	1	2e4
Halby H2O 1				0	0	0	0			–	4	1	0	0	1		70
2				0	0	0	0			–	6	1	0	0	0		60
3				0	0	0	0	0		–	8	1	0	0	0		80
4				0	0	0	0	0		–	17	0	0	0	0		170

*Comments (Specific Conditions): note Halby sed plate was very hard to count # is not accurate count

ORIGINAL (Red)

MICROBIOLOGY ANALYSIS

Site Location: Holly

Site Code:

Date Rec'd:

Collected By: DG

Date Collected:

Sample Type:

Sample Condition:

INITIAL ENUMERATION

Set-up By:

Date: 4/13/90

Counted By: DG

Date: 4/22/90

ENUMERATION METHOD:

PLATE COUNT

TOTAL BACTERIA

Aerobic

Dilution Factor: 1 2 3 4 5 6

MPN TUBES

CONTAMINANT DEGRADERS*

Anaerobic

Dilution Factor: 1 2 3 4 5 6

CS₂

Well/Core#	Wet Wt.	Dry Wt.	%H₂O	No. of Colonies 1	2	3	4	5	6	CFU/g(ml)	No. of Colonies 1	2	3	4	5	6	CFU/g(ml)
Holly Seline				TN	TNTC	187	26	6	4	2.2e5*	TC	56*	23	0	0		2e5 [illegible] 5.6e4
Holly 1		Water		18	2	0	0	0		1.8e2	0	0	0	0	0		—
2				135	7	1	0	0		1e3	12	1	0	0	0		120
3				7	0	0	0	0		70	25	2	0	0	0		220
4				13	1	0	0	0		1.3e2	12	0	0	0	0		120

*Comments (Specific Conditions):

ORIGINAL (Red)

Project No.______

E Halby Book No.______

n Page No.__

ORIGINAL (Red)

Sample	PO_4 ppm	NH_4 ppm	Ph
Sediment	190.1	49.1	5.9

To Page No.____

essed & Understood by me,	Date	Invented by R. Dameron	Date 4/24/90
		Recorded by	

INTERNATIONAL TECHNOLOGY CORPORATION

ORIGINAL (Red)

By JN Date 5/7 Subject Halby Sheet No.__ of

Chkd. By__ Date__ Proj. No.

Standards

Phosphate		Ammonium	
RB	.035	RB	.045
0.5	.124	0.5	.109
1.0	.212	3.0	.565
2.0	.383	9.0	1.566

Corr = .9999
DL = 0.5

Corr = .9998
DL = 0.5

Samples	Phosphate	Ammonium	pH
1	< DL	200.6	6.5
2	< DL	195.9	6.3
3	< DL	190.6	6.3
4	< DL	211.2	6.3

INTERNATIONAL TECHNOLOGY CORPORATION

By ______ Date 4/13/90 Subject PEI - TOC Sheet No. ORIGINAL (Red) of ______
Chkd. By ______ Date ______ ______ Proj. No. ______

SAMPLE	Total Carbon	Total Inorganic	Total Organic
1	435.6	183.6	252.0
2	465,1	210.6	254.5
3	470,7	213,3	257.4
4	467.6	209.7	257.9

INTERNATIONAL TECHNOLOGY CORPORATION

By JN Date 4/30 Subject Moisture Sheet No. ___ of ___
Chkd. By ___ Date ___ Proj. No. ___

Keith, here are moistures, I don't know where summation sheets are

[illegible]	Wet	Dry	% H2O
0.5%	4.6	4.06	11.7
1.0%	2.77	2.28	17.7
No Nut	3.48	2.05	17.6
Nut	3.81	3.31	13.1

HRI	Wet	avg	% H2O
SBw	6.76	6.38	5.7
SB1	7.11	5.68	20.1
Tr Sample	28.14	24.18	14.1
Lined 7	15.58	12.59	19.2

	Wet	Dry	% H2O
Halby comp	21.63	16.25	25.0

ORIGINAL
(Red)

APPENDIX A-2

TESTING DATA MANAGEMENT SHEETS FOR XANTHATE STUDIES

Halby Xanthate Treatability Study

7 August 1990

8:40 Cleaned + rinsed w/DI H_2O:

1 4l Flask
Stirring paddle assembly
Misc. beakers

9:30 Filled 2 - 8 oz glass sample bottles w/ raw soil

XF-US-A
XF-US-B

10:30 Test Run I

1) Added 500 g (498.6 g) soil to 4l beaker Filled w/ 3.5l DI water.

2) Begin stirring + bubbling

ORIGINAL (Red)

10:32 7.8.90

3) Added 1 pellet xanthate and 1 drop Frother @ 10:32

4) No visible bubbles

5) Stirring stopped @ 10:37 Stirring destroys the bubbles. Still very little.

6) Increased Frother to 6 drops and 3 pellets of xanthate

7) No improvement in bubble froth production

8) Increased Frother to 16 drops and 5 pellets of xanthate

9) Very little froth appers

10) Treatment stopped @ 11:12

11:12 7.8.90

11) Soil allowed to settle in Flask

12) Call to Judy - Continue with test. Try to collect whatever froth forms.

13) Decant water from Flask into HW liquid barrel.

14) Fill 1 - 8oz glass sample jar w/ treated soil

XF-S-M-A

15) Did not sample the "froth"

16) Cleaned Flask and glassware. Rinsate placed in HW liquid barrel

ORIGINAL (Red)

11:44 Test Run II 7.8.90

1) Dissolved 16 drops Frother and 8 pellets of xanthate in ~3500 ml D.I. water. Mixed.

2) Added 500.1 g soil to the water/reagent mix.

3) Began slow mixing, bubbling

4) Mixing stopped at 12:00.

5) Bubbles "coaxed" manually with the bubble stone.

6) Froth removed manually by ~~spatula~~ spoon. Skimmed the froth.

7) Only very little froth produced

8) Treatment stopped @ 12:15

12:16 7.8.90

9) Soil allowed to settle. Top
liquid decanted into HW
liquid drum.

10) Filled 1-8oz glass sample jar
w/ treated soil

YF-S-M-B

11) Filled 1-8oz glass sample jar
w/ "froth" (more water than froth)

YF-Fl-M-B

1:30

All glassware + equipment
deconned w/ soap + Hot water

Samples packed in cooler w/ ice
bottles for transport.

APPENDIX A-3

TESTING DATA MANAGEMENT SHEETS FOR LOW-TEMPERATURE THERMAL DESORPTION STUDIES

APPENDIX A

LABORATORY MEASUREMENTS FOR LOW TEMPER

THERMAL DESORPTION OF SOIL SAMPLES

Testing of Soil Samples for Low-Temperature Thermal Desorption Process

Test 1 (run 1)

Date: 6/1/90

Reaction temperature = 300°F (149°C)

Reaction time = 15 minutes

Weight of the untreated (raw) soil sample = 1200 g
Weight of the treated soil residue and reaction flask = 1871 g
Weight of the treated soil residue = 870 g

Stirring rate = 115 rpm

TABLE A-1. Time, Temperature, and Heating Mantle Controller Setting for the Soil Samples During LTTD Test

Time, minutes	Temperature, °C	Heating mantle controlle set point
0:00	23	55
0:15	30	"
0:30	42	"
0:45	51	"
1:00	57	"
1:15	80	60
1:30	100	"
1:45	100	"
2:00	100	"

2:15	100	″	
2:30	108	″	
2:45	112	70	
3:00	115	″	
3:15	118	″	
3:30	123	60	
3:45	125	″	
4:00	148	″	
4:01	149	″	start of testi
4:06	155	″	
4:11	152	″	
4:16	149	″	End of testing

Test 1 (run 2)

Date = 6/1/90

Reaction temperature = 300°F (149°C)

Reaction time = 15 minutes

Weight of the untreated (raw) soil sample = 1200 g
Weight of the treated soil residue and reaction flask = 1942 g
Weight of the treated soil residue = 890 g

Stirring rate = 115 rpm

TABLE A-2. Time, Temperature, and Heating Mantle Controller Setting for the Soil Samples During LTTD

Test 1

Time, minutes	Temperatures, °C	Heating mantle controller set point
0:00	23	55
0:15	40	"
0:30	63	"
0:45	69	"
1:00	71	"
1:15	91	60
1:30	100	"

.. 1:45	100	"	
.. 2:00	100	"	
.. 2:15	100	"	
.. 2:30	108	70	
.. 2:45	115	"	
.. 3:00	118	"	
.. 3:15	125	"	
3:30	138	55	
..3:37	150	"	Start of testing ..
..4:42	160	"	
..4:47	160	"	
4:52	150	"	End of testing

Test 2(run1)

Date : 6/4/90

ORIGINAL
(Red)

Reaction temperature = 500°F (260°C).

Reaction time = 15 minutes

Weight of the untreated (raw) soil sample = 800g
Weight of the treated soil residue and reaction flask = 1554 g
Weight of the treated soil residue = 552 g

Stirring speed = ≈ 100 rpm

TABLE A.3. Time, Temperature, and Heating Mantle Controlle

Readings during LTTD Test.

Time, minutes	Temperature, °C	Heating Mantle Controller Set Point
0:00	22	55
0:15	44	″
0:30	55	″
0:45	70	″
1:00	70	″
1:15	70	″
1:30	70	60
1:45	75	″
2:00	80	″

2:15	80	60	
2:30	80	//	
2:45	85	//	
3:00	112	//	
3:15	115	//	
3:30	122	//	
3:45	137	//	
4:00	160	//	
4:15	180	//	
4:30	195	//	
4:45	210	//	
5:00	225	//	
5:15	240	//	
5:30	255	//	
5:40	260	//	Start of test
5:45	260	//	
5:50	260	//	
5:55	260	//	End of testin

ORIGINAL
(Red)

Test 2 (run2).

Date: 6/4/90
Reaction temperature = 500°F (260°C)
Reaction time = 15 minutes
Weight of the untreated (raw) soil sample = 800 g
Weight of the treated soil residue and reaction flask = 1598 g
Weight of the treated soil residue = 548 g
Stirring speed = 112 rpm

TABLE A-4. Time, Temperature, and Heating Mantle Controller Setting during LTTD test.

Time, minutes	Temperature, °C	Heating Mantle Controller Set Point
0:00	24	55
0:15	45	"
0:30	60	"
0:45	65	"
1:00	70	"
1:15	75	"
1:30	75	"
1:45	75	"
2:00	78	60
2:15	78	"
2:30	80	"
2:45	80	"
3:00	100	"
3:15	100	"
3:30	110	"
3:45	118	"

4:00	130	60	
4:15	150	"	
4:30	155	65	
4:45	170	"	
5:00	180	"	
5:15	180	"	
5:30	195	"	
5:45	215	"	
6:00	230	"	
6:15	246	"	
6:30	260	"	Start of
6:35	262	60	
6:40	260	"	
6:45	260	"	End of t

Test 3(run1):

Date = 6,5,90

Reaction temperature = 500°F (260°C)

Reaction time = 15 minutes

Weight of the untreated (raw) soil sample = 800g
weight of the treated soil residue and reaction flask = 1636 g
Weight of the treated soil residue = 635 g

Stirring rate = 116 rpm

TABLE A.5. Time, Temperature, and Heating Mantle Controller Setting During LTTD Test.

Time, minutes	Temperature, °C	Heating mantle controlle set point
0:00	20	60
0:15	40	〃
0:30	60	〃
0:45	75	〃
1:00	76	〃
1:15	82	〃
1:30	82	〃
1:45	82	〃
2:00	82	〃
2:15	85	〃

2:30	100	//	
2:45	110	//	
3:00	120	//	
3:15	130	//	
3:30	145	//	
3:45	175	//	
4:00	180	//	
4:15	195	//	
4:30	210	//	
4:45	230	//	
5:00	235	//	
5:15	235	//	
5:40	244	65	
5:55	250	//	
6:10	260	55	Start of
6:15	260	//	
6:20	260	//	
6:25	260	//	End of test.

Test 3 (run 2)

ORIGINAL
(Red)

Date = 6,5,90

Reaction temperature = 500°F (260°C)

Reaction time = 15 minutes

Weight of the untreated (raw) soil sample = 800g
Weight of the treated soil residue and reaction flask = 1674 g
Weight of the treated soil residue = 622 g

Stirring rate = 114 rpm

TABLE A-6. Time, Temperature, and Heating Mantle Controller Setting during LTTD Test.

Time, minutes	Temperature, °C	Heating Mantle Controller Set Point
0:00	20	60
0:15	40	"
0:30	60	"
0:45	80	"
1:00	82	"
1:15	82	"
1:30	84	"
1:45	90	"
2:00	92	"

2:15	94	60	
2:30	105	"	
2:45	125	"	
3:00	160	"	
3:15	170	"	
3:30	190	"	
3:45	216	"	
4:00	220	"	
4:15	240	55	
4:30	246	"	
4:40	260	50	Start o testin
4:45	265	"	
4:50	270	"	
4:55	260	"	End of

Test 4 (run 1)

Date = 6/6/90

Reaction temperature = 500°F (260°C)

Reaction time = 30 minutes

Weight of the untreated (raw) soil sample = 800 g
Weight of the treated soil residue and reaction flask = 1636 g
Weight of the treated soil residue = 635 g

Stirring rate = 115 rpm

TABLE A-7. Time, Temperature, and Heating Mantle Controller Setting during LTTD Test.

Time, minutes	Temperature, °C	Heating Mantle Controller Set Point
0:00	23	60
0:15	44	〃
0:30	75	〃
0:45	90	〃
1:00	90	〃
1:15	90	〃
1:30	90	〃
1:45	90	〃
2:00	90	〃

2:15	90	"
2:30	90	"
2:45	95	"
3:00	95	"
3:15	102	"
3:30	108	"
3:45	125	"
4:00	140	"
4:15	170	"
4:30	185	"
4:45	210	"
5:00	230	"
5:15	240	"
5:30	250	"
5:37	260	55 Start of tes
5:42	261	"
5:47	260	"
5:52	260	"
5:57	261	"
6:02	260	"
6:07	260	End of testing

ORIGINAL (Red)

Test 4 (run 2)

Date = 6/6/90

Reaction temperature = 500°F (260°C)

Reaction time = 30 minutes

Weight of the untreated (raw) soil sample = 800 g
weight of the treated soil residue and reaction flask = 1682 g
Weight of the treated soil residue = 632 g

Stirring rate = 115 rpm

TABLE A.8. Time, Temperature, and Heating Mantle Controller Sets during LTTD Test.

Time, minute	Temperature, °C	Heating mantle controller set point
0:00	23	60
0:15	49	"
0:30	65	"
0:45	80	"
1:00	80	"
1:15	80	"
1:30	85	"
1:45	95	"
2:00	95	"
2:15	95	"

2:30	95	"
2:45	95	"
3:00	106	"
3:15	125	"
3:30	145	"
3:45	165	"
4:00	201	"
4:15	216	"
4:30	230	"
4:45	242	65
5:00	250	"
5:12	260	" Start of
5:17	250	"
5:22	262	"
5:27	263	"
5:32	260	"
5:37	260	"
5:42	260	" End of Tes

Test 5 (run)

Date = 6/7/90

Reaction temperature = 500°F (260°C)

Reaction time = 30 minutes

Weight of the untreated (raw) soil sample = 800g

Weight of the treated soil residue and reaction flask = 1579

Weight of the treated soil residue = 578g

Stirring rate = 115 rpm

TABLE. A-9. Time, Temperature, and Heating Mantle Controller Setting During LTTD Test

Time, minute	Temperature, °C	Heating Mantle Controller Set Point
0:00	21	65
0:15	25	〃
0:30	50	〃
0:45	60	〃
1:00	80	〃
1:15	82	〃
1:30	82	〃
1:45	82	〃
2:00	82	〃

2:15	88	〃	
2:30	91	〃	
2:45	95	〃	
3:00	125	〃	
3:15	155	〃	
3:30	195	〃	
3:45	204	〃	
4:00	216	60	
4:15	228	〃	
4:30	252	〃	
4:32	260	〃	Start of test
4:37	260	〃	
4:42	260	〃	
4:47	262	〃	
4:52	263	〃	
4:57	263	〃	
5:02	263	〃	End of testi

Test 5 (run 2)

Date = 6/7/90

Reaction temperature = 500°F (260°C)

Reaction time = 30

Weight of the untreated (raw) soil sample = 800 g
Weight of the treated soil residue and reaction flask = 1634 g
weight of the treated soil residue = 584 g

Stirring rate = 115 rpm

TABLE A-10. Time, Temperature, and Heating Mantle Controller Setting during LTTD Test

Time, minute	Temperature, °C	Heating Mantle Controller Set Point
0:00	21	65
0:15	35	〃
0:30	70	〃
0:45	85	〃
1:00	85	〃
1:15	85	〃
1:30	85	〃
1:45	85	〃
2:00	85	〃
2:15	120	〃

2:30	155	〃	
2:45	188	60	
3:00	210	〃	
3:15	245	〃	
3:30	250	〃	
3:40	260	〃	Start of testi
3:45	263	〃	
3:50	263	〃	
3:55	263	55	
4:00	263	〃	
4:05	261	〃	
4:10	260	〃	End of testin

ORIGINAL

Test 6 (run 1)

Date = 6/8/90

Reaction temperature = 300°F (149°C)

Reaction time = 30 minutes

weight of the untreated (raw) soil sample = 800 g
weight of the treated soil residue and reaction flask = 1624 g
Weight of the treated soil residue = 622 g

Stirring rate = 115 rpm

TABLE A-11. Time, Temperature, and Heating Mantle Controller Setting during LTTD Test.

Time, minute	Temperatures °C	Heating Mantle Controller Set Point
0:00	25	65
0:15	40	″
0:30	80	″
0:45	95	″
1:00	95	″
1:15	95	″
1:30	95	″
1:45	95	″

2:00	95	"	
2:15	95	"	
2:30	120	60	
2:45	150	55	Start of testing
2:50	160	50	
2:55	160	40	
3:00	160	30	
3:05	160	"	
3:10	160	"	
3:15	160	"	End of testing

Test 6 (run 2)

[illegible]

Date = 6/8/90

Reaction temperature = 300°F (149°C)

Reaction time = 30 minutes

Weight of the untreated (raw) soil sample = 800 g
Weight of the treated soil residue and reaction flask = 1684 g
Weight of the treated soil residue = 631 g

Stirring rate = 115 rpm

TABLE A-12. Time, Temperature, and Heating Mantle Controller Setting for Soil Sample During LTTD Test

Time, minute	Temperature, °C	Heating Mantle Controller Set Point
0:00	25	65
0:15	40	"
0:30	70	"
0:45	90	"
1:00	95	"
1:15	100	"
1:30	100	"
1:45	100	"
2:00	105	"

..	2:15	125	60	Start of Testin
..	2:30	150	55	
..	2:35	150	"	
..	2:40	150	"	
..	2:45	155	"	
..	2:50	160	50	
..	2:55	160	40	
..	3:00	160	"	End of testing
..				
..				
..				

Test 7 (run 1)

Date = 6/8/90

Reaction temperature = 300° F (149°C)

Reaction time = 30 minutes

Weight of the treated (raw) soil sample = 800g
weight of the treated soil residue and reaction flask = 1641 g
weight of the treated soil residue = 639 g

Stirring rate = 115 rpm

TABLE A-13. Time, Temperature, and Heating Mantle Controll

Setting for the Soil Sample During LTTD Test

Time, minute	Temperatures °C	Heating Mantle Controller Set Poi
0:00	25	65
0:15	65	"
0:30	90	60
0:45	95	"
1:00	95	"
1:15	95	"
1:30	95	"
1:45	95	"
2:00	110	50

2:15	125	"	
2:30	150	"	Start of tes
2:35	155	40	
2:40	155	"	
2:45	155	"	
2:50	152	"	
2:55	150	"	
3:00	150	"	End of testin

Test 7 (run 2)

Date = 6/8/90

Reaction temperature = 300°F (149°C)

Reaction time = 30 minutes

Weight of the untreated (raw) soil sample = 800 g
Weight of the treated soil residue and reaction flask = 1684 g
Weight of the treated soil residue = 631 g

Stirring rate = 115 rpm

TABLE A-14. Time, Temperature, and Heating Mantle Controller Setting for the Soil Sample During LTTD Test

Time, minute	Temperature, °C	Heating Mantle Controller Setting Poi
0:00	32	65
0:15	80	〃
0:30	100	60
0:45	100	〃
1:00	100	〃
1:15	100	〃
1:30	105	〃
1:45	135	45

2:00	135	50	
2:15	145	〃	
2:24	149	〃	Start of test
2:29	150	〃	
2:34	155	45	
2:39	155	40	
2:44	160	35	
2:49	158	〃	
2:54	155	〃	End of testing

Test 8 (run 1) -

Date = 6/11/90.

Reaction temperature = 500°F (260°C)

Reaction time = 30 minutes

Weight of the untreated (raw) soil sample = 800 g
Weight of the treated soil residue and reaction flask = 1620 g
Weight of the treated soil residue = 628 g

Stirring rate = 115 rpm

TABLE A.15. Time, Temperature, and Heating Mantle Controller Setting for the Soil Sample During LTTD Test

Time, minute	Temperature, °C	Heating Mantle Controller Set Point
0:00	28	65
0:15	40	"
0:30	55	"
0:45	95	"
1:00	100	"
1:15	100	"
1:30	105	"
1:45	115	"
2:00	128	"

2:15	170	60	
2:30	205	"	
2:45	205	"	
3:00	235	"	
3:10	260	"	Start of test
3:15	265	55	
3:20	270	"	
3:25	270	45	
3:30	270	"	
3:35	265	"	
3:40	265	"	End of testing

Test 8 (run 2)

Date: 6/11/90

Reaction temperature = 500°F (260°C)

Reaction time = 30 minutes

Weight of the untreated (raw) soil sample = 800 g
Weight of the treated soil residue and reaction flask = 1650 g
Weight of the treated soil residue = 597 g

Stirring rate = 115

TABLE A-16 Time, Temperature, and Heating Mantle Controlle

Setting for the Soil Sample During LTTD Test

Time, minute	Temperature, °C	Heating Mantle Contoller Set Point
0:00	25	65
0:15	55	"
0:30	80	"
0:45	90	"
1:00	100	"
1:15	100	"
1:30	100	"
1:45	102	"

2100	102	〃	
2115	135	〃	
2130	200	60	
2145	205	〃	
3:00	240	〃	
3:12	260	〃	Start of testin
3:17	263	〃	
3:22	265	55	
3:27	267	50	
3:32	265	〃	
3:37	263	〃	
3:42	260	〃	End of testing

Test 9 (run 1)

Date = 6/12/90

Reaction temperature = 500° F (260°C)

Reaction time = 30 minutes

Weight of the untreated (raw) soil sample = 800 g
Weight of the treated soil residue and reaction flask = 1642 g
Weight of the treated soil residue = 640 g

Stirring rate = 110

TABLE A-17 Time, Temperature, and Heating Mantle Controller Setting for the Soil Sample During LTTD Test

Time, minute	Temperature, °C	Heating Mantle Controller Set Point
0:00	28	65
0:15	55	〃
0:30	90	〃
0:45	90	〃
1:00	100	〃
1:15	100	〃
1:30	100	〃
1:45	102	〃
2:00	105	〃

2:15	105	"	
2:30	115	"	
2:45	135	"	
3:00	150	"	
3:15	170	"	
3:30	195	"	
3:45	220	60	
4:00	235	"	
4:15	245	65	
4:24	260	60	Start of testing
4:29	267	55	
4:34	268	"	
4:39	265	"	
4:44	262	"	
4:49	260	"	
4:54	260	"	End of testing

Test 9 (run 2).

Date = 6/12/90

Reaction temperature = 500°F (260°C).

Reaction time = 30

Weight of the untreated (raw) soil sample = 800g

Weight of the treated soil residue and reaction flask = 1685 g

Weight of the treated soil residue = 632 g

Stirring rate = 110 rpm

TABLE A-18. Time, Temperature, and Heating Mantle Controll Setting for the Soil Sample During LTTD Test

Time, minute	Temperature °C	Heating Mantle Controller Set Point
0:00	23	65
0:15	50	″
0:30	70	″
0:45	85	″
1:00	100	″
1:15	100	″
1:30	100	″
1:45	100	″
2:00	102	″

2:15	110	"
2:30	115	"
2:45	140	"
3:00	155	"
3:15	165	"
3:30	210	60
3:45	230	"
4:00	240	65
4:13	260	60 Start of testin
4:18	263	55
4:23	265	"
4:28	262	"
4:33	261	"
4:38	260	"
4:43	260	" End of testing

Test 10 (run 1)

Date = 6/13/90

Reaction temperature = 500°F (260°C)

Reaction time = 30 minutes

Weight of the untreated (raw) soil sample = 800
Weight of the treated soil residue and reaction flask = 1580 g
Weight of the treated soil residue = 577 g

Stirring rate = 115 rpm

TABLE A-19. Time, Temperature, and Heating Mantle Controller Setting for the Soil Sample During LTTD Test.

Time, minute	Temperature, °C	Heating mantle controller set point
0:00	21	65
0:15	55	"
0:30	92	"
0:45	99	"
1:00	100	"
1:15	100	"
1:30	100	"
1:45	102	"
2:00	105	"

2:15	105	"	
2:30	115	"	
2:45	140	"	
3:00	155	"	
3:15	180	"	
3:30	205	"	
3:45	225	60	
4:00	250	"	
4:10	262	55	Start of testin
4:15	265	"	
4:20	268	50	
4:25	264	"	
4:30	262	"	
4:35	262	"	
4:40	261	"	End of testing

Test 10 (run 2)

Date = 6/13/90

Reaction temperature = 500°F (260°C)

Reaction time = 30 minutes

Weight of the untreated (raw) soil sample = 800 g

Weight of the treated soil residue and reaction flask = 1650 g

Weight of the treated soil residue = 597 g

Stirring rate = 115 rpm

TABLE A-20 Time, Temperature, and Heating Mantle Controller Setting for the Soil Sample During LTTD Test.

Time, minute	Temperature, °C	Heating mantle controller set point
0:00	21	65
0:15	55	〃
0:30	82	〃
0:45	91	〃
1:00	100	〃
1:15	100	〃
1:30	100	〃
1:45	102	〃
2:00	102	〃

2:15	132	"	
2:30	180	"	
2:45	204	"	
3:00	234	"	
3:15	245	60	
3:30	262	"	Start of testing
3:35	264	55	
3:40	264	50	
3:45	262	"	
3:50	260	"	
3:55	260	"	
4:00	260	"	End of testing

Test 11 (run 1)

Date = 6/14/90

Reaction temperature = 500°F (260°C)

Reaction time = 30 minutes

Weight of the untreated (raw) soil sample = 800g

Weight of the treated soil residue and reaction flask = 1609 g

Weight of the treated soil residue = 605 g

Stirring rate = 115 rpm

TABLE A-21 Time, Temperature, and Heating Mantle Controller Setting for the Soil Sample During LTTD Test.

Time, minute	Temperature, °C	Heating mantle controller set point
0:00	28	65
0:15	56	〃
0:30	82	〃
0:45	92	〃
1:00	100	〃
1:15	100	〃
1:30	100	〃
1:45	100	〃
2:00	100	〃

2:15	100	"	
2:30	100	"	
2:45	100	"	
3:00	110	"	
3:15	136	"	
3:30	150	"	
3:45	165	"	
4:00	225	"	
4:15	250	62	
4:20	265	"	Start of testing
4:25	262	"	
4:30	262	"	
4:35	260	"	
4:40	260	"	
4:45	260	"	
4:50	260	"	End of testing

Test 11 (run 2)

Date = 6/14/90

Reaction temperature = 500° F (260°C)

Reaction time = 30 minutes

Weight of the untreated (raw) soil sample = 800 g
Weight of the treated soil residue and reaction flask = 1687
Weight of the treated soil residue = 634 g

Stirring rate = 115 rpm

TABLE A.22. Time, Temperature and Heating Mantle Controller Setting for the Soil Sample During LTTD Test.

Time, minute	Temperature, °C	Heating mantle contr set point
0:00	27	65
0:15	58	〃
0:30	80	〃
0:45	94	〃
1:00	100	〃
1:15	100	〃
1:30	102	〃
1:45	102	〃
2:00	102	〃

2:15	102	"	
2:30	102	"	
2:45	102	"	
3:00	102	"	
3:15	114	"	
3:30	128	"	
3:45	142	"	
4:00	170	"	
4:15	200	"	
4:30	235	"	
4:45	245	"	
4:50	260	"	Start of testing
4:55	268	"	
5:00	268	60	
5:05	266	55	
5:10	264	"	
5:15	262	"	
5:20	260	"	End of testing

Test 12 (run 1)

Date = 6/15/90

Reaction temperature = 500°F (260°C)

Reaction time = 30 minutes

Weight of the untreated (raw) soil sample = 800 g

Weight of the treated soil residue and reaction flask = 1621.

Weight of the treated soil residue = 618 g

Stirring rate = 115 rpm

TABLE A.23. Time, Temperature, and Heating Mantle Controller Setting for the Soil Sample During LTTD Test

Time, minute	Temperature, °C	Heating mantle contr set point
0:00	28	65
0:15	50	〃
0:30	62	〃
0:45	68	〃
1:00	78	〃
1:15	82	〃
1:30	84	〃
1:45	90	〃
2:00	90	〃

2:15	105	"	
2:30	115	"	
2:45	118	"	
3:00	157	"	
3:15	185	"	
3:30	210	"	
3:45	228	"	
4:00	254	"	
4:05	264	60	Start of testing
4:10	268	55	
4:15	266	"	
4:20	260	60	
4:25	262	"	
4:30	264	55	
4:35	260	"	End of Testing

Test 12 (run 2)

Date = 6/15/90

Reaction temperature = 500°F (260°C)

Reaction time = 30 minutes

Weight of the untreated (raw) soil sample = 800g

weight of the treated soil residue and reaction flask = 1668

weight of the treated soil residue = 615 g

Stirring rate = 115 rpm

TABLE A-24. Time, Temperature, and Heating Mantle Controller

Setting for the Soil Sample During LTTD Test.

Time, minute	Temperature °C	Heating Mantle Contr Set Point
0:00	24	65
0:15	50	"
0:30	70	"
0:45	84	"
1:00	88	"
1:15	90	"
1:30	90	"
1:45	92	"
2:00	92	

2:15	95	〃	
2:30	110	〃	
2:45	115	〃	
3:00	150	〃	
3:15	175	〃	
3:30	205	〃	
3:45	225	〃	
4:00	245	70	
4:15	264	〃	Start of testing
4:20	266	〃	
4:25	264	65	
4:30	262	55	
4:35	262	〃	
4:40	262	〃	
4:45	261	〃	End of testing

Test 13 (run 1)

Date = 6/18/90

Reaction temperature = 500°F (260°C)

Reaction time = 30 minutes

Weight of the untreated (raw) soil sample = 800 g
Weight of the treated soil residue and reaction flask = 1612 g
Weight of treated soil residue = 610 g

Stirring rate = 120 rpm

TABLE A-25. Time, Temperature, and Heating Mantle Contr

Setting for the Soil Sample During LTTD Test.

Time, minute	Temperature °C	Heating mantle control set point
0:00	26	65
0:15	55	"
0:30	64	"
0:45	74	"
1:00	80	"
1:15	80	"
1:30	81	"
1:45	82	

2:00	96	〃
2:15	102	〃
2:30	138	〃
2:45	152	〃
3:00	178	〃
3:15	195	〃
3:30	225	〃
3:45	235	〃
4:00	245	〃
4:15	252	〃
4:20	264	60 Start of testing
4:25	266	55
4:30	266	〃
4:35	264	〃
4:40	264	〃
4:45	262	〃
4:50	262	〃 End of testing

Test 13 (run 2)

Date: 6/18/90

Reaction temperature = 500°F (260°C)

Reaction time = 30 minutes

Weight of untreated (raw) soil sample = 800 g
Weight of the treated soil residue and reaction flask = 164
Weight of treated soil residue = 598 g

Stirring rate = 120 rpm

TABLE A.26. Time, Temperature, and Heating Mantle Controller Setting for the Soil Sample During LTTD Te

Time, minute	Temperature °C	Heating mantle contro set point
0:00	20	65
0:15	52	"
0:30	64	"
0:45	84	"
1:00	86	"
1:15	86	"
1:30	85	"
1:45	86	"
2:00	88	"

2:15	92	"	
2:30	118	"	
2:45	138	"	
3:00	160	"	
3:15	190	"	
3:30	225	"	
3:45	230	"	
4:00	240	"	
4:10	262	60	Start of testing
4:15	264	"	
4:20	266	55	
4:25	266	50	
4:30	264	"	
4:35	260	"	
4:40	260	"	End of testing

Test 14 (run 1)

Date = 6/19/90

Reaction temperature = 500°F (260°C)

Reaction time = 30 minutes

Weight of the untreated (raw) soil sample = 800 g
weight of the treated soil residue and reaction flask = 1621. g
Weight of the treated soil residue = 618 g

Stirring rate = 112 rpm

TABLE A.27. Time, Temperature, and Heating Mantle Controller Setting for the Soil Sample During LTTD Test

Time, minute	Temperature, °C	Heating mantle controlle set point
0:00	24	65
0:15	54	"
0:30	90	"
0:45	95	"
1:00	95	"
1:15	95	"
1:30	95	"
1:45	95	"
2:00	100	"

2:15	110	"
2:30	125	"
2:45	140	"
3:00	160	"
3:15	180	"
3:30	185	"
3:45	205	"
4:00	225	"
4:15	255	"
4:20	262	" Start of testing
4:25	268	60
4:30	268	"
4:35	264	"
4:40	264	55
4:45	262	"
4:50	262	" End of Testing

Test 14 (run 2)

Date = 6,19,90

Reaction temperature = 500°F (260°C)

Reaction time = 30 minutes

Weight of the untreated (raw) soil sample = 800g
Weight of the treated soil residue and reaction flask = 167
Weight of treated residue = 625 g

Stirring rate = 115 rpm

TABLE A.28; Time, Temperature, and Heating Mantle Contro

Setting for the Soil Sample During LTTD Test

Time, minute	Temperature, °C	Heating mantle controlle set point
0:00	24	65
0.15	52	〃
0:30	80	〃
0:45	90	〃
1:00	95	〃
1:15	95	〃
1:30	95	〃
1:45	95	〃
2:00	95	〃

2:15	102	〃	
2:30	120	〃	
2:45	140	〃	
3:00	165	〃	
3:15	180	〃	
3:30	192	〃	
3:45	225	〃	
4:00	240	〃	
4:15	245	〃	
4:30	245	〃	
4:35	262	60	Start of testing
4:40	266	55	
4:45	264	〃	
4:50	264	〃	
4:55	262	〃	
5:00	262	〃	
5:05	260	〃	End of testing

APPENDIX A-4

TESTING DATA MANAGEMENT SHEETS FOR SOLIDIFICATION/STABILIZATION STUDIES

1
9/25/90

LABORATORY MEASUREMENTS FOR SOLIDIFICATION AND STABILIZATION OF SOIL SAMPLES

Laboratory measurements for preparation of

asphalt-based solidified samples are as follows

Set 1

binder-To-soil ratio = 1:1

Weight of soil residue in Reaction Flask No.1 = 500g

Temperature of soil residue in Reaction Flask No.1 =

Mixing duration of soil = 10 minutes

Weight of asphalt in Reaction Flask No. 2 = 1200g

Temperature of asphalt in Reaction Flask No. 2 = 14

For preparation of solidified samples with B/S of 1:1

500 g asphalt was added to 500g soil in Reaction Flas

Mixing duration of soil and asphalt = 10 minutes

Temperature of soil and asphalt mix = 140°C

Size of solidified samples prepared for UCS Tests = 3.8 cm diameter by

cm long

Number of solidified samples prepared for UCS test

Size of solidified samples prepared for TCLP tests = 4.5 cm diamete

by 10 cm long

Number of solidified samples prepared for TCLP tests = 2

Set 2

Binder-to-soil ratio = 0.5:1

Weight of soil residue in Reaction Flask No.1 = 500g

Temperature of soil residue in Reaction Flask No.1 = 6

Mixing duration of soil = 10 minutes

Weight of asphalt in Reaction Flask No.2 = 654 g

Temperature of asphalt in Reaction Flask No.2 = 1

For preparation of solidified samples with B/S o

0.5:1, 250g asphalt was added to the 500 g soi

Reaction Flask No.1

Mixing duration of soil and asphalt = 10 minutes

temperature of soil and asphalt mix = 137°C

Size of solidified samples for UCS tests = 3.8 cm diam

by 8.4 cm long

Number of solidified samples for UCS tests = 2

Size of solidified samples for TCLP tests = 4.5 cm diameter by 10 cm long

Number of solidified samples for TCLP tests = 2

Laboratory measurements for preparation of blank asphalt based solidified samples are as follo

Set 1

Binder-to-soil ratio = 1:1

Weight of sand in Reaction Flask No.1 = 200 g

Temperature of sand in Reaction Flask No.1 = 63°C

Mixing duration of sand = 10 minutes

Weight of asphalt in Reaction Flask No.2 = 500 g

Temperature of asphalt in Reaction Flask No.2 = 138

For preparation of blank solidified sample with B/S of 1:1, 200g asphalt was added to the sand in R.F.

Mixing duration of sand and asphalt = 10 minutes

Temperature of sand and asphalt mix = 139°C

Size of solidified samples prepared for TCLP tests = 4.5 cm dia by 10 cm long

Number of solidified samples prepared for TCLP tests = 1

Set 2

Binder-to-soil ratio = 0.5 : 1

Weight of sand in Reaction Flask No. 1 = 300 g

Temperature of sand in Reaction Flask No. 1 = 63°C

Mixing duration of sand = 10 minutes

Weight of asphalt in Reaction Flask No. 2 = 500 g

Temperature of asphalt in Reaction Flask No. 2 = 135°C

For preparation of a blank solidified sample with B/S of 0.5:1, 150 g asphalt was added to the sand in R.F No. 1.

Mixing duration of sand and asphalt = 10 minutes

Temperature of sand and asphalt mix = 135°C

Size of blank solidified samples prepared for TCLP tests = 4.5 cm diameter by 10 cm long

Number of blank solidified samples prepared for TCLP tests = 1

Laboratory measurements for preparation of cemen

based thermally treated solidified samples are as foll

Set 1

~~Binder to soil ratio = 0.40~~

Volume of water in Reaction Flask No. 1 = 280 ml.

Weight of soil in Reaction Flask No. 2 = 800 g

Mixing duration of soil = 10 minutes

Weight of portland cement (Type II) added to soil in Reactio

Flask No. 2 = 320 g

Mixing duration of soil and cement = 10 minutes

Mixing duration of soil, cement and water = 10 minu.

Size of solidified samples prepared for UCS tests = 7

diameter by 14.22 cm long

Number of solidified samples prepared for UCS tests

Size of solidified samples prepare[illegible]sts

9

Number of solidified samples prepared for TCLP tests = 2

Set 2

Binder-to-soil ratio = 0.25

Volume of water in Reaction Flask No. 1 = 250 ml

Weight of soil in Reaction Flask No. 2 = 800 g

Mixing duration of soil = 10 minutes

Weight of portland cement added to soil in React

Flask No. 2 = 200 g

Mixing duration of soil and cement = 10 minutes

Mixing duration of soil, cement and water = 10 mi

Size of solidified samples prepared for UCS Tes

7cm diameter by 14.22 cm long

Number of solidified samples prepared for UCS tes

Size of solidified samples prepared for TCLP te

4.5 cm diameter by 10 cm long

Number of solidified samples prepared for TCLP T

Laboratory measurements for preparation of blank cement based solidified samples are as follows:

Set 1

~~Binder-to-soil ratio = 0.40~~

Volume of water in Reaction Flask No. 1 = 280 ml

Weight of sand in Reaction Flask No. 2 = 800 g

Mixing duration of sand = 10 minutes

Weight of portland cement (Type II) added to sand = 320 g

Mixing duration of sand and cement = 10 minutes

Mixing duration of sand, cement, and water = 10 minutes

Size of solidified samples prepared for TCLP tests = 4.5 cm diameter by 10 cm long

Number of solidified samples prepared for TCLP tests = 1

Set 2

Binder - to - soil ratio = 0.25

Volume of water in Reaction Flask No. 1 = 250 ml

Weight of sand in Reaction Flask No. 2 = 800g.

Mixing duration of sand = 10 minutes

Weight of portland cement added to sand = 200g

Mixing duration of sand and cement = 10 minutes

Mixing duration of sand, cement, and water = 10 minu

Size of solidified samples prepared for TCL tes

4.5 cm diameter by 10 Cm long

Number of solidified samples prepared for TCL

tests = 1

Laboratory measurements for preparation of cement based raw solidified samples are as follows:

Set 1

~~Binder-to-soil ratio = 0.20~~

Volume of water in Reaction Flask No. 1 = 24 ml

Weight of soil in Reaction Flask No. 2 = 800 g

Moisture content of soil in Reaction Flask No. 2 = 31 perce

Mixing duration of soil = 10 minutes

Weight of portland cement added to soil in Reaction Flask No. 2 = 244.3 g

Mixing duration of soil and cement = 10 minutes

Mixing duration of soil, cement and water = 10 minu

Size of solidified samples prepared for UCS tests = 7 cm diameter by 14.22 cm long

Number of solidified samples prepared for UCS tests =

Size of solidified samples prepared for TCLP tests = 4.5cm diameter

10 cm long

Number of solidified samples prepared for TCLP tests =

Set 2

Binder-to-soil ratio = 0.25

Volume of water in Reaction Flask No.1 = 1.5 ml

Weight of soil in Reaction Flask No.2 = 800g

Moisture content of soil in Reaction Flask No.2 = 31 percen

Mixing duration of soil = 10 minutes

Weight of portland cement added to soil in Reaction Flask No.2 = 152.7 g

Mixing duration of soil and cement = 10 minutes

Mixing duration of soil, cement and water = 10 minute

Size of solidified samples prepared for UCS tests 7 cm diameter by 14.22 cm long

Number of solidified samples prepared for UCS tests = 2

Size of solidified samples prepared for TCLP tests =

Number of solidified samples prepared for TC[illegible]P Te

APPENDIX B

STANDARD TEST AND OPERATING PROCEDURES FOR TREATABILITY TESTING

CONTENTS

APPENDIX B

APPENDIX B-1

STANDARD TEST METHODS USED FOR BIOLOGICAL STUDIES

distillate free of contact with the delivery tube and continue distillation during the last minute or two to cleanse the condenser and delivery tube. Dilute to 500 ml with ammonia-free water.

e. Ammonia determination: Determine the ammonia by the nesslerization method (Section 418B), the phenate method (Section 418C), or acidimetric method (Section 418D).

418 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 ammonia nitrogen concentrations exceeding 20 μg/l. Apply the direct nesslerization method to domestic wastewaters only when errors of 1 to 2 mg/l are acceptable. 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. The addition of EDTA or Rochelle salt solution inhibits the precipitation of residual calcium and magnesium ions in the presence of the alkaline nessler reagent. However, the use of EDTA demands an extra amount of nessler reagent to insure a sufficient nessler 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 (20 to 250 μg/50 ml) 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 range of 5 to 60 μg. The reddish brown hues typical of ammonia nitrogen levels approaching 500 μg may be measured in the wavelength region of 450 to 500 nm. A judicious selection of light path and wavelength thus enables 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, the calibration curve should be prepared 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 ammonia nitrogen. In direct nesslerization, this represents 20 μg/l. However, reproducibility below 5 μg 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.

2) *Filter photometer*, providing a light path of 1 cm or longer and equipped with a violet filter having maximum transmittance at 400 to 425 nm. A blue filter can be used for higher ammonia nitrogen concentrations.

3) *Nessler tubes*, matched, 50-ml, tall form.

b. pH meter, equipped with a high pH electrode.

3. Reagents

All the reagents listed in the Preliminary Distillation, Section 418A, except the borate buffer and absorbent solution, are required, plus the following. (Prepare all reagents with ammonia-free water.)

a. Zinc sulfate solution: Dissolve 100 g $ZnSO_4 \cdot 7H_2O$ and dilute to 1 l.

b. Stabilizer reagent: Use either EDTA or Rochelle salt to prevent calcium or magnesium precipitation in undistilled samples following the addition of the alkaline nessler reagent.

1) *EDTA reagent:* Dissolve 50 g disodium ethylenediamine tetraacetate dihydrate, also called (ethylenedinitrilo) tetraacetic acid disodium salt, in 60 ml water containing 10 g NaOH. If necessary, apply gentle heat to complete dissolution. Cool to room temperature and dilute to 100 ml.

2) *Rochelle salt solution:* Dissolve 50 g potassium sodium tartrate tetrahydrate, $KNaC_4H_4O_6 \cdot 4H_2O$, in 100 ml water. Remove ammonia usually present in the salt by boiling off 30 ml of solution. After cooling, dilute to 100 ml.

c. Nessler reagent: Dissolve 100 g HgI_2 and 70 g KI in a small quantity of water and add this mixture slowly, with stirring, to a cool solution of 160 g NaOH in 500 ml water. Dilute to 1 l. Store in rubber-stoppered pyrex glassware and out of sunlight to maintain reagent stability for periods up to a year under normal laboratory conditions. Check the reagent to make sure that it yields the characteristic color with 100 µg/l ammonia nitrogen within 10 min after addition and does not produce a precipitate with small amounts of ammonia within 2 hr. (CAUTION: *Toxic—take care to avoid ingestion.*)

d. Stock ammonium solution: Dissolve 3.819 g anhydrous NH_4Cl, dried at 100 C, in water, and dilute to 1,000 ml; 1.00 ml = 1.00 mg N = 1.22 mg NH_3.

e. Standard ammonium solution: Dilute 10.00 ml stock ammonium solution to 1,000 ml with water; 1.00 ml = 10.0 µg N = 12.2 µg NH_3.

f. Permanent color solutions:

1) *Potassium chloroplatinate solution:* Dissolve 2.0 g K_2PtCl_6 in 300 to 400 ml distilled water; add 100 ml conc HCl and dilute to 1 l.

2) *Cobaltous chloride solution:* Dissolve 12.0 g $CoCl_2 \cdot 6H_2O$ in 200 ml distilled water. Add 100 ml conc HCl and dilute to 1 l.

4. Procedure

a. Treatment of undistilled samples: If necessary, remove the residual chlorine of the sample with an equivalent amount of *N/70* dechlorinating agent. Add 1 ml $ZnSO_4$ solution to 100 ml sample and mix thoroughly. Add 0.4 to 0.5 ml NaOH solution to obtain a pH of 10.5, as determined with a pH meter and a high-pH glass electrode and mix thoroughly. Let the treated sample stand for a few minutes, whereupon a heavy flocculent precipitate should fall, leaving a clear and colorless supernate. Clarify by centrifuging or filtering. Pretest any filter paper used to be sure no ammonia is present as a contaminant. Do this by running ammonia-free water through and testing the filtrate by nesslerization. Filter the sample, discarding the first 25 ml filtrate.

b. Color development:

1) Undistilled samples—Use 50.0 ml sample or a portion diluted to 50.0 ml with ammonia-free water. If the undistilled portion contains sufficient concentrations of calcium, magnesium, or other ions that produce a turbidity or precipitate with nessler reagent, add 1 *drop (0.05 ml) EDTA reagent or 1 to 2* drops (0.05 to 0.1 ml) Rochelle salt solution. Mix well. Add 2.0 ml nessler reagent if EDTA reagent is used, or 1.0 ml nessler reagent if Rochelle salt is used.

2) Distilled samples—Neutralize the boric acid used for absorbing the ammonia distillate in one of two ways. Add *2 ml nessler reagent, an excess that* raises the alkalinity to the desired high level. Alternatively, neutralize the boric acid with NaOH before adding 1 ml nessler reagent.

3) Mix the samples by capping the nessler tubes with clean rubber stoppers (which have been washed thoroughly with ammonia-free water) and then inverting the tubes at least six times. Keep such experimental conditions as temperature and reaction time the same in the blank, samples, and standards. Let the reaction proceed for at least 10 min after addition of the nessler reagent. Read the color in the sample and in the standards. If the ammonia nitrogen is very low use a 30-min contact time for sample, blank, and standards. Measure the color either photometrically or visually as directed in ¶*c* or *d* below:

c. Photometric measurement: Measure the absorbance or transmittance in a spectrophotometer or a filter photometer. Prepare the calibration curve at the same temperature and reaction time *used for the samples. Make the trans*mittance readings against a reagent blank and run parallel checks frequently against standards in the nitrogen range of the samples. Redetermine the complete calibration curve for each new batch of nessler reagent.

For distilled samples, prepare the standard curve under the same conditions as the samples. *Distill the reagent* blank and appropriate standards—each diluted to 500 ml—in the same manner as the samples. Bring the 300 ml distillate and 50 ml boric acid absorbent to 500 ml and take a 50-ml portion for nesslerization.

d. Visual comparison: Compare the colors produced in the sample against *those of the ammonia standards.* Prepare temporary or permanent standards as directed below:

1) Temporary standards—Prepare a series of visual standards in nessler tubes by adding the following volumes of standard NH_4Cl solution and diluting to 50 ml with ammonia-free water: 0, 0.2, 0.4, 0.7, 1.0, 1.4, 1.7, 2.0, 2.5, 3.0, 3.5, 4.0, 4.5, 5.0, and 6.0 ml. Nesslerize the standards and the portions of distillate by adding 1.0 ml nessler reagent to each tube and mixing well.

2) Permanent standards—Measure into 50-ml nessler tubes the volumes of potassium chloroplatinate and cobaltous

TABLE 418:II. PREPARATION OF PERMANENT COLOR STANDARDS FOR VISUAL DETERMINATION OF AMMONIA NITROGEN

Value in Ammonia Nitrogen µg	Approximate Volume of Platinum Solution ml	Approximate Volume of Cobalt Solution ml
0	1.2	0.0
2	2.8	0.0
4	4.7	0.1
7	5.9	0.2
10	7.7	0.5
14	9.9	1.1
17	11.4	1.7
20	12.7	2.2
25	15.0	3.3
30	17.3	4.5
35	19.0	5.7
40	19.7	7.1
45	19.9	8.7
50	20.0	10.4
60	20.0	15.0

chloride solutions indicated in Table 418:II, dilute to the mark, and mix thoroughly. The values given in the table are *approximate*: actual equivalents of the ammonium standards will differ with the quality of the nessler reagent, the kind of illumination used, and the color sensitiveness of the analyst's eye. Therefore, compare the color standards with the nesslerized temporary ammonia standards and modify the tints as necessary. Make such comparisons for each newly prepared nessler reagent and satisfy each analyst as to the aptness of the color match. Protect the standards from dust to extend their usefulness for several months. Compare either 10 or 30 min after nesslerization, depending on the reaction time used in the preparation of the nesslerized ammonium standards against which they were matched.

5. Calculation

a. Deduct the amount of nitrogen in the ammonia-free water used for diluting the original sample before computing the final nitrogen value.

b. Deduct also the reagent blank for the volume of borate buffer and 6*N* NaOH solutions used with the sample.

c. Compute the total ammonia nitrogen by the following equation:

$$\text{mg/l ammonia N} = \frac{A}{\text{ml sample}} \times \frac{B}{C}$$

where A = µg N found colorimetrically, B = total distillate collected, including the acid absorbent, and C = ml distillate taken for nesslerization. The ratio B/C applies only to the distilled samples and should be ignored in direct nesslerization.

6. Precision and Accuracy

See Section 418A and Table 418:I.

423 D. Bibliography

Foulk, C. W. & A. T. Bawden. 1926. A new type of endpoint in electrometric titration and its application of iodimetry. *J. Amer. Chem. Soc.* 48:2045.

Hann, V. A. & T. C. Manley. 1952. Ozone. pp. 735-753 *in* Encyclopedia of Chemical Technology. Vol. 9. Interscience Publishers, New York, N.Y.

Birdsall, C. M., A. C. Jenkins & E. Spadinger. 1952. The iodometric determination of ozone. *Anal. Chem.* 24:662.

Zehender, F. & W. Stumm. 1953. Determination of ozone in drinking water (in German). *Mitt. Gebiete Lebensm. Hyg.* 44:206.

Ingols, R. S. & R. H. Fetner. 1956. Sterilization of water by ozone under arctic conditions. Rept. PB 124786, Office of Technical Services, U. S. Dept. Commerce, Washington, D. C.

Ingols, R. S., R. H. Fetner & W. H. Eberhardt. 1956. Determination of ozone in solution. *Proc. Int. Ozone Conf.*, American Chemical Society, Advan. Chem. Ser. No. 21.

Hann, V. A. 1956. Disinfection of drinking water with ozone. *J. Amer. Water Works Ass.* 48:1316

Ingols, R. S. & R. H. Fetner. 1957. Some studies of ozone for use in water treatment. *Proc. Soc. Water Treat. Exam.* 6:8.

424 pH VALUE

The pH of most natural waters falls within the range of 4 to 9. The majority of waters are slightly basic because of the presence of carbonates and bicarbonates. A departure from a normal pH for a given water could be caused by the influx of acidic or alkaline industrial wastes. Neutralization of spent acids or bases is an important waste treatment practice, and measurement and control of pH in industrial effluents is often required for water pollution control. It is also relatively common to practice pH adjustment of water treatment plant effluents to control corrosion in distribution systems.

The pH of a solution refers to its hydrogen ion activity and is expressed as the logarithm of the reciprocal of the hydrogen ion activity in moles per liter at a given temperature. It is used in the calculation of carbonate, bicarbonate, and carbon dioxide, corrosion and stability index, and other acid-base equilibria of importance to water and wastewater analysis and treatment control. The practical pH scale extends from 0, very acidic, to 14, very alkaline, with 7 corresponding to exact neutrality at 25 C. Whereas "alkalinity" and "acidity" are measures of the total resistance to pH change or buffering capacity of a sample, pH represents the free hydrogen ion activity not bound by carbonate or other bases.

The pH can be measured either colorimetrically or electrometrically. The colorimetric method is less expensive but suffers from interferences due to color, turbidity, salinity, colloidal matter, and various oxidants and reductants. The indicators are subject to deterioration as are the color standards with which they are compared. Moreover, no single indicator encompasses the pH range of interest in waters and wastewaters. In poorly buffered liquids, the indicators themselves may alter the pH of the sample unless preadjusted to nearly the same pH as the sample. For these reasons, the colorimetric method is suitable only for rough estimation and is not described herein. (For details on the colorimetric method, see Clark,[1] Kolthoff,[2] and AWWA.[3]) The glass electrode method is the standard technic.

1. General Discussion

a. Principle: Although the hydrogen electrode is recognized as the primary standard, the glass electrode is less subject to interferences and is used in combination with a calomel reference electrode. The glass-reference electrode pair produces a change of 59.1 mV/pH unit at 25 C.

b. Interferences: The glass electrode is relatively free from interference from color, turbidity, colloidal matter, oxidants, reductants, or high salinity, except for a sodium error at high pH. This error at a pH above 10 may be reduced by using special "low sodium error" electrodes. When using ordinary glass electrodes, make approximate corrections for the sodium error in accordance with information supplied by the manufacturer. Temperature exerts two significant effects on pH measurement: the pH potential, i.e., the change in potential per pH unit, varies with temperature; and ionization in the sample also varies.* The first effect can be overcome by a temperature compensation adjustment provided on the better commercial instruments. The second effect is inherent in the sample and is taken into consideration by recording both temperature and pH of each sample.

2. Apparatus

a. Electronic pH meter with temperature compensation adjustment.

b. Glass electrode: Glass electrodes are available for measurement over the entire pH range with minimum-sodium-ion-error types for high pH-high sodium samples.

c. Reference electrode: Use a calomel, silver-silver chloride, or other constant-potential electrode.

d. Magnetic stirrer, with teflon-coated stirring bar or a mechanical stirrer with inert plastic-coated or glass impeller.

e. Flow chamber for measurement of continuously flowing or unbuffered solutions.

3. Standard Solutions

a. General preparation: Calibrate the electrode system against standard buffer solutions of known pH. Because buffer solutions may deteriorate as a result of mold growth or contamination, prepare fresh as needed for accurate work by weighing the amounts of chemicals specified in Table 424:I, dissolving in dis-

---

* This ionization, dependent on values of the ionization constants for the various weak acids and bases in the sample at a particular temperature, is to a significant extent related to the alkalinity. Increasing alkalinity reduces the effect of temperature change on the pH. This effect of alkalinity is not a direct relationship but it can be quite pronounced even at very low concentrations of alkalinity.

The temperature dial on pH meters is designed only to correct for the temperature characteristics of the electrodes. Instruments without a temperature dial are often provided with data from which this correction for the characteristics of the electrodes may be calculated.

Data for calculating, by interpolation, the pH of natural waters at temperatures other than that of the measurement have been provided by Langelier.[4]

TABLE 424:I. PREPARATION OF pH STANDARD SOLUTIONS

Standard Solution (molality)	pH at 25 C	Weight of Chemicals Needed/1,000 ml Aqueous Solution at 25 C
Primary standards:		
Potassium hydrogen tartrate (saturated at 25 C)	3.557	6.4 g $KHC_4H_4O_6$*
0.05 potassium dihydrogen citrate	3.776	11.41 g $KH_2C_6H_5O_7$
0.05 potassium hydrogen phthalate	4.008	10.12 g $KHC_8H_4O_4$
0.025 potassium dihydrogen phosphate+0.025 disodium hydrogen phosphate	6.865	3.388 g KH_2PO_4†+3.533 g Na_2HPO_4†‡
0.008695 potassium dihydrogen phosphate+0.03043 disodium hydrogen phosphate	7.413	1.179 g KH_2PO_4†+4.302 g Na_2HPO_4†‡
0.01 sodium borate decahydrate (borax)	9.180	3.80 g $Na_2B_4O_7 \cdot 10H_2O$‡
0.025 sodium bicarbonate+0.025 sodium carbonate	10.012	2.092 g $NaHCO_3$+2.640 g Na_2CO_3
Secondary standards:		
0.05 potassium tetroxalate dihydrate	1.679	12.61 g $KH_3C_4O_8 \cdot 2H_2O$
Calcium hydroxide (saturated at 25 C)	12.454	1.5 g $Ca(OH)_2$*

* Approximate solubility.

† Dry chemical at 110 to 130 C for 2 hr.

‡ Prepare with freshly boiled and cooled distilled water (carbon-dioxide-free).

tilled water at 25 C, and diluting to 1,000 ml. This procedure is particularly important for the borate and carbonate buffers.

Use distilled water having a conductivity of less than 2 μsiemens at 25 C and a pH 5.6 to 6.0 for the preparation of all standard solutions. Freshly boil and cool this distilled water to expel the carbon dioxide to produce a pH of 6.7 to 7.3 for the preparation of the borate and phosphate solutions. Dry the potassium dihydrogen phosphate at 110 C to 130 C for 2 hr before weighing. Do not heat the unstable hydrated potassium tetroxalate above 60 C nor dry the other specified buffer salts.

Although ACS-grade chemicals are generally satisfactory for the preparation of buffer solutions, use certified materials available as NBS standard samples from the National Bureau of Standards where the greatest accuracy is required. For routine analysis, commercially available buffer tablets, powders, or solutions of tested quality also are permissible. In preparing buffer solutions from solid salts, dissolve all the material; otherwise, the pH calibration will be incorrect. Prepare and calibrate the electrode system with buffer solutions with pH approximating that of the sample to minimize error resulting from nonlinear response of the electrode.

As a rule, select and prepare the buffer solutions classed as primary standards in Table 424:I; reserve the secondary standards for extreme situations

ORIGINAL
(Red)

encountered in wastewater measurements. Consult Table 424:II for the accepted pH of the standard buffer solutions at temperatures other than 25 C. Where the intent is to apply them for routine control, store the buffer solutions and samples preferably in polyethylene bottles or, at least, pyrex glassware. Even in such circumstances, replace buffer solutions every 4 wk.

b. Saturated potassium hydrogen tartrate solution: Shake vigorously an excess (5 to 10 g) of finely crystalline $KHC_4H_4O_6$ with 100 to 300 ml distilled water at 25 C in a glass-stoppered bottle. Separate the clear solution from the undissolved material by decantation or filtration. If this solution is to be used for routine control, preserve for 2 months or more by adding a thymol crystal (8 mm diam) for each 200 ml solution.

c. Saturated calcium hydroxide solution: Place the well-washed, low-alkali-grade calcium carbonate, $CaCO_3$, in a platinum dish and ignite for 1 hr at 1,000 C. After cooling the calcium oxide, hydrate by slowly adding distilled water with stirring and heating to boiling. Cool and filter the suspension and collect the solid calcium hydroxide on a fritted glass filter of medium porosity. Dry the calcium hydroxide in an oven at 110 C, cool, and pulverize to uniformly fine granules. Vigorously shake an excess of fine granules with distilled water in a stoppered polyethylene bottle, allowing the temperature to come to 25 C after mixing. Filter the supernatant under suction through a sintered glass filter of medium porosity and use the filtrate as the buffer solution. Discard the buffer solution when atmospheric carbon dioxide causes turbidity to appear.

4. Procedure

Because of the differences between the many makes and models of commercially available pH meters, it is impossible to provide detailed instructions for the proper operation of every instrument. In each case, follow the manufacturer's instructions. Thoroughly wet the glass and reference electrodes by immersing the tips in water overnight or in accordance with instructions. Thereafter, when the meter is not in use for pH measurement, keep the tips of the electrodes immersed in water.

Before use, remove the electrodes from the water and rinse with distilled or demineralized water. Dry the electrodes by gentle wiping with a soft tissue. Standardize the instrument with the electrodes immersed in a buffer solution with a pH approaching that of the sample and note the temperature of the buffer and the pH at the measured temperature. Remove the electrodes from the buffer, rinse thoroughly, and dry. Immerse in a second buffer approximately 4 pH units different from the first and note the pH reading; the reading should be within 0.1 unit of the pH for the second buffer. Rinse electrodes thoroughly, dry, and immerse in the sample. Agitate the sample sufficiently to provide homogeneity and keep solids in suspension. If the sample temperature is different from that of the buffers, let the electrodes equilibrate with the sample. Measure the sample temperature and set the temperature compensator on the pH meter to the measured temperature. Note and record the pH and temperature. Rinse electrodes and immerse in water until the next measurement.

When only occasional pH measurements are made, standardize the instrument before each measurement. Where frequent measurements are made, less frequent standardization (every 1 or 2 hr) is satisfactory. However, if sample pH values vary widely, standardize more frequently with a buffer having a pH within 1 to 2 pH units of that of the sample. Measure with two or more buffers of different pH at least once daily and more frequently if samples contain abrasive solids or dissolved fluorides, in order to check the linearity of response. When electrode response to two buffers 4 pH units different shows differences greater than 0.1 pH unit, replace the glass electrode.

pH measurements in high-purity waters such as condensate or demineralizer effluents are subject to atmospheric contamination and require special procedures for accurate pH measurement.

5. Precision and Accuracy

The precision and accuracy attainable with a given pH meter will depend on the type and condition of the instrument and the care used in standardization and operation. Guard against possible erratic results arising from mechanical or electrical failures—weak batteries, damaged electrodes, plugged liquid junctions, and fouling of the electrodes with oily or precipitated materials. With the proper care, a precision of ±0.02 pH unit and an accuracy of ±0.05 pH unit can be achieved with many of the new models. However, ±0.1 pH unit represents the limit of accuracy under normal conditions. For this reason, report pH values to the nearest 0.1 pH unit. A synthetic sample consisting of a Clark and Lubs buffer solution of pH 7.3 was analyzed electrometrically by 30 laboratories, with a standard deviation of ±0.13 pH unit.

6. References

1. Clark, W.M. 1928. The Determination of Hydrogen Ions. 3rd ed. Williams & Wilkins Co., Baltimore, Md.
2. Kolthoff, I.M. 1937. Acid-Base Indicators. 4th ed. Macmillan Co., New York, N.Y.
3. American Water Works Association. 1964. Simplified Procedures for Water Examination. Manual M12. AWWA, New York, N.Y.
4. Langelier, W.F. 1946. Effect of temperature on the pH of natural waters. *J. Amer. Water Works Ass.* 18:179.

7. Bibliography

Dole, M. 1941. The Glass Electrode. John Wiley & Sons, New York, N.Y.

Bates, R.G. & S.F. Acree. 1945. pH of aqueous mixtures of potassium dihydrogen phosphate and disodium hydrogen phosphate at 0 to 60 C. *J. Res. Nat. Bur. Standards* 34:373.

Bates, R.G. 1954. Electrometric pH Determinations. John Wiley & Sons, New York, N.Y.

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.

Bates, R.G. 1964. Determination of pH. John Wiley & Sons, New York, N.Y.

Staples, B.R. & R.G. Bates. 1969 Two new standards for the pH scale. *J. Res. Nat. Bur. Standards* 73A:37.

ORIGINAL
(Red)

425 F. Ascorbic Acid Method

1. General Discussion

a. Principle: Ammonium molybdate and potassium antimonyl tartrate react in an acid medium with dilute solutions of orthophosphate to form a heteropoly acid—phosphomolybdic acid—that is reduced to the 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 0.10 mg/l arsenic interfere with the phosphate determination. Hexavalent chromium and nitrite interfere to give results about 3% low at concentrations of 1.0 mg/l and 10 to 15% low at concentrations of 10 mg/l chromium and nitrite. Sulfide (Na_2S) and silicate do not interfere in concentrations of 1.0 and 10.0 mg/l.

c. Minimum detectable concentration: Approximately 10 μg P/l. P ranges are as follows:

Approximate P Range *mg/l*	Light Path Length *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 (1 in.) 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 Method D, ¶2*b* above.

3. Reagents

a. Sulfuric acid solution, 5N: Dilute 70 ml conc H_2SO_4 with distilled water to 500 ml.

b. Potassium antimonyl tartrate solution: Dissolve 1.3715 g $K(SbO)C_4H_4O_6 \cdot 1/2\ H_2O$ 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 plastic bottle at 4 C.

d. Ascorbic acid, 0.1M: Dissolve 1.76 g ascorbic acid in 100 ml distilled water. The solution is stable for about 1 wk 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.* All reagents must reach room temperature before they are mixed and must be mixed in the order given. If turbidity forms in the combined reagent, shake and let it stand for a few minutes until the turbidity disappears before proceeding. The reagent is stable for 4 hr.

f. Stock phosphate solution: See Method D, ¶3*e*.

g. Standard phosphate solution: Dilute 50.0 ml stock phosphate solution to 1,000 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 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 longer than 30 min, measure the color absorbance of each sample at 880 nm, using the 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. In the case of highly colored or turbid waters, prepare a blank by adding all the reagents except ascorbic acid and antimonyl potassium tartrate to the sample. Subtract the absorbance of the blank from the absorbance of each of the unknown samples.

c. Preparation of calibration curve: Prepare individual calibration graphs from a series of six standards within the phosphate ranges indicated in Section 425F.1c. Use a distilled water blank with the combined reagent to make the 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

$$\text{mg/l P} = \frac{\text{mg P} \times 1{,}000}{\text{ml sample}}$$

6. Precision and Accuracy

The precision and accuracy values given in Table 425:II are for a single solution procedure given in the previous edition. The technic presented differs in reagent-to-sample ratios, no addition of solvent, and acidity conditions. It is superior in precision and accuracy to the 13th edition technic in the analysis of both distilled water and river water at the 228 μg P/l level (Table 425:III).

TABLE 425:III. COMPARISON OF PRECISION AND ACCURACY OF ASCORBIC ACID METHODS

Ascorbic Acid Method	Phosphorus Concentration, Filtrable Orthophosphate *mg/l*	No. of Laboratories	Relative Standard Deviation %		Relative Error %	
			Distilled Water	River Water	Distilled Water	River Water
13th Edition (Edwards, Molof, and Schneeman) .	0.228	8	3.87	2.17	4.01	2.08
Current method (Murphy and Riley)	0.228	8	3.03	1.75	2.18	1.39

505 ORGANIC CARBON (TOTAL)

Combustion-Infrared Method*

The total organic carbon concentration generally falls below the true value of organic contaminants because other constituent elements are excluded. After an empirical relationship has been established between the total organic carbon, and the biochemical oxygen demand or the chemical oxygen demand, the total organic carbon (TOC) provides a speedy and convenient way of estimating the other parameters that express the [illegible] of organic contamination.

Furthermore, total organic carbon is a more direct expression of the organic chemical content of water than either of the two other analyses. As such, TOC can be used to monitor processes for the treatment or removal of organic contaminants without undue dependence on the oxidation states, and can do so at low concentrations.

1. General Discussion

The carbon analyzer offers a means of measuring total organic carbon in the range of 1 to 150 mg/l in water and wastewater. Appropriate dilution of the sample enables the determination of greater carbon concentrations as well as the analysis of water samples bearing a high salt, acid, or base content. Smaller carbon concentrations can be estimated by suitable concentration of the sample or through the use of larger portions. The procedure yields the best results with homogeneous samples that are reproducibly (±1.0 mg/l carbon) injectable into the apparatus by a microliter-type syringe. The needle opening o the syringe restricts the maximum size o particles that may be included in th samples.

a. Principle: The water sample is homogenized or diluted as necessary and microportion is injected into a heated packed tube in a stream of oxygen o purified air. The water is vaporized an the organic matter is oxidized to carbo dioxide, which is measured by means a nondispersive type of infrared analyzer. Because the carbon analyze measures all of the carbon in a sampl after injection into the combustion tub procedural modifications are needed limit the determination to organic carbon. Inorganic carbonates may be decomposed with acid and volatilized i the form of carbon dioxide before the organic carbon is determined. Alternatively, the total organic and inorganic carbon determination can be followed by separate determination for the inorgan carbon. The difference between the tot and the inorganic carbon then yields th organic carbon.

b. Interference: Removal of carbona and bicarbonate by means of acidification and purging with nitrogen g can result in the loss of very volatile organic substances. Another importan loss can occur if large carbon-containin particles in the sample fail to enter th hypodermic needle used for injectio Filtration, although desirable to lim the insoluble inorganic matter, can result in loss or gain of TOC, dependin on the physical properties of the carbo

---

*This method is identical in substance to ASTM D2579-69.

ORIGINAL
(Red)

containing compounds and the adsorption or desorption of carbonaceous matter from the filter. Any sample treatment may alter the measurable carbon. Record such treatment and consider it in any interpretation of results.

c. Minimum detectable concentration: 1 mg/l carbon. This concentration may be lowered by concentrating the sample or by increasing the portion taken for analysis.

d. Sampling and storage: Collect and store samples in bottles made of glass, preferably brown. Plastic containers are acceptable after tests have demonstrated the absence of extractable carbonaceous substances. Use a Kemmerer or similar type sampler for collection of samples from a depth exceeding 2m (5 ft). Protect samples that cannot be examined promptly from decomposition or oxidation by preservation at ice temperatures, minimal exposure to light and atmosphere, or acidification with hydrochloric acid to a pH not over 2. Under any conditions, minimize storage time.

2. Apparatus

a. Sample blender or homogenizer, Waring type or ultrasonic.

b. Magnetic stirrer.

c. Hypodermic syringe, 0 to 50 or 0 to 500 µl capacity.†

d. Total organic carbon analyzer.‡

3. Reagents

a. Redistilled water: Prepare the blank and standard solutions with redistilled water.

b. Hydrochloric acid, HCl, conc.

c. Standard carbon solution: Dissolve 5.571 g anhydrous sodium oxalate, $Na_2C_2O_4$, in redistilled water and dilute to 1,000 ml; 1.00 ml = 1.00 mg carbon. Alternatively, use any other carbon-containing compound of adequate purity, stability, and water solubility.

d. Packing for oxidation tube: Follow the directions supplied with the total organic carbon analyzer.

e. Oxygen gas, carbon-dioxide-free.

f. Nitrogen gas, carbon-dioxide-free.

4. Procedure

a. Instrument operation: The differences between satisfactory analyzers make it impossible to give detailed instructions applicable to every instrument. Therefore, follow the manufacturer's instructions for assembly, testing, calibration, and operation of the analyzer on hand. Vary the injected sample size from that normally recommended according to manufacturer's instructions or if an enlarged combustion tube is available.

b. Sample treatment: If the sample contains gross solid or insoluble liquid matter, homogenize sample with a blender or ultrasonics, until satisfactory repeatability is obtained.

If inorganic carbon must be removed before analysis, transfer a representative portion of 10 to 15 ml to a 30-ml beaker, add 2 drops (0.1 ml) conc HCl to reduce the pH to 2 or less, and purge with carbon-dioxide-free nitrogen gas for 10 min. Do not use plastic tubing. While stirring on a magnetic stirrer, withdraw the sample from the beaker by means of a hypodermic needle with a 150-µm opening. Inject the sample into

---

†Hamilton No. 705 N or 750 N; CR-700-20 or CR 700-200 with needle point style No. 1.

‡Beckman Instruments, Inc., or equivalent.

the analyzer and obtain the peak-height reading. Repeat the injection twice or until three consecutive peaks are obtained that are reproducible to within ±3%.

If the available instrument provides for a separate determination of the carbonate-plus-bicarbonate carbon, omit the decarbonation step with conc HCl and proceed according to the manufacturer's directions.

c. Preparation of standard curve: Prepare a standard carbon series of 10, 20, 30, 40, 50, 60, 80, and 100 mg/l with redistilled water by diluting 10, 20, 30, 40, and 50 ml standard carbon solution to 1,000 ml, and 30, 40, and 50 ml standard carbon solution to 500 ml. Inject and record the peak heights of these standards and dilution water blank. Correct peak height for blank.

Plot the carbon concentrations of the standards in milligrams per liter versus the corrected peak height in millimeters on rectangular coordinate paper.

Inject samples and reagent blanks § and ascertain the sample concentrations from the corrected peak heights of the samples by reference to this calibration curve.

5. Calculation

a. Calculate the corrected peak height in millimeters by deducting the blank correction in the standards and samples as follows:

Corrected peak height, mm $= A - B$

where A = peak height in mm of the standards or sample and B = peak height in mm of the blank.

b. Apply the appropriate dilution factor when necessary.

6. Precision

The difficulty of sampling particulate matter on unfiltered samples limits the precision of the method to approximately 5 to 10%. On clear samples or on those that have been filtered before analysis, the precision approaches 1 to 2% or 1 to 2 mg/l carbon, whichever is greater.

7. Bibliography

Van Hall, C.E., J. Safranko & V.A. Stenger. 1963. Rapid combustion method for the determination of organic substances in aqueous solutions. *Anal. Chem.* 35:315.

Van Hall, C.E., D. Barth & V.A. Stenger. 1965. Elimination of carbonates from aqueous solutions prior to organic carbon determinations. *Anal. Chem.* 37:769.

Schaffer, R.B. et al. 1965. Application of a carbon analyzer in waste treatment. *J. Water Pollut. Control Fed.* 37:1545.

Busch, A.W. 1966. Energy, total carbon, and oxygen demand. *Water Resour. Res.* 2:59.

Williams, R.T. 1967. Water-pollution instrumentation—Analyzer looks for organic carbon. *Instrum. Technol.* 14:63.

Blackmore, R.H. & D. Voshel. 1967. Rapid determination of total organic carbon (TOC) in sewage. *Water Sewage Works* 114:398.

Van Hall, C.E. & V.A. Stenger. 1967. An instrumental method for rapid determination of carbonate and total carbon in solutions. *Anal. Chem.* 39:503.

---

§Redistilled dilution water and HCl may be contaminated with organic carbon so results must be corrected if blank TOC results are significant.

ORIGINAL
(Red)

ples stored for one day at atmospheric temperature. *Proc. Soc. Water Treat. Exam.* 13:350.

Lonsane, B.K., N.M. Parhad & N.U. Rao. 1967. Effect of storage temperature and time on the coliform in water samples. *Water Res.* (Britain) 1:109.

Lucking, H.E. 1967. Death rate of coliform
teria in stored Montana water sampl
Environ. Health 29:576.

Van Donsel, D.J. & E.E. Geldreich.
Relationships of Salmonellae to fecal
forms in bottom sediments. *Water*
5:1079.

907 STANDARD PLATE COUNT

1. Introduction

The Standard Plate Count procedure provides a standardized means of determining the density of aerobic and facultative anaerobic heterotrophic bacteria in water. This is an empirical measurement because bacteria occur singly, in pairs, chains, clusters, or packets, and no single growth medium or set of physical and chemical conditions can satisfy the physiological requirements of all bacteria in a water sample. Consequently, the number of colonies may be lower substantially than the actual number of viable bacteria present. To facilitate the collection of reliable data for water quality control measurements, especially for comparative and legal purposes, a standardized plate count procedure is essential.

2. Work Area

A level table or bench top with ample area should be available in a clean, draft-free, well-lighted room. Table and bench tops should have a nonporous surface and should be disinfected before any analysis is conducted.

3. Samples

Potable water samples from a distribution system should be collected as di-
rected in Section 906A. Initiate san
analysis as soon as possible to minin
changes in the bacterial population.
recommended maximum elapsed
between collection and examination
unrefrigerated samples is 8 hr (m
mum transit time 6 hr, maximum p
essing time 2 hr). When analysis can
begin within 8 hr, maintain the sam
at a temperature below 10 C.
maximum elapsed time between
lection and analysis shall not exceed
hr.

Bottled water samples obtained f
retail outlets may be held or transpo
unrefrigerated provided the temperat
does not exceed 20 to 25 C. Exam
freshly bottled samples (less than 4
old) within 6 hr of collection if unre
erated and within 30 hr if refrigerate

4. Sample Preparation

Mark each plate with sample nu
ber, dilution, date, and any other ne
sary information before sample ex
ination. Prepare duplicate plates
each volume of sample or sample d
tion examined.

Thoroughly mix all samples by m
ing 25 complete up-and-down (or b
and-forth) movements of about 0.3
ft) in 7 sec. Optionally, use a mech
cal shaker to shake the dilution bl
for 15 sec.

5. Sample Dilution

Prepare water used for dilution blanks as directed in Media Specifications, Section 905 C.

a. Selecting dilutions: Select the dilution(s) so that the total number of colonies on a plate will be between 30 and 300 (Figure 907.1). For example, where a Standard Plate Count as high as 3,000 may be suspected, prepare plates containing 1:100 dilution.

For most potable water samples, plates suitable for counting will be obtained by planting 1 ml and 0.1 ml of undiluted sample and 1 ml of sample diluted 1:100.

b. Measuring sample portions: Use a sterile pipet for initial and subsequent transfers from each container. If the pipet becomes contaminated before the transfers are completed, replace it with a sterile pipet. Use a separate sterile pipet for transfers from each different dilution. Do not prepare dilutions and pour plates in direct sunlight. Use caution when removing sterile pipets from the container; to avoid contamination of the pipet, do not drag the tip across the exposed ends of pipets or across the lips and necks of dilution bottles. When removing sample, do not insert pipets more than 2.5 cm (1 in.) below the surface of the sample or dilution.

c. Measuring dilutions: When measuring diluted samples of water, hold the pipet at an angle of about 45° with the tip touching the inside cover of the petri dish or the inside neck of the dilution bottle. Lift the cover of the petri dish just

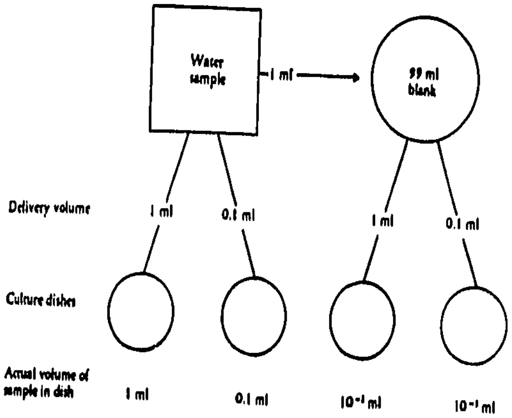


Figure 907:1. Preparation of dilutions.

ORIGINAL
(Red)

high enough to insert the pipet. Allow 2 to 4 sec for the liquid to drain from the 1 ml graduation mark to the tip of the pipet. If the pipet is not a blow-out type, touch the tip of the pipet *once* against a dry spot in the petri plate. If the pipet is, less preferably, a blow-out type, it must have a cotton plug in the mouthpiece; gently blow out the remaining volume of sample dilution. When 0.1-ml quantities are measured, let the diluted sample drain from the chosen reference graduation until 0.1 ml has been delivered. Remove the pipet without retouching it to the plate. Pipet 1 ml, 0.1 ml, or other suitable volume of the dilution to be used for plating in the sterile petri dish before adding the melted culture medium. It is recommended that decimal dilutions be used in preparing sample volumes of less than 1 ml; in the examination of sewage or turbid water, do not measure a 0.1-ml inoculum of the original sample, but prepare an appropriate dilution. Prepare at least two replicate plates for each sample dilution used. After depositing test portions for each series of plates, pour the culture medium.

6. Plating

a. Melting medium: Melt sterile solid agar medium in boiling water or by exposure to flowing steam in a partially closed container, but avoid prolonged exposure to unnecessarily high temperatures during and after melting. Do not resterilize the plating medium. If the medium is melted in two or more batches, use all of each batch in order of melting, provided that the contents in separate containers remain fully melted. Discard melted agar that contains precipitate.

Temper the melted medium in a water bath between 44 C and 46 C until used. In a separate container place a thermometer in water or medium that has been exposed to the same heating and cooling as the plating medium. Do not depend on the sense of touch to indicate the proper temperature of the medium when pouring agar.

Use tryptone glucose extract agar or plate count agar, as specified in Section 905 C.

b. Pouring the plates: Limit the number of samples to be plated in any one series so that no more than 20 min (preferably 10 min) elapse between dilution of the first sample and pouring of the last plate in the series. Pour at least 10 to 12 ml of liquefied medium at 44 to 46 C into each plate by gently lifting the cover of the petri dish just high enough to pour the medium. Carefully avoid spilling the medium on the outside of the container or on the inside of the plate lid when pouring. As each plate is poured mix the melted medium thoroughly with the test portions in the petri dish, taking care not to splash mixture over the edge, by rotating the dish first in one direction and then in the opposite direction, or by rotating and tilting the dish. Allow the plates to solidify (within 10 min) on a level surface. After the medium solidifies, invert the plates and place them in the incubator.

c. Sterility controls: Check the sterility of the medium and the dilution water blanks by pouring control plates for each series of samples. Additional controls also may be prepared to determin contamination of plates, pipets, room air.

ORIGINAL
(Red)

7. Incubation

Incubate for the Standard Plate Count for all water samples except bottled water at a temperature of 35±0.5 C for 48±3 hr.

For the Standard Plate Count of bottled water, the plates shall be incubated at 35±0.5 C for 72±4 hr. Since many of the bacteria found in bottled water demonstrate a prolonged lag phase during adaptation to growth on tryptone glucose extract agar or plate count agar, such bacteria do not form colonies that can be counted after 48 hr incubation so that an additional 24 hr incubation is required to obtain a reliable Standard Plate Count.

Plates should be packed as directed under Laboratory Apparatus, Section 903, without crowding in the incubator. Any deviation from this method must be stated in the examination report.

8. Counting and Recording

Count all colonies on selected plates promptly after the incubation period. If counting must be delayed temporarily, store plates at 5 to 10 C for a period of no more than 24 hr, but avoid this as routine practice. Record the results of sterility controls on the report for each lot of samples.

Use an approved counting aid, such as the Quebec colony counter, for manual counting. If such equipment is not available, counting may be done with any other counter provided that it gives equivalent magnification and illumination. Automatic plate counting instruments are now available. These generally use a television scanner coupled to a magnifying lens and an electronics package. Their use is acceptable if evaluation in parallel with manual counting gives comparable results.

In preparing plates, volumes of sample should be planted that will give from 30 to 300 colonies on a plate. The aim should be to have at least one dilution for which the replicate plates give colony counts between these limits, except as provided below.

Ordinarily, it is not desirable to plant more than 1.0 ml of water in a plate; therefore, when the total number of colonies developing from 1.0 ml is less than 30, it is necessary to disregard the rule above and record the result as observed. With this exception, only plates showing 30 to 300 colonies should be considered in determining the Standard Plate Count. Compute the bacterial count per milliliter by multiplying the average number of colonies per plate by the dilution used. Report as the "Standard Plate Count" per milliliter.

If there is no plate with 30 to 300 colonies, and one or more plates have more than 300 colonies, use the plate(s) having a count nearest 300 colonies. Compute the count by multiplying the average count per plate by the dilution used and report as the "Estimated Standard Plate Count" per milliliter.

If plates from all dilutions of any sample have no colonies, report the count as less than one (<1) times the corresponding lowest dilution. For example, if no colonies develop on the 1:100 dilution, report the count as "less than 100 (<100) Estimated Standard Plate Count" per milliliter.

If the number of colonies per plate far exceeds 300, do not report the result as "too numerous to count" (TNTC). If there are fewer than 10 colonies/cm^2, count colonies in 13 squares (of the col-

ORIGINAL
(Red)

ony counter) having representative col*ony distribution. If possible, select seven* consecutive squares horizontally across the plate and six consecutive squares at right angles, being careful not to count a square more than once. Multiply the sum of the colonies in 13 representative cm^2 by 5 to compute the estimated colonies per plate when the area of the plate is 65 cm^2. When there are more than 10 colonies/cm^2, count four representative *squares, take the average count per* square cm and multiply by the appropriate factor to estimate the colonies per plate (usually about 65). When bacterial counts on crowded plates are greater than 100 colonies/cm^2, report the result as greater than (>) 6,500 times the highest dilution plated.

If spreading colonies (spreaders) are encountered on the plate(s) selected, *count colonies on representative portions* only when (*a*) colonies are well distributed in spreader-free areas, and (*b*) the area covered by the spreader(s) does not exceed one-half the plate area.

When spreading colonies must be counted, count each unit of the following types as one: (*a*) The first is a chain of colonies that appears to be caused by disintegration of a bacterial clump as the *agar and sample were mixed.* Count each such chain as a single colony, do not count each individual colony in the chain; (*b*) The second type of spreader develops as a film of growth between the agar and the bottom of the petri dish; (*c*) The third type forms in a film of water at the edge or over the surface of the agar. Types *b* and *c* largely develop because of an accumulation of moisture at *the point from which the spreader originates.* They frequently cover more than half the plate and interfere with obtaining a reliable plate count.

If plates prepared from the samples *have excessive spreader growth, report* as "Spreaders" (Spr). When plates are uncountable because of missed dilution, accidental dropping, and contamination, or the control plates indicate that the medium or other material or labware was contaminated, report as "Laboratory Accident" (LA).

9. Computing and Recording Counts

To compute the Standard Plate Count, multiply the total number of colonies or the average number (if duplicate plates of the same dilution) per plate by the reciprocal of the dilution used. Record the dilutions used and the number of colonies on each plate counted or estimated.

When colonies on duplicate plates and/or consecutive dilutions are counted and the results are averaged before being recorded, round off counts to two significant figures only at the time of conversion to the Standard Plate Count.

Avoid creating fictitious ideas of precision and accuracy when computing Standard Plate Counts, by recording only the first two left-hand digits. Raise *the second digit to the next highest number* only when the third digit from the left is 5, 6, 7, 8, or 9; use zeros for each successive digit toward the right from the second digit. For example, a count of 142 is recorded as 140, and a count of 155 as 160, whereas a count of 35 is recorded as 35.

10. Reporting Counts

Report counts as "Standard Plate Count" or "Estimated Standard Plate Count" per milliliter.

ORIGINAL
(Red)

APPENDIX B-2

EXPERIMENTAL PROCEDURES FOR XANTHATE FLOTATION

ORIGINAL
(Red)

APPENDIX B-2

EXPERIMENTAL PROCEDURE FOR XANTHATE FLOTATION

Equipment:

One 4-liter heavy-duty beaker
500-ml collection beakers
Mechanical stirrer (from KPEG reactor setup)

Chemicals:

Potassium amyl xanthate (AERO 350)
2-Ethylhexanol (AEOFROTH 88 Frother)
Deionized water

Procedure -- Run 1:

1. Filled 2 eight-ounce glass sample jars with raw (untreated) soils/sediment from a previously homogenized container of soil/sediment.

2. Placed 500 g of untreated soil/sediment in the 4-L beaker. Tilted the beaker and secured in place with clamps. Added 3.5 L deionized water. Began slow stirring of the soil/sediment slurry. Began bubbling air through the mixture. Added 1 pellet of potassium amyl xanthate (xanthate) and 1 drop of 2-ethylhexanol (frother).

3. No froth appeared. Stopped mixing after 5 minutes...the action destroyed the limited bubbles which were forming.

4. Increased xanthate to 3 pellets (~0.27 g) and increased frother to 6 drops. No improvement in frothing observed.

5. Increased xanthate to 5 pellets (~0.45 g) and increased to 16 drops. No improvement in frothing observed.

6. Mixed slurry by hand and skimmed off existing bubbles with a plastic spoon. Treatment was stopped after 40 minutes.

7. The soil/sediment was allowed to settle in the flask. The aqueous layer was decanted and placed in a liquid hazardous waste collection drum.

ORIGINAL
(Red)

8. Filled one eight-ounce sample jar with treated soil/sediment.

9. Cleaned glassware with soap and hot water. Rinsed with methanol and deionized water. Rinsate placed in liquid hazardous waste collection drum.

<u>Procedure -- Run 2</u>:

1. Dissolved 8 pellets (~0.75 g) of xanthate and 16 drops of frother in 3.5 L deionized water in the 4-L beaker.

2. Placed 500 g of untreated soil/sediment in the 4-L beaker. Tilted the beaker and secured in place with clamps. Began slow stirring of the soil/sediment slurry. Began bubbling air through the mixture.

3. Very little froth appeared. Stopped mixing after 15 minutes...the action destroyed the limited bubbles which were forming.

4. Mechanically "coaxed" bubble formation by hand stirring the slurry and moving the bubbler closer to the surface.

5. Skimmed off froth bubbles with a plastic spoon. Treatment was stopped after 3[illegible] minutes.

6. The soil/sediment was allowed to settle in the flask. The aqueous layer was decanted and placed in a liquid hazardous waste collection drum.

7. Filled one eight-ounce sample jar with treated soil/sediment.

8. Cleaned glassware with soap and hot water. Rinsed with methanol and deionized water. Rinsate placed in liquid hazardous waste collection drum.

9. Samples were labeled, packed in ice, and shipped.

ORIGINAL
(Red)

APPENDIX B-3

STANDARD OPERATING PROCEDURE FOR LOW-TEMPERATURE THERMAL DESORPTION

APPENDIX B-3

STANDARD OPERATING PROCEDURE FOR LOW-TEMPERATURE THERMAL DESORPTION

Equipment:

Aluminum tray
Reaction flask with stirring paddle and motor assembly
Thermometer
heating mantle with variable transformer
Nitrogen gas cylinder
Water-cooled condenser
Laboratory scale
Stainless steel spoon
1 liter volumetric flask
Vacuum

Reagents:

Deionized water
Laboratory detergent (for decontamination)
10% nitric acid (for decontamination)
Methanol (for decontamination)

Procedure:

1. Inside fume hood and using a stainless steel spoon, transfer 800-g aliquot of homogenized surface soil/sediment to a desorption vessel and spread it in a uniform layer on the bottom of the vessel (see Figure 6-2).

2. Place the glass cover on the desorption vessel and close the clamps. Turn on the condenser water, nitrogen gas, and vacuum.

3. Turn on the heating mantle and s[illegible] the temperature control point for 300°F (149°C) test temperature to heat the soil inside the reaction flask. Monitor the temperature

of the soil continuously (at every 15 minutes interval) until it reaches 300°F. Start the timer and record the time and temperature of the soil every 5 minutes.

4. When the prescribed residence time (15 or 30 minutes) at the target temperature of 300°F is reached, turn off the heating mantle. Continue the nitrogen purge until the soil cools to ambient temperature.

5. Remove the vessel cover and weigh the reaction vessel. Scrape as much soil as possible out of the vessel with a stainless steel spoon and place it in a clean empty aluminum tray. Weigh the empty reaction vessel.

6. Collect samples of the soil for analysis and cool them to 4°C. Repeat steps 1 through 6 until sufficient soil has been collected for analysis. If multiple runs are required, collect all soil in one jar and homogenize it before submitting it for analysis.

7. Decontaminate the equipment in the following order: detergent solution, tap water, nitric acid, tap water, methanol, and deionized water. Allow the equipment to dry.

8. In addition to runs at 300°F, tests will also be conducted at 500°F. For these tests...*repeat steps 1 through 7, except at step 3 reset the temperature controller* for the 500°F test temperature.

9. Sufficient soil must be prepared at 500°F and 30 minutes to conduct further treatability studies (solidification/stabilization).

ORIGINAL
(Red)

APPENDIX B-4

STANDARD OPERATING PROCEDURES FOR SOLIDIFICATION/STABILIZATION

APPENDIX B-4.1

STANDARD OPERATING PROCEDURE FOR CEMENT-BASED SOLIDIFICATION/STABILIZATION

Equipment:

Reaction flask with stirring paddle and motor assembly
Laboratory scale
Stainless steel spoons
Cylindrical molds 6.5 cm in diameter by 10 cm long for TCLP tests, and 7 cm in diameter by 14.22 cm long for UCS tests
Tamping tool
Hermetically sealed chamber

Reagents:

Deionized water
Portland cement (Type II)
10% nitric acid (for decontamination)
Methanol (for decontamination)
Laboratory detergent (for decontamination)

Procedure:

1. Based on the water-to-total solids ratio of 0.25, determine the amount of deionized water that is required to solidify the soil samples with a binder-to-soil ration of 0.4.

2. Inside fume hood, place the water in a reaction flash (No. 1) which is fitted with a stirring paddle. A motor assembly is attached to the shaft of the stirring paddle (see Figure B-2).

3. Place 800 g of the soil residue from low-temperature thermal desorption in a second reaction flash (No. 2). Place the glass cover on the flask and seal with the metal clamps.

ORIGINAL
(Red)

4. Homogenize the soil inside reaction flask No. 2 by turning on the stirrers for 10 minutes.

5. Based on the binder-to-soil ration of 0.4, add 320 g of portland cement (Type II) to the soil in reaction flash No. 2. Again, mix the contents of the flask for 10 minutes.

6. Gradually empty the soil-cement mixture from reaction flask No. 2 to the deionized water in reaction flask No. 1. Use a stainless steel spoon to homogenize the material for 10 minutes.

7. Place the mixture in clear-rigid cylindrical molds to provide solidified samples for the unconfined compressive strength (UCS) and modified TCLP leach tests. The molds should be thoroughly sealed to prevent migration of water out of the soil-cement mixtures. UCS sample will be placed in 7-cm-diameter by 14.22-cm-long molds. TCLP sample will be placed in 4.5-cm-diameter by 10-cm-long molds.

8. To assure that the molds are completely filled and any air bubbles and voids that may have developed during sample mixing are removed, tamp the walls of the molds lightly with a plastic tamping tool until the samples no longer seem to reduce volume with repeated tamping.

9. Place the molds inside zip-lock storage bags at 72°F (± 5°F) and allow them to cure for at least 14 days.

10. Decontaminate the equipment in the following order: detergent solution, tap water, 0.1 N nitric acid, tap water, methanol, and deionized water. Allow the equipment to dry.

11. Repeat steps 1 through 10 using a binder-to-soil ratio of 0.25. At step 5 , add 200 g of portland cement to the soil in reaction flash No. 2.

12. For solidification of the raw (untreated) soil samples, repeat steps 1 through 11, except in step 5 based on the 31.1 percent water content of the soil samples, add 244.3 g and 152.7 g portland cement to the soils. To prepare solidified samples with B/S ratios of 0.4 and 0.25 respectively.

ORIGINAL
(Red)

APPENDIX B-4.2

STANDARD OPERATING PROCEDURE FOR ASPHALT-BASED SOLIDIFICATION/STABILIZATION

Equipment:

- Reaction flask with stirring paddle and motor assembly
- Thermometer
- Heating mantle with variable transformer
- Cylindrical molds 4.5 cm in diameter by 10 cm long for TCLP tests, and 3.8 cm in diameter by 8.4 cm long for UCS test
- Laboratory scale
- Stainless steel spoons
- Tamping tool

Reagents:

- Deionized water
- 10% nitric acid (for decontamination)
- Methanol (for decontamination)
- Laboratory detergent (for decontamination)
- Kerosene (for decontamination)
- Asphalt

Procedures:

1. Inside fume hood, place 500 g of the soil residue from the low-temperature thermal desorption process in a reaction flask (No. 1) that has been fitted with a thermometer and a stirring paddle. A motor assembly is attached to the shaft of the stirring paddle (see Figure B-3). Place the glass cover on the flask and close the metal clamps.

2. Place 1200 g of asphalt in a second reaction flask (No. 2) that is fitted with a thermometer. Place the reaction flask into the heating mantle and hit it until the

asphalt inside the flash melts. Adjust the temperature of the heating mantle so that the asphalt remains in a liquid state throughout the experiment. Record the temperature of the asphalt.

3. Homogenize the soil residue inside reaction flash No. 1 by turning on the stirrer for 10 minutes. Heat the soil to ~60°C for butter mixing with asphalt.

4. *Based on B/S ration of 1, remove 500 g of liquid asphalt from reaction flask No.* 2 and add it to the soil in reaction flash No. 1.

5. Use a stainless steel spoon to mix the soil and asphalt inside reaction flask No. 1 for 10 minutes. The temperature of the mixture should be adjusted so it is at the same temperature recorded in step 2,

6. place the mixture in clear-rigid cylindrical molds to provide samples for the modified TCLP leach tests and the UCS test. The UCS samples will be placed in 3.8 cm in diameter by 8.4-cm-long cylindrical molds. The TCLP samples will be prepared by placing the soil asphalt mixture in 4.5-cm in diameter by 10-cm-long cylindrical molds.

7. To assure that the molds are completely filled and any voids that may have developed during sample preparation are removed, tamp the walls of the molds lightly with a plastic tamping tool until the samples no longer seem to reduce in volume with repeated tamping.

8. Place the molds inside zip-lock bags at 72°F (15°F) and allow them to cure for at least 14 days.

9. Decontaminate the equipment using the following sequence: kerosene, tap water, laboratory detergent, tap water, 10 percent nitric acid, tap water, methanol, and deionized water. Allow the equipment to dry.

10. Repeat steps 2 through 9, at step 4 remove 250 g of liquid asphalt from reaction flash No. 2 and add it to the soil in reaction flash No. 1 to prepare solidified samples with B/S *ratio of 0.5.*

ORIGINAL
(Red)

APPENDIX C

ANALYTICAL DATA FOR UNTREATED SOIL AND GROUNDWATER, LOW TEMPERATURE THERMAL DESORPTION STUDIES, AND SOLIDIFICATION/STABILIZATION STUDIES

ORIGINAL
(Red)

CONTENTS

APPENDIX C

ORIGINAL
(Red)

APPENDIX C-1

ANALYTICAL RESULTS FOR HSL COMPOUNDS IN UNTREATED SOILS, SEDIMENT AND GROUNDWATER SAMPLE

IT INTERNATIONAL TECHNOLOGY CORPORATION

ANALYTICAL SERVICES

ORIGINAL
(Red)

CERTIFICATE OF ANALYSIS

Halby T&E Facility   Date: May 24, 1990

Attn: Judy Hessling (PEI)

Job Number PN 3741-60-2

This is the Certificate of Analysis for the following samples:

Client Project ID:	Halby T&E Facility
Date Received:	May 17, 1990
Work Order:	X0-05-134
Number of Samples:	3
Sample Type:	Soil and Water

I. Introduction

Two soil and one water sample arrived at ITAS Cincinnati on May 17, 1990. The samples were sent for analytical work in support of work at Halby T&E Facility. The samples were collected on May 16, 1990 and were labeled as follows:

Soil Sample 3741-X02-5/16   Sediment 3741-X03-5/16   Water Sample 3741-X01-5/16

II. Analytical Results/Methodology

The analytical results for this report are presented by analytical test. Each set of data will include sample identification information, the analytical results, and the appropriate detection limits.

The analyses requested and methods used are listed on Table I.

Reviewed and Approved by:

Ken Mueller

Ken Mueller
Inorganic Group Leader
005134

ORIGINAL
(Red)

III. Quality Control

Immediately following the analytical data for the samples can be found the QA/QC information that pertains to these samples. The purpose of this information is to demonstrate that the data enclosed is scientifically valid and defensible. This QA/QC data is used to assess the laboratory's performance during the analysis of the samples it accompanies. All quantitations were performed from within the calibrated range of the analytical instrument.

Client: Halby T&E Facility
Work Order: X0-05-134
00513402

ORIGINAL
(Red)

TABLE I

Parameter	Methods for Soils	Methods for Water
Cyanide	9012	9012
Phenolics	9065	9065
Aluminum	3050/6010	3010/6010
Antimony	3050/6010	3010/6010
Arsenic	3050/7060	7060
Barium	3050/6010	3010/6010
Beryllium	3050/6010	3010/6010
Cadmium	3050/6010	3010/6010
Calcium	3050/6010	3010/6010
Chromium	3050/6010	3010/6010
Cobalt	3050/6010	3010/6010
Copper	3050/6010	3010/6010
Iron	3050/6010	3010/6010
Lead	3050/6010	3010/6010
Magnesium	3050/6010	3010/6010
Manganese	3050/6010	3010/6010
Mercury	7471	7470
Nickel	3050/6010	3010/6010
Potassium	3050/6010	3010/6010
Selenium	3050/7740	7740
Silver	3050/6010	3010/6010
Sodium	3050/6010	3010/6010
Thallium	3050/6010	3010/6010
Vanadium	3050/6010	3010/6010
Zinc	3050/6010	3010/6010

ORIGINAL
(Red)

IT ANALYTICAL SERVICES
CINCINNATI, OH

Client: Halby T&E Facility
Work Order: X0-05-134
00513403

Analytical Results, ug/g

Client Sample ID	Soil Sample 3741-X02-5/16	Sediment 3741-X03/5/16	
Lab No.	01	02	
Parameter			Detection Limit
Cyanide	2.78	39.2	1
Phenolics	0.70	2.4	0.6
Aluminum	6240	4150	2
Antimony	ND	ND	8
Arsenic	619	322	0.03
Barium	65.6	83.0	0.07
Beryllium	ND	4.74	0.8
Cadmium	5.55	7.21	0.2
Calcium	840	2370	0.03
Chromium	27.0	233	0.2
Cobalt	104	9.04	2
Copper	323	219	0.3
Iron	23,000	64,100	0.5
Lead	120	152	5
Magnesium	1230	782	0.2
Manganese	184	312	0.2
Mercury	1.46	3.47	0.2
Nickel	20.2	25.3	3
Potassium	429	289	50
Selenium	0.50	0.12	0.04
Silver	0.95	ND	0.2
Sodium	345	315	2
Thallium	ND	ND	7
Vanadium	23.0	28.8	0.7
Zinc	3030	788	0.4

ND= Not Detected

Client: Halby T&E Facility
Work Order: X0-05-134
00513404

IT ANALYTICAL SERVICES
CINCINNATI, OH

ORIGINAL
(Red)

Analytical Results, mg/L

Client Sample ID	Water Sample 3741-X01-5/16	
Lab No.	03	
Parameter		**Detection Limit**
Cyanide	0.06	0.02
Phenolics	ND	0.03
Aluminum	0.07	0.03
Antimony	ND	0.2
Arsenic	0.11	0.0005
Barium	0.17	0.001
Beryllium	ND	0.001
Cadmium	0.03	0.004
Calcium	86.2	0.001
Chromium	ND	0.002
Cobalt	ND	0.04
Copper	0.01	0.005
Iron	51.7	0.008
Lead	ND	0.09
Magnesium	28.4	0.003
Manganese	8.32	0.003
Mercury	0.0001	0.0001
Nickel	ND	0.06
Potassium	25.0	1
Selenium	0.003	0.0008
Silver	ND	0.003
Sodium	262	0.03
Thallium	ND	0.2
Vanadium	0.02	0.02
Zinc	0.18	0.008

ND = Not Detected

Client: Halby T&E Facility
Work Order: XO-5-134
00513405

ORIGIN
(Red)

Quality Control
Standard Reference Solutions

Parameter	Theoretical Value, mg/L	Percent Recovery
Cyanide	10	94.8
Phenolics	2	101, 106
Aluminum	1	101, 97.2
Antimony	2	94.2, 90.9
Arsenic	0.075	90.4, 89.1, 91.7
Barium	1	105, 101
Beryllium	1	93.6, 98.0
Cadmium	1	92.8, 97.1
Calcium	1	107, 98.3
Chromium	1	97.3, 93.5
Cobalt	1	96.0, 93.5
Copper	1	92.5, 93.6
Iron	2	95.9, 97.9
Lead	2	102, 95.6
Magnesium	1	98.0, 104
Manganese	1	104, 101
Mercury	0.01	101, 92.0, 98.0
Nickel	1	91.6, 95.0
Potassium	25	95.5, 92.9
Selenium	0.075	90.5, 89.5, 92.5
Silver	1	110, 103
Sodium	2	90.2, 97.4
Thallium	2	98.0, 89.6
Vanadium	1	99.3, 97.8
Zinc	1	95.0, 94.6

IT INTERNATIONAL TECHNOLOGY CORPORATION

ANALYTICAL SERVICES

ORIGINAL
(Red)

CERTIFICATE OF ANALYSIS

ITAS Cincinnati
11499 Chester Road
Cincinnati, OH 45246
Attn: Mr Michael Taylor

Date: June 14, 1990

NJ Lab Certification ID#: 12064

Job No.: 805317

P.O. Number: 805317

This is the Certificate of Analysis for the following samples:

Client Project ID: PEI - Halby
Date Received: 05/17/90
Number of Samples: 2
Sample Type: Soil

I Samples were labeled as follows:

SAMPLE IDENTIFICATION	LABORATORY #
3741-X02	F0-05-175-01
3741-X03	F0-05-175-02

Reviewed and Approved:

Jackie Redington

Jackie Redington
Project Manager

American Council of Independent Laboratories
International Association of Environmental Testing Laboratories
American Association for Laboratory Accreditation

IT Analytical Services • 165 Fieldcrest Avenue, Edison, NJ 08837 • (201) 225-2000

Company: ITAS Cincinnati
Date: June 14, 1990
Client Job No.: 805317

IT ANALYTICAL SERVICE
EDISON, NJ ORIGINAL
(201) 225-2000 (Red)
Work Order: FO[illegible]-175

SAMPLE ID	3741-X02	3741-X03	
SAMPLED	05/16/90	05/16/90	
TEST			UNITS
Total Solids	44 [0.01]	77 [0.01]	Percent

Company: ITAS Cincinnati
Date: June 14, 1990
Client Job No.: 805317

IT ANALYTICAL SERVICES
EDISON, NJ
(201) 225-2000
ORIGINAL
(Red)
Work Order: F0-05-175

TEST NAME: Pesticides & PCB's

SAMPLE ID: 3741-X02
SAMPLE DATE: 05/16/90
ANALYSIS DATE: 05/22/90

	Results in ug/Kg Dry Wt.	Detection Limit
Aldrin	ND	76
Alpha-BHC	ND	76
Beta-BHC	ND	76
Delta-BHC	ND	76
Gamma-BHC	ND	76
4,4'-DDD	ND	150
4,4'-DDE	ND	150
4,4'-DDT	ND	150
Dieldrin	ND	150
Endosulfan I	ND	150
Endosulfan II	ND	150
Endosulfan Sulfate	ND	150
Endrin	ND	150
Endrin Aldehyde	ND	150
Heptachlor	ND	76
Heptachlor Epoxide	ND	76
Toxaphene	ND	7600
Arochlor 1016	ND	1500
Arochlor 1221	ND	1500
Arochlor 1232	ND	1500
Arochlor 1242	ND	1500
Arochlor 1248	ND	1500
Arochlor 1254	ND	1500
Arochlor 1260	ND	1500
Chlordane	ND	760
Methoxychlor	ND	760

Comments: ND indicates the compound is not detected at the level indicated.

Page: 4

Company: ITAS Cincinnati
Date: June 14, 1990
Client Job No.: 805317

IT ANALYTICAL SERVICES
EDISON, NJ ORIGINAL
(201) 225-2000 (Red)
Work Order: F0-[illegible]-175

TEST NAME: Acid/Base Neutrals

SAMPLE ID: 3741-X02
SAMPLE DATE: 05/16/90
ANALYSIS DATE: 05/18/90

Results in	ug/Kg Dry Wt.	Detection Limit
Acenaphthene	ND	1500
Acenaphthylene	ND	1500
Anthracene	ND	1500
Benzidine	ND	1500
Benzo(a)Anthracene	ND	1500
Benzo(b)Fluoranthene	ND	1500
Benzo(k)Fluoranthene	ND	1500
Benzo(a)Pyrene	ND	1500
Benzo(g,h,i)perylene	ND	1500
bis(2-Chloroethyl)Ether	ND	1500
bis(2-Chloroethoxy)Methane	ND	1500
bis(2-Ethylhexyl)Phthalate	ND	1500
bis(2-Chloroisopropyl)Ether	ND	1500
4-Bromophenyl Phenyl Ether	ND	1500
Butyl Benzyl Phthalate	ND	1500
4-Chloroaniline	ND	1500
2-Chloronaphthalene	ND	1500
4-Chlorophenyl Phenyl Ether	ND	1500
Chrysene	ND	1500
Dibenzo(a,h)anthracene	ND	1500
Dibenzofuran	ND	1500
Di-n-butylphthalate	ND	1500
1,2-Dichlorobenzene	ND	1500
1,3-Dichlorobenzene	ND	1500
1,4-Dichlorobenzene	ND	1500
3,3'-Dichlorobenzidine	ND	1500
Diethylphthalate	ND	1500
Dimethylphthalate	ND	1500
2,4-Dinitrotoluene	ND	1500
2,6-Dinitrotoluene	ND	1500
Di-n-Octylphthalate	ND	1500
1,2-Diphenylhydrazine	ND	1500
Fluoranthene	ND	1500
Fluorene	ND	1500
Hexachlorobenzene	ND	1500

Company: ITAS Cincinnati
Date: June 14, 1990
Client Job No.: 805317

IT ANALYTICAL SERVICES
EDISON, NJ ORIGINAL
(201) 225-2000 (Red)
Work Order: F0-05-175

TEST NAME: Acid/Base Neutrals

SAMPLE ID: 3741-X02
SAMPLE DATE: 05/16/90

Hexachlorobutadiene	ND	1500
Hexachloroethane	ND	1500
Hexachlorocyclopentadiene	ND	1500
Indeno(1,2,3-cd)pyrene	ND	1500
Isophorone	ND	1500
2-Methylnaphthalene	ND	1500
Naphthalene	ND	1500
2-Nitroaniline	ND	7500
3-Nitroaniline	ND	7500
4-Nitroaniline	ND	7500
Nitrobenzene	ND	1500
N-nitroso-dimethylamine	ND	1500
N-Nitrosodipropylamine	ND	1500
N-Nitrosodiphenylamine	ND	1500
Phenanthrene	ND	1500
Pyrene	ND	1500
1,2,4-Trichlorobenzene	ND	1500
Benzoic Acid	ND	7500
Benzyl Alcohol	ND	1500
4-Chloro-3-methylphenol	ND	1500
2-Chlorophenol	ND	1500
2,4-Dichlorophenol	ND	1500
2,4-Dimethylphenol	ND	1500
2,4-Dinitrophenol	ND	7500
4,6-Dinitro-2-methylphenol	ND	7500
2-Methylphenol	ND	1500
4-Methylphenol	ND	1500
2-Nitrophenol	ND	1500
4-Nitrophenol	ND	7500
Pentachlorophenol	ND	7500
Phenol	ND	1500
2,4,5-Trichlorophenol	ND	7500
2,4,6-Trichlorophenol	ND	1500

Comments: ND indicates the compound is not detected at the level indicated.

Page: 6

Company: ITAS Cincinnati
Date: June 14, 1990
Client Job No.: 805317

IT ANALYTICAL SERVICES
EDISON, NJ ORIGINAL
(201) 225-2000 (Red)
Work Order: F0-[illegible]175

TEST NAME: Volatile Organics

SAMPLE ID: 3741-X02
SAMPLE DATE: 05/15/90
ANALYSIS DATE: 05/23/90

	Results in ug/Kg Dry Wt.	Detection Limit
Acrolein	ND	11000
Acrylonitrile	ND	11000
Benzene	ND	1100
Bromoform	ND	1100
Bromomethane	ND	1100
Carbon Tetrachloride	ND	1100
Chlorobenzene	ND	1100
Chlorodibromomethane	ND	1100
Chloroethane	ND	1100
2-Chloroethylvinyl Ether	ND	1100
Chloroform	ND	1100
Chloromethane	ND	1100
Dichlorobromomethane	ND	1100
1,1-Dichloroethane	ND	1100
1,2-Dichloroethane	ND	1100
1,1-Dichloroethene	ND	1100
1,2-Dichloropropane	ND	1100
cis-1-3-Dichloropropene	ND	1100
trans-1-3-Dichloropropene	ND	1100
Ethylbenzene	ND	1100
Methylene Chloride	ND	1100
1,1,2,2-Tetrachloroethane	ND	1100
Tetrachloroethene	ND	1100
Toluene	3000	1100
1,1,1-Trichloroethane	ND	1100
1,1,2-Trichloroethane	ND	1100
Trichloroethene	ND	1100
Trichlorofluoromethane	ND	1100
Vinyl Chloride	ND	1100
Acetone	5500	2300
2-Butanone	9500	2300
Vinyl Acetate	ND	2300
2-Hexanone	ND	2300
4-Methyl-2-Pentanone	ND	2300
Styrene	ND	2300
Xylenes	ND	2300

Page: 7

Company: ITAS Cincinnati
Date: June 14, 1990
Client Job No.: 805317

IT ANALYTICAL SERVICES
EDISON, NJ ORIGINAL
(201) 225-2000 (Red)
Work Order: F0-05-175

TEST NAME: Volatile Organics

SAMPLE ID: 3741-X02
SAMPLE DATE: 05/16/90

Carbon disulfide	5400	2300
Total 1,2-Dichloroethene	ND	1100

Comments: ND indicates the compound is not detected at the level indicated.

Page: 8

Company: ITAS Cincinnati
Date: June 14, 1990
Client Job No.: 805317

IT ANALYTICAL SERVICES
EDISON, NJ
(201) 225-2000
Work Order: F0-[illegible]-175

ORIGINAL
(Red)

TEST NAME: Pesticides & PCB's

SAMPLE ID: 3741-X03
SAMPLE DATE: 05/16/90
ANALYSIS DATE: 05/22/90

	Results in ug/Kg Dry Wt.	Detection Limit
Aldrin	47	21
Alpha-BHC	ND	21
Beta-BHC	ND	21
Delta-BHC	40	21
Gamma-BHC	ND	21
4,4'-DDD	ND	43
4,4'-DDE	ND	43
4,4'-DDT	ND	43
Dieldrin	ND	43
Endosulfan I	ND	43
Endosulfan II	ND	43
Endosulfan Sulfate	ND	43
Endrin	ND	43
Endrin Aldehyde	ND	43
Heptachlor	ND	21
Heptachlor Epoxide	ND	21
Toxaphene	ND	2100
Arochlor 1016	ND	430
Arochlor 1221	ND	430
Arochlor 1232	ND	430
Arochlor 1242	ND	430
Arochlor 1248	ND	430
Arochlor 1254	ND	430
Arochlor 1260	5000	430
Chlordane	ND	210
Methoxychlor	ND	210

Comments: ND indicates the compound is not detected at the level indicated.

Company:	ITAS Cincinnati
Date:	June 14, 1990
Client Job No.:	805317

IT ANALYTICAL SERVICES
EDISON, NJ ORIGINAL
(201) 225-2000 (Red)
Work Order: F0-05-175

TEST NAME: Acid/Base Neutrals

SAMPLE ID: 3741-X03
SAMPLE DATE: 05/16/90
ANALYSIS DATE: 05/18/90

Results in	ug/Kg Dry Wt.	Detection Limit
Acenaphthene	ND	860
Acenaphthylene	ND	860
Anthracene	ND	860
Benzidine	ND	860
Benzo(a)Anthracene	ND	860
Benzo(b)Fluoranthene	2100	860
Benzo(k)Fluoranthene	1200	860
Benzo(a)Pyrene	ND	860
Benzo(g,h,i)perylene	ND	860
bis(2-Chloroethyl)Ether	ND	860
bis(2-Chloroethoxy)Methane	ND	860
bis(2-Ethylhexyl)Phthalate	1300	860
bis(2-Chloroisopropyl)Ether	ND	860
4-Bromophenyl Phenyl Ether	ND	860
Butyl Benzyl Phthalate	ND	860
4-Chloroaniline	ND	860
2-Chloronaphthalene	ND	860
4-Chlorophenyl Phenyl Ether	ND	860
Chrysene	1300	860
Dibenzo(a,h)anthracene	ND	860
Dibenzofuran	ND	860
Di-n-butylphthalate	ND	860
1,2-Dichlorobenzene	ND	860
1,3-Dichlorobenzene	ND	860
1,4-Dichlorobenzene	ND	860
3,3'-Dichlorobenzidine	ND	860
Diethylphthalate	ND	860
Dimethylphthalate	ND	860
2,4-Dinitrotoluene	ND	860
2,6-Dinitrotoluene	ND	860
Di-n-Octylphthalate	ND	860
1,2-Diphenylhydrazine	ND	860
Fluoranthene	1400	860
Fluorene	ND	860
Hexachlorobenzene	ND	860

Company: ITAS Cincinnati
Date: June 14, 1990
Client Job No.: 805317

IT ANALYTICAL SERVICES
EDISON, NJ ORIGINAL
(201) 225-2000 (Red)
Work Order: F0-[illegible]175

TEST NAME: Acid/Base Neutrals

SAMPLE ID: 3741-X03
SAMPLE DATE: 05/16/90

Hexachlorobutadiene	ND	860
Hexachloroethane	ND	860
Hexachlorocyclopentadiene	ND	860
Indeno(1,2,3-cd)pyrene	ND	860
Isophorone	ND	860
2-Methylnaphthalene	ND	860
Naphthalene	ND	860
2-Nitroaniline	ND	4300
3-Nitroaniline	ND	4300
4-Nitroaniline	ND	4300
Nitrobenzene	ND	860
N-nitroso-dimethylamine	ND	860
N-Nitrosodipropylamine	ND	860
N-Nitrosodiphenylamine	ND	860
Phenanthrene	ND	860
Pyrene	2600	860
1,2,4-Trichlorobenzene	ND	860
Benzoic Acid	ND	4300
Benzyl Alcohol	ND	860
4-Chloro-3-methylphenol	ND	860
2-Chlorophenol	ND	860
2,4-Dichlorophenol	ND	860
2,4-Dimethylphenol	ND	860
2,4-Dinitrophenol	ND	4300
4,6-Dinitro-2-methylphenol	ND	4300
2-Methylphenol	ND	860
4-Methylphenol	ND	860
2-Nitrophenol	ND	860
4-Nitrophenol	ND	4300
Pentachlorophenol	ND	4300
Phenol	ND	860
2,4,5-Trichlorophenol	ND	4300
2,4,6-Trichlorophenol	ND	860

Comments: ND indicates the compound is not detected at the level indicated.

Company: ITAS Cincinnati
Date: June 14, 1990
Client Job No.: 805317

IT ANALYTICAL SERVICES
EDISON, NJ ORIGINAL
(201) 225-2000 (Red)
Work Order: FO-05-175

TEST NAME: Volatile Organics

SAMPLE ID: 3741-X03
SAMPLE DATE: 05/16/90
ANALYSIS DATE: 05/22/90

	Results in ug/Kg Dry Wt.	Detection Limit
Acrolein	ND	13000
Acrylonitrile	ND	13000
Benzene	ND	1300
Bromoform	ND	1300
Bromomethane	ND	1300
Carbon Tetrachloride	ND	1300
Chlorobenzene	ND	1300
Chlorodibromomethane	ND	1300
Chloroethane	ND	1300
2-Chloroethylvinyl Ether	ND	1300
Chloroform	ND	1300
Chloromethane	ND	1300
Dichlorobromomethane	ND	1300
1,1-Dichloroethane	ND	1300
1,2-Dichloroethane	ND	1300
1,1-Dichloroethene	ND	1300
1,2-Dichloropropane	ND	1300
cis-1-3-Dichloropropene	ND	1300
trans-1-3-Dichloropropene	ND	1300
Ethylbenzene	ND	1300
Methylene Chloride	ND	1300
1,1,2,2-Tetrachloroethane	ND	1300
Tetrachloroethene	ND	1300
Toluene	11000	1300
1,1,1-Trichloroethane	ND	1300
1,1,2-Trichloroethane	ND	1300
Trichloroethene	ND	1300
Trichlorofluoromethane	ND	1300
Vinyl Chloride	ND	1300
Acetone	4000	2600
2-Butanone	9500	2600
Vinyl Acetate	ND	2600
2-Hexanone	ND	2600
4-Methyl-2-Pentanone	ND	2600
Styrene	ND	2600
Xylenes	ND	2600

Company: ITAS Cincinnati
Date: June 14, 1990
Client Job No.: 805317

TEST NAME: Volatile Organics

SAMPLE ID: 3741-X03
SAMPLE DATE: 05/16/90

Carbon disulfide	ND	2600
Total 1,2-Dichloroethene	ND	1300

Comments: ND indicates the compound is not detected at the level indicated.

ORIGINAL
(Red)

Company: ITAS Cincinnati
Date: June 04, 1990
Client Job No.: 805317
Work Order: F0-05-175

III QUALITY CONTROL

The Determinations were performed in accordance with EPA/NJDEP approved methodology.

Bis(2-Ethylhexyl)phthalate was observed to be present in soil QC blank 203 at a concentration of 172 ug/Kg.

II ANALYTICAL RESULTS/METHODOLOGY

The analytical results for this report are presented by Analytical test. Each set of data will include sample identification information, the analytical results, and the appropriate detection limits. Detection limits may vary due to factors arising from concentration/dilution of the sample and sample matrix. ND denotes that the compound is not detected at or above the indicated detection limit. The methodologies for the analytical results requested are described below.

Pesticides/PCB's (Soil)

The analysis of pesticides and PCB's is based on Test Methods for Evaluating Solid Waste (SW-846), 3rd Edition, Method 3550 and Method 8080. An aliquot of sample is sonicated three times with a 1:1 solution of methylene chloride/acetone and exchanged to hexane. The extracts are dried through sodium sulfate and concentrated. The extract is then separated by gas chromotography and the analytes are measured using an electron capture detector.

Base/Neutral and Acid Extractable Organics - GC/MS (Solid)

The analysis of base/neutral and acid extractables organics is based ob Test Methods for Evaluating Soild Waste (SW-846), 3rd Edition Method 3550 and 8270. An aliquot of sample is sonicated three times with methylene chloride. The extracts are dried through sodium sulfate, concentrated and analyzed by mass spectroscopy.

Volatile Organics - GC/MS (Solid)

For the analysis of volatile organics in soils, SW-846, 3RD Edition, Method 8240 is employed. The volatile organic compounds are introduced into the gas chromatograph by the purge and trap method. The purgeables are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column where the purgeables are trapped. After purging is completed, the sorbent column is heated and backflushed with the inert gas to desorb the purgeables onto a gas chromatographic column. The gas chromatograph is temperature programmed to separate the purgeables which are then detected with a mass spectrometer.

Total Solids

The analysis of total solids is based on Standard Methods, 16th Edition - (209F). A well mixed sample is evaporated in a weighed dish and dried to constant weight. The increase in weight over that of the [illegible] represents the total solids.

INTERNATIONAL
TECHNOLOGY
CORPORATION

ANALYTICAL SERVICES

ORIGINAL
(Red)

CERTIFICATE OF ANALYSIS

ITAS Cincinnati
11499 Chester Road
Cincinnati, OH 45246
Attn: Mr Michael Taylor

Date: June 04, 1990

NJ Lab Certification ID#: 12064

Job No.: 805317

P.O. Number: 805317

This is the Certificate of Analysis for the following samples:

Client Project ID: PEI/Halby
Date Received: 05/17/90
Number of Samples: 1
Sample Type: WATER

I Samples were labeled as follows:

SAMPLE IDENTIFICATION	LABORATORY #
3741-X01	F0-05-174-01

Reviewed and Approved:

Jackie Redington
Jackie Redington
Project Manager

American Council of Independent Laboratories
International Association of Environmental Testing Laboratories
American Association for Laboratory Accreditation

IT Analytical Services • 165 Fieldcrest Avenue, Edison, NJ 08837 • (201) 225-2000

Page: 2

Company: ITAS Cincinnati
Date: June 04, 1990
Client Job No.: 805317

IT ANALYTICAL SERVICES
EDISON, NJ ORIGINAL
(201) 225-2000 (Red)
Work Order: F0-[illegible]174

TEST NAME: Pesticides & PCB's

SAMPLE ID: 3741-X01
SAMPLE DATE: 05/16/90
ANALYSIS DATE: 05/22/90

	Results in ug/L	Detection Limit
Aldrin	ND	0.25
Alpha-BHC	ND	0.25
Beta-BHC	ND	0.25
Delta-BHC	ND	0.25
Gamma-BHC	ND	0.25
4,4'-DDD	ND	0.50
4,4'-DDE	ND	0.50
4,4'-DDT	ND	0.50
Dieldrin	ND	0.50
Endosulfan I	ND	0.50
Endosulfan II	ND	0.50
Endosulfan Sulfate	ND	0.50
Endrin	ND	0.50
Endrin Aldehyde	ND	0.50
Heptachlor	ND	0.25
Heptachlor Epoxide	ND	0.25
Toxaphene	ND	25
Arochlor 1016	ND	5.0
Arochlor 1221	ND	5.0
Arochlor 1232	ND	5.0
Arochlor 1242	ND	5.0
Arochlor 1248	ND	5.0
Arochlor 1254	ND	5.0
Arochlor 1260	ND	5.0
Tech Chlordane	ND	2.5
Methoxychlor	ND	2.5

Comments: ND indicates the compound is not detected at the level indicated.

Company: ITAS Cincinnati
Date: June 04, 1990
Client Job No.: 805317

IT ANALYTICAL SERVICES
EDISON, NJ
(201) 225-2000
Work Order: FO-05-174

ORIGINAL
(Red)

TEST NAME: Acid/Base Neutrals

SAMPLE ID: 3741-X01
SAMPLE DATE: 05/16/90
ANALYSIS DATE: 05/18/90

	Results in ug/L	Detection Limit
Acenaphthene	ND	10
Acenaphthylene	ND	10
Anthracene	ND	10
Benzidine	ND	10
Benzo(a)Anthracene	ND	10
Benzo(b)Fluoranthene	ND	10
Benzo(k)Fluoranthene	ND	10
Benzo(a)Pyrene	ND	10
Benzo(g,h,i)perylene	ND	10
bis(2-Chloroethyl)Ether	ND	10
bis(2-Chloroethoxy)Methane	ND	10
bis(2-Ethylhexyl)Phthalate	31	10
bis(2-Chloroisopropyl)Ether	ND	10
4-Bromophenyl Phenyl Ether	ND	10
Butyl Benzyl Phthalate	ND	10
4-Chloroaniline	ND	10
2-Chloronaphthalene	ND	10
4-Chlorophenyl Phenyl Ether	ND	10
Chrysene	ND	10
Dibenzo(a,h)anthracene	ND	10
Dibenzofuran	ND	10
Di-n-butylphthalate	ND	10
1,2-Dichlorobenzene	ND	10
1,3-Dichlorobenzene	ND	10
1,4-Dichlorobenzene	ND	10
3,3'-Dichlorobenzidine	ND	10
Diethylphthalate	ND	10
Dimethylphthalate	ND	10
2,4-Dinitrotoluene	ND	10
2,6-Dinitrotoluene	ND	10
Di-n-Octylphthalate	ND	10
1,2-Diphenylhydrazine	ND	10
Fluoranthene	ND	10
Fluorene	ND	10
Hexachlorobenzene	ND	10

Page: 4

Company: ITAS Cincinnati
Date: June 04, 1990
Client Job No.: 805317

IT ANALYTICAL SERVICES
EDISON, NJ ORIGINAL
(201) 225-2000 (Red)
Work Order: FO-[illegible]-174

TEST NAME: Acid/Base Neutrals

SAMPLE ID: 3741-X01
SAMPLE DATE: 05/16/90

Hexachlorobutadiene	ND	10
Hexachloroethane	ND	10
Hexachlorocyclopentadiene	ND	10
Indeno(1,2,3-cd)pyrene	ND	10
Isophorone	ND	10
2-Methylnaphthalene	ND	10
Naphthalene	ND	10
2-Nitroaniline	ND	50
3-Nitroaniline	ND	50
4-Nitroaniline	ND	50
Nitrobenzene	ND	10
N-nitroso-dimethylamine	ND	10
N-Nitrosodipropylamine	ND	10
N-Nitrosodiphenylamine	ND	10
Phenanthrene	ND	10
Pyrene	ND	10
1,2,4-Trichlorobenzene	ND	10
Benzoic Acid	ND	50
Benzyl Alcohol	ND	10
4-Chloro-3-methylphenol	ND	10
2-Chlorophenol	ND	10
2,4-Dichlorophenol	ND	10
2,4-Dimethylphenol	ND	10
2,4-Dinitrophenol	ND	50
4,6-Dinitro-2-methylphenol	ND	50
2-Methylphenol	ND	10
4-Methylphenol	ND	10
2-Nitrophenol	ND	10
4-Nitrophenol	ND	50
Pentachlorophenol	ND	50
Phenol	ND	10
2,4,5-Trichlorophenol	ND	50
2,4,6-Trichlorophenol	ND	10

Comments: ND indicates the compound is not detected at the level indicated.

Page: 5

Company: ITAS Cincinnati
Date: June 04, 1990
Client Job No.: 805317

IT ANALYTICAL SERVICES
EDISON, NJ
(201) 225-2000 ORIGINAL (Red)
Work Order: F0-05-174

TEST NAME: Volatile Organics

SAMPLE ID: 3741-X01
SAMPLE DATE: 05/16/90
ANALYSIS DATE: 05/19/90

	Results in ug/L	Detection Limit
Acrolein	ND	10000
Acrylonitrile	ND	10000
Benzene	ND	1000
Bromoform	ND	1000
Bromomethane	ND	1000
Carbon Tetrachloride	ND	1000
Chlorobenzene	ND	1000
Chlorodibromomethane	ND	1000
Chloroethane	ND	1000
2-Chloroethylvinyl Ether	ND	1000
Chloroform	ND	1000
Chloromethane	ND	1000
Dichlorobromomethane	ND	1000
1,1-Dichloroethane	ND	1000
1,2-Dichloroethane	ND	1000
1,1-Dichloroethene	ND	1000
1,2-Dichloropropane	ND	1000
cis-1-3-Dichloropropene	ND	1000
trans-1-3-Dichloropropene	ND	1000
Ethylbenzene	ND	1000
Methylene Chloride	ND	1000
1,1,2,2-Tetrachloroethane	ND	1000
Tetrachloroethene	ND	1000
Toluene	1500	1000
1,1,1-Trichloroethane	ND	1000
1,1,2-Trichloroethane	ND	1000
Trichloroethene	ND	1000
Trichlorofluoromethane	ND	1000
Vinyl Chloride	ND	1000
Acetone	ND	2000
2-Butanone	ND	2000
Vinyl Acetate	ND	2000
2-Hexanone	ND	2000
4-Methyl-2-Pentanone	ND	2000
Styrene	ND	2000
Xylenes	ND	2000

Page: 6

Company: ITAS Cincinnati
Date: June 04, 1990
Client Job No.: 805317

TEST NAME: Volatile Organics

SAMPLE ID: 3741-X01
SAMPLE DATE: 05/16/90

Carbon Disulfide	73000	5000
Total 1,2-Dichloroethene	ND	1000

Comments: ND indicates the compound is not detected at the level indicated.

II ANALYTICAL RESULTS/METHODOLOGY

The analytical results for this report are presented by Analytical test. Each set of data will include sample identification information, the analytical results, and the appropriate detection limits. Detection limits may vary due to factors arising from concentration/dilution of the sample and sample matrix. ND denotes that the compound is not detected at or above the indicated detection limit. The methodologies for the analytical results requested are described below.

Pesticides/PCB's (Water)

The analysis of pesticides and PCB's is based on EPA Method 608. An aliquot of sample is extracted three times with methylene chloride and exchanged to hexane. The extracts are dried through sodium sulfate and concentrated. The extract is then separated by gas chromatography, and the analytes are measured using an electron capture detector.

Base/Neutral and Acid Extractable Organics - GC/MS (Water)

The analysis of base/neutral and acid extractable organics is based on EPA Method 625. An aliquot of sample is serially extracted with methylene chloride at a pH greater than 11.0 and again at a pH less than 2.0 using a separatory funnel. The extracts are dried through sodium sulfate, concentrated and analyzed by mass spectroscopy.

Volatile Organics - GC/MS (Water)

For the analysis of volatile organics, EPA Methods 624 is used. An inert gas is bubbled through a sample contained in a specifically designed purging chamber. The purgeables are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column where the purgeables are trapped. After purging is completed, the sorbent column is heated and backflushed with the inert gas to desorb the purgeables onto a gas chromatographic column. The gas chromatograph is temperature programmed to separate the purgeables which are then detected with a mass spectrometer.

III QUALITY CONTROL

The Determinations were performed in accordance with EPA/NJDEP approved methodology.

Bis(2-Ethylhexyl)phthalate was observed to be present in water QC blank 200 at a concentration of 13 ug/L.

ORIGINAL
(Red)

APPENDIX C-2

ANALYTICAL RESULTS FOR INDICATOR COMPOUNDS IN UNTREATED AND LTTD-TREATED SOILS AND CONDENSATE

ORIGINAL
(Red)

APPENDIX C-2.1

Analytical Results For Indicator Compounds In Untreated Soils

IT INTERNATIONAL TECHNOLOGY CORPORATION

ANALYTICAL SERVICES

ORIGINAL
(Red)

CERTIFICATE OF ANALYSIS

Halby T&E Facility

Date: September 21, 1990

Attn: Judy Hessling (PEI)

Job Number PN 3741-60-2

This is the Certificate of Analysis for the following samples:

Client Project ID:	Halby T&E Facility
Date Received:	June 1, 1990
Work Order:	X0-06-009
Number of Samples:	4
Sample Type:	Soil

I. Introduction

Four soil samples arrived at ITAS Cincinnati on June 1, 1990. The samples were sent for analytical work in support of monitoring work at Halby T&E Facility. The samples were labeled as follows:

Sediment # LT-US-V-A
Sediment # LT-US-V-B
Sediment # LT-US-K-A
Sediment # LT-US-K-B

II. Analytical Results/Methodology

The analytical results for this report are presented by analytical test. Each set of data will include sample identification information, the analytical results, and the appropriate detection limits.

The analyses requested and methods used are listed on Table I. Organic data are not blank corrected; inorganic data are.

Reviewed and Approved by:

Ken Mueller

Ken Mueller
Inorganic Group Leader
006009

American Council of Independent Laboratories
International Association of Environmental Testing Laboratories
American Association for Laboratory Accreditation

Client: Halby T&E Facility
Work Order: X0-06-009
00600901

IT ANALYTICAL SERVICES
CINCINNATI, OH

ORIGINAL
(Red)

III. Quality Control

Immediately following the analytical data for the samples can be found the QA/QC information that pertains to these samples. The purpose of this information is to demonstrate that the data enclosed is scientifically valid and defensible. This QA/QC data is used to assess the laboratory's performance during the analysis of the samples it accompanies. All quantitations were performed from within the calibrated range of the analytical instrument.

As requested, MS/MSD analyses were performed. The results are included in this report.

IV. Comments

This report is being re-issued to replace the report sent September 11, 1990. The original report has incorrect compound names for the semi-volatiles.

Client: Halby T&E Facility
Work Order: X0-06-009
00600902

IT ANALYTICAL SERVICE
CINCINNATI, OH

ORIGINAL
(R

TABLE I

Parameter	Methods for Soils
Sieve Analysis	Performed at FC Broeman
Cyanide	9012
Moisture	ASTM D3173
Ammonia	Water Extraction/350.2
Arsenic	3050/7060
Cobalt	3050/6010
Copper	3050/6010
Zinc	3050/6010
5 Volatile Organic Compounds as specified	8240

Client: Halby T&E Facility
Work Order: X0-06-009
00600903

IT ANALYTICAL SERVICES
CINCINNATI, OH
ORIGINAL
(Red)

Analytical Results, ug/g

Client Sample ID		Sediment # LT-US-K-A	Sediment # LT-US-K-B	
Lab No.		03	04	
Parameter	Units			Detection Limit
Cyanide	ug/g	41.0	32.6	1.0
Moisture	%	20.1	20.8	0.1
Ammonia	ug/g	116	115	4
Arsenic	ug/g	217	248	0.03
Cobalt	ug/g	11.0	11.7	2
Copper	ug/g	285	328	0.3
Zinc	ug/g	1380	1230	0.625

Sieve Analysis

Sieve No. 4	41.01	19.90
Sieve No. 8	4.62	7.18
Sieve No. 16	5.80	7.79
Sieve No. 50	25.20	33.61
Sieve No. 100	10.24	13.25
Sieve No. 200	5.48	7.81
Passing through No. 200	7.65	10.35

Samples were dried before analysis.

Client: Halby T&E Facility
Work Order: X0-06-009
00600905

IT ANALYTICAL SERVICES
CINCINNATI, OH

ORIGINAL
(Red)

Quality Assurance Data

Matrix Spike/Duplicate Recovery Data

Client Sample ID: Sediment # LT-US-K-B
Lab Sample ID: X0-06-009-04

Compound	Amt Added	Sample Amt.	Conc MS	% Recov	Conc MSD	% Recov	RPD
Cyanide	300	32.6	625	99.7	644	106	3
Ammonia	300	115	390	92	370	85	5
Arsenic	200	248	388	70	495	123	24
Cobalt	200	11.7	190	89	183	86	3
Copper	200	328	462	67	446	59	4
Zinc	1200	1230	2430	99.8	2280	87.1	6.5

% Recov = (Conc MS (or MSD)-Sample Amt)/ Amt Added)X 100

RPD = ((Conc.MS-Conc MSD)/(Conc MS + Conc MSD)/2))X 100

Client: Halby T&E Facility
Work Order: X0-06-009
00600904

IT ANALYTICAL SERVICES
CINCINNATI, OH
ORIGINAL
(Red)

Quality Control
Standard Reference Solutions

Analyte	Theoretical Value	Percent Recovery
Cyanide	10.0	93.5, 92.1
Ammonia	300	98.0
Arsenic	0.075	95.1, 103
Chromium	1	97.0
Cobalt	1	102.4
Copper	1	91.4
Zinc	1	97.0

Client: Halby T&E Facility
Work Order: X0-06-009
00600920

IT ANALYTICAL SERVICES
CINCINNATI, OH
(Red)

Volatile Analytical Results, ug/Kg

Client Sample ID	Sediment # LT-US-V-A	Sediment LT-US-V-B		
Lab No.	01	02		
Analyte			Method Blank	Detection Limit
2-Butanone	ND	ND	ND	10
Carbon Disulfide	ND	ND	ND	5
Methylene Chloride	21	24	5	5
Tetrachloroethene	180	150	ND	5
Toluene	370	150	ND	5

ND = Not Detected

lient: Halby T&E Facility
ork Order: X0-06-009
0600921

IT ANALYTICAL SERVICES
CINCINNATI, OH

Volatile Matrix Spike/Duplicate Recovery Data (1)

lient Sample ID: Sediment # LT-US-V-8
ab Sample ID: X0-06-009-02

ompound	Amt Spiked	Sample Result	Conc MS	MS % Recov	Conc MSD	MSD % Recov	RPD
oluene	50	150	180	60	170	40	6
etrachloroethene	50	150	170	40	170	40	0
-Butanone	50	ND	25	50	16	32	44
arbon disulfide	50	ND	48	96	54	108	12
ethylene chloride	50	2+	120	190	130	212	8

% Recov = (Conc MS (or MSD) - Sample Conc) / Conc Added) X 100

RPD = ((Conc.MS-Conc MSD) / (Conc MS + Conc MSD) / 2)) X 100

(1) = The MS/MSD analysis was performed approximately one month after the sample analysis because the project had been placed on hold. These QC results should be used with caution.

Client: Halby T&E Facility
Work Order: X0-06-009
00600910

IT ANALYTICAL SERVICES
CINCINNATI, OH
ORIGINAL
(Red)

Quality Assurance Data

Surrogate Recovery, Percent

Client Sample ID	Lab No.	d4-1,2-Dichloro-ethane	d8-Toluene	p-Bromo-fluoro-benzene
Sediment # LT-US-V-A	01	96	113	75
Sediment # LT-US-V-A	01 Dil	99	103	87
Sediment # LT-US-V-B	02 Re	96	117	86
Sediment # LT-US-V-B	02 MS	97	119*	92
Sediment # LT-US-V-B	02 MSD	99	119*	94
Method Blank	VBLKE6	98	100	97
Method Blank	VBLKE5	102	101	86
Method Blank	VBLKE4	97	99	99

* Surrogate recovery outside acceptance limits. Matrix effect also seen in sample analysis.

Client: Halby T&E Facility
Work Order: X0-06-009
00600930

IT ANALYTICAL SERVICES
CINCINNATI, OH
ORIGINAL
(Red)

Semi-Volatile Analysis Data Sheet, ug/Kg

Client Sample ID	Sediment # LT-US-K-A	Sediment # LT-US-K-B		
Lab No.	03	04	Method Blank SBLK838	Detection Limit
Analyte				
Chrysene	4800	3500	ND	660
Pyrene	7100	8300	ND	660
Fluoranthene	7800	7900	ND	660
benzo(b)fluoranthene	4500	3800	ND	660
benzo(k)fluoranthene	1600	1900	ND	660
benzo(a)pyrene	1900	1700	ND	660

ND = Not Detected

Client: Halby T&E Facility
Work Order: X0-06-009
00600906

ORIGINAL
(Red)

Quality Assurance Data

Semivolatile Matrix Spike/Duplicate Recovery Data

Client Sample ID: Sediment # LT-US-K-B
Lab Sample ID: X0-06-009-04

Compound	Amt Added	Sample Amt.	Conc MS	% Recov	Conc MSD	% Recov
1,2,4 Trichlorobenzene	3300	0	3400	102	4120	124
Acenaphthene	3300	970	6020	152	6300	160
2,4 Dinitrotoluene	3300	0	4050	122	4400	131
Pyrene	3300	8250	12400	124	12700	135
N-Nitrosodi-n-propylamine	3300	0	3700	111	4200	126
1,4 Dichlorobenzene	3300	0	1710	51	3990	120
Pentachlorophenol	6660	0	9460	142	9530	143
Phenol	6660	0	7830	117	7880	118
2-Chlorophenol	6660	0	7060	106	7970	120
4-Chloro-3-methyl phenol	6660	0	8620	129	8420	126
4-Nitrophenol	6660	0	7030	106	6830	103

% Recov = (Conc MS (or MSD)-Sample Amt)/ Amt Added)X 100

RPD = ((Conc.MS-Conc MSD)/(Conc MS + Conc MSD)/2))X 100

Note: Due to a miscommunication, the sample was spiked with the routine internal spiking compounds.

Client: Halby T&E Facility
Work Order: X0-06-009
00600908

IT ANALYTICAL SERVICES
CINCINNATI, OH

ORIGINAL
(Red)

Quality Assurance Data

Semi-Volatile Surrogate Recovery, Percent

Client Sample ID	Lab No.	d5-Nitro benzene	2Fluoro biphenyl	d14-Terphenyl	d6-Phenol	2Fluoro phenol	2,4,6-Tribromo phenol
Sediment LT-US-K-A	03	78	84	56	73	78	69
Sediment LT-US-K-B	04	90	97	82	93	99	110
Method Blank	SBLK838	75	80	73	67	61	39

Associ_tes, Inc.

PN# 3741-60-2

CHAIN-OF-CUSTODY RECORD

PROJECT NA... NUMBER Halby Chemical Site Treatability Studies LAB DESTINATION ITAS Cincinnati

SAMPLE TEAM MEMBERS Steve Giti-Pour, Michael L. Smith CARRIER/WAYBILL NO.

SAMPLE NUMBER	SAMPLE LOCATION AND DESCRIPTION	DATE AND TIME COLLECTED	SAMPLE TYPE	CONTAINER TYPE	CONDITION ON RECEIPT (NAME AND DATE)
LT-US-V-A1	From drums located	5/31/90 14:30	Sediment	4 OZ Glass Jar	Samples were hand
LT-US-V-A2	in soil area.	↓	soil ↓	↓	delivered and received
LT-US-V-B1					in good condition.
LT-US-V-B2					
LT-US-K-A1				8 OZ Glass Jar	Dawn Webber
LT-US-K-B1					June 1, 1990

SPECIAL INSTRUCTIONS:

POSSIBLE SAMPLE HAZARDS

SIGNATURES: (NAME, COMPANY, DATE, AND TIME)

1. RELINQUISHED BY: Steve Giti-Pour
   RECEIVED BY: Dawn Webber 6-1-90 11:00 AM
2. RELINQUISHED BY:
   RECEIVED BY:
3. RELINQUISHED BY:
   RECEIVED BY:
4. RELINQUISHED BY:
   RECEIVED BY:

ORIGINAL (Red)

WHITE - To accompany samples
YELLOW - Field copy

600-90-02

PEI Associates, Inc.

PN#3741-60-2

REQUEST FOR ANALYSIS

PROJECT NAME Halby Chemical Site Treatability Studies

PROJECT NUMBER 3741-60-2

PROJECT MANAGER Ms. Judy Hessling

BILL TO 3741-60-2

PURCHASE ORDER NO. ______

DATE SAMPLES SHIPPED ITAS Cincinnati (6-1-90)

LAB DESTINATION ITAS Cincinnati

LABORATORY CONTACT Craig Caldwell

SEND LAB REPORT TO Judy Hessling

DATE REPORT REQUIRED ______

PROJECT CONTACT ______

PROJECT CONTACT PHONE NO. ______

SAMPLE NO.	SAMPLE TYPE	SAMPLE VOLUME	PRESERVATIVE	REQUESTED TESTING PROGRAM	SPECIAL INSTRUCTIONS
LT-US-V-A1	Sediment	4oz glass Jar	None	VOAs (see the attached	page for further information)
LT-US-V-A2	↓	↓	↓		
LT-US-V-B1	↓	↓	↓		
LT-US-V-B2	↓	↓	↓		
LT-US-K-A1	Sediment	8oz glass Jar	↓	BNAs, Metals, PCBs, Pest.	(See the attached page
LT-US-K-B1	↓	↓	↓		for further information)
					BNAs & metals get TCLP
					extract as well as Total

TURNAROUND TIME REQUIRED: (RUSH MUST BE APPROVED BY PROJECT MANAGER)

NORMAL X RUSH ______ (SUBJECT TO RUSH SURCHARGE)

POSSIBLE HAZARD IDENTIFICATION: (PLEASE INDICATE IF SAMPLE(S) ARE HAZARDOUS MATERIALS AND/OR SUSPECTED TO CONTAIN HIGH LEVELS OF HAZARDOUS SUBSTANCES)

NONHAZARD ____ FLAMMABLE ____ SKIN IRRITANT ____ HIGHLY TOXIC ____ OTHER ____ (PLEASE SPECIFY)

SAMPLE DISPOSAL: (PLEASE INDICATE DISPOSITION OF SAMPLE FOLLOWING ANALYSIS. LAB WILL CHARGE FOR PACKING, SHIPPING, AND DISPOSAL)

RETURN TO CLIENT ____ DISPOSAL BY LAB ____

FOR LAB USE ONLY

RECEIVED BY Dawn Webber DATE/TIME June 1, 1990 11:00AM

WHITE - Original, to accompany samples
YELLOW - Field copy

ORIGINAL
(Red)

[illegible]

Halby Treatability Studies
Indicator Compounds

<u>Volatile Organics</u>
Toluene
~~Acetone~~
2-Butanone
Carbon disulfide

<u>Semivolatile Organics</u>
Chrysene
Pyrene
Fluoranthene
Benzo(b)fluoranthene
Benzo(k)fluoranthene

<u>PCB's/Pesticides</u>
Aroclor 1260
Aldrin
δ-BHC

Metals ~~Inorganics~~
Arsenic
Chromium (total, ~~VI~~)
~~Lead~~
Zinc
~~Nickel~~
Copper

Other Parameters
Moisture Content
Particle-size analysis
pH
Total organic Carbon
~~VOCs~~
TCLP

ORIGINAL
(Red)

APPENDIX C-2.2

Analytical Results For Indicator Compounds In Thermally-Treated Soils

IT INTERNATIONAL TECHNOLOGY CORPORATION

ANALYTICAL SERVICES

ORIGINAL
(Red)

CERTIFICATE OF ANALYSIS

PEI Associates, Inc. Date: September 4, 1990

Attn: Ms. Judy Hessling

Job Number PN 3761-60-2

This is the Certificate of Analysis for the following samples:

Client Project ID: Halby T&E Facility
Date Received: July 3, 1990
Work Order: X0-07-030
Number of Samples: 1
Sample Type: Water

I. Introduction

One water sample arrived at ITAS Cincinnati on July 3, 1990. The sample was sent for analytical work in support of monitoring work for Halby Chemical Site Treatability. The sample was labeled as Water # SS-CON-V-500-30.

II. Analytical Results/Methodology

The analytical results for this report are presented by analytical test. Each set of data will include sample identification information, the analytical results, and the appropriate detection limits.

The analyses requested are listed on Table I of this report.

Reviewed and Approved by: (for KM)

Craig

Ken Moeller
Inorganic Group Leader
007030

ORIGINAL
(Red)

III. Quality Control

Immediately following the analytical data for the samples can be found the QA/QC information that pertains to these samples. The purpose of this information is to demonstrate that the data enclosed is scientifically valid and defensible. This QA/QC data is used to assess the laboratory's performance during the analysis of the samples it accompanies. All quantitations were performed from within the calibrated range of the analytical instrument.

IV. Comments

The volatiles portion of the sample was received unpreserved.

Client: Halby T&E Facility
Work Order: X0-07-030
00703001

IT ANALYTICAL SERVICES
CINCINNATI, OH

ORIGINAL
(Red)

TABLE I

Arsenic	7060
Chromium	3010/6010
Cobalt	3010/6010
Copper	3010/6010
Zinc	3010/6010
5 Volatile Organic Compounds as Specified	8240
6 Semi-Volatile Organic Compounds as Specified	8270

Client: Halby T&E Facility
Work Order: X0-07-030
00703003

IT ANALYTICAL SERVICES
CINCINNATI, OH

ORIGINAL
(Red)

Analytical Results, mg/L

Client Sample ID	Water # SS-CON-V-500-30	
Lab No.	01	
Analyte		Detection Limit
Arsenic	0.0165	0.0005
Chromium	0.033	0.007
Cobalt	ND	0.03
Copper	0.142	0.005
Zinc	0.533	0.02

ND = Not Detected

Quality Control
Standard Reference Solutions

Analyte	Theoretical Value	Percent Recovery
Arsenic	0.0075	92.8
Chromium	1.0	93.0
Cobalt	1.0	93.7
Copper	1.0	91.6
Zinc	1.0	88.6

Client: Halby T&E Facility
Work Order: X0-07-030
00703005

IT ANALYTICAL SERVICES
CINCINNATI, OH

ORIGINAL
(Red)

Volatile Organics Analysis Data Sheet, ug/L

Client Sample ID	Water # SS-CON-V-500-30		
Lab No.	01		
Analyte		Method Blank	Detection Limit
2-Butanone	170	ND	10
Carbon Disulfide	ND	ND	5
Methylene Chloride	ND	ND	5
Tetrachloroethene	ND	ND	5
Toluene	ND	ND	5

ND = Not Detected

Client: Halby T&E Facility
Work Order: X0-07-030
00703030

IT ANALYTICAL SERVICES
CINCINNATI, OH

ORIGINAL (Red)

Semi-Volatile Analysis Data Sheet, ug/L

Client Sample ID	Water # SS-CON-V-500-30		
Lab No.	01		
Analyte		Method Blank	Detection Limit
Chrysene	14	ND	10
Pyrene	140	ND	10
Fluoranthene	190	ND	10
benzo(b)pyrene	ND	ND	10
benzo(k)pyrene	ND	ND	10
benzo(a)pyrene	ND	ND	10

ND = Not Detected

Client: Halbe T&E Facility
Work Order: X0-07-030
00703006

IT ANALYTICAL SERVICES
CINCINNATI, OH
ORIGINAL
(Red)

Quality Assurance Data

Volatile
Surrogate Recovery, Percent

Client Sample ID	Lab No.	d4-1,2-Dichloro-ethane	d8-Toluene	p-Bromo-fluoro-benzene
Water # SS-CON-V-500-30	030-01	95	95	102
Method Blank	VBLKT6	98	94	97

Client: Halby T&E Facility
Work Order: X0-07-030
00703020

IT ANALYTICAL SERVICES
CINCINNATI, OH

ORIGINAL
(Red)

Quality Assurance Data

Semi-Volatile
Surrogate Recovery, Percent

Client Sample ID	Lab No.	d5-Nitro benzene	2Fluoro biphenyl	d14-Terphenyl	d6-Phenol	2Fluoro phenol	2,4,6-Tribrom phenol
Water # SS-CON-V-500-30	030-01	61	55	13*	67	64	6
Water # SS-CON-V-500-30	030-01 Re	86	74	19*	90	0*	7
Method Blank	SBLK832	69	73	71	72	72	7
Method Blank	SBLK701	75	81	93	67	88	9

* Surrogate recoveries outside acceptance windows. Reanalysis confirms matrix effects.

APPENDIX C-2.3

Analytical Results For Indicator Compounds In Condensate Sample From 500°F/30 Minute Test Runs

IT INTERNATIONAL TECHNOLOGY CORPORATION

ANALYTICAL SERVICES

ORIGINAL
(Red)

CERTIFICATE OF ANALYSIS

PEI Associates, Inc. Date: September 4, 1990

Attn: Ms. Judy Hessling

Job Number PN 3761-60-2

This is the Certificate of Analysis for the following samples:

Client Project ID:	Halby T&E Facility
Date Received:	June 18, 1990
Work Order:	X0-06-197
Number of Samples:	22
Sample Type:	Sediment

I. Introduction

Twenty two sediment samples arrived at ITAS Cincinnati on June 18, 1990. The samples were sent for analytical work in support of monitoring work for Halby T&E Facility. The samples were labeled as follows:

LT-S-300-15-V-A
LT-S-300-15-V-B
LT-S-300-30-V-A
LT-S-300-30-V-B
LT-S-500-15-V-A
LT-S-500-15-V-B
LT-S-500-30-V-A
LT-S-500-30-V-B
LT-S-300-15-K-A
LT-S-300-15-K-B
LT-S-300-15-V-B
LT-S-500-15-K-B
LT-S-300-30-K-A
LT-S-300-30-K-B
LT-S-500-30-K-A
LT-S-500-30-K-B

Samples LT-S-300-30-K-A, LT-S-300-30-K-B, LT-S-500-15-K-A, LT-S-500-15-B, LT-S-500-30-K-A, and LT-S-500-50-K-B were placed on hold by client; no analyses were performed on these samples.

II. Analytical Results/Methodology

The analytical results for this report are presented by analytical test. Each set of data will include sample identification information, the analytical results, and the appropriate detection limits.

The analyses requested are listed on Table I of this report.

Reviewed and Approved by: Craig (for KM)

Ken Mueller
Inorganic Group Leader
006197

American Council of Independent Laboratories
International Association of Environmental Testing Laboratories
American Association for Laboratory Accreditation

ORIGIN (Red)

III. Quality Control

Immediately following the analytical data for the samples can be found the QA/QC information that pertains to these samples. The purpose of this information is to demonstrate that the data enclosed is scientifically valid and defensible. This QA/QC data is used to assess the laboratory's performance during the analysis of the samples it accompanies. All quantitations were performed from within the calibrated range of the analytical instrument.

Client: Halby T&E Facility
Work Order: X0-06-197
00619701

IT ANALYTICAL SERVICES
CINCINNATI, OH

ORIGINAL
(Red)

TABLE I

Mositure	ASTM D3173
Ammonia	Water Extraction/350.2
Arsenic	3050/7060
Chromium	3050/6010
Cobalt	3050/6010
Copper	3050/6010
Cyanide	9012
Zinc	3050/6010
5 Volatile Organic Compounds as Specified	8240
6 Semi-Volatile Organic Compounds as Specified	8270

Client: Halby T&E Facility
Work Order: X0-06-197
00619720

IT ANALYTICAL SERVICES
CINCINNATI, OH

ORIGIN
(Red)

Analytical Results

Client Sample ID	Lab No.	Percent Moisture
Sediment # LT-S-300-15-K-A	197-09	0.05
Sediment # LT-S-300-15-K-B	197-10	0.21
Sediment # LT-S-500-15-K-A	197-11	0.09
Sediment # LT-S-500-15-K-B	197-12	0.05
Sediment # LT-S-300-30-K-A	197-13	0.24
Sediment # LT-S-300-30-K-A	197-14	0.45
Sediment # LT-S-500-30-K-A	197-15	0.25
Sediment # LT-S-500-30-K-B	197-16	0.10

Client: Halby T&E Facility
Work Order: X0-06-197
00619703

IT ANALYTICAL SERVICES
CINCINNATI, OH

ORIGINAL
(Red)

Analytical Results, ug/g

Client Sample ID	Sediment # LT-S-300-15-K-A	Sediment # LT-S-300-15-K-B	
Lab No.	09	10	
Analyte			Detection Limit
Ammonia	196	195	4
Arsenic	288	283	0.03
Chromium	317	310	0.4
Cobalt	19	19	2
Copper	377	406	0.3
Cyanide	16.0	22.8	1.0
Zinc	1410	1580	0.7

Client Sample ID	Sediment # LT-S-500-15-K-A	Sediment # LT-S-500-15-K-B	
Lab No.	11	12	
Analyte			Detection Limit
Ammonia	275	287	4
Arsenic	349	300	0.03
Chromium	272	234	0.4
Cobalt	17.9	16.0	2
Copper	390	379	0.3
Cyanide	6.26	6.19	1.0
Zinc	1480	1600	0.7

ND = Not Detected

Client: Halby T&E Facility
Work Order: X0-06-197
00619704

IT ANALYTICAL SERVICES
CINCINNATI, OH

ORIGINAL
(Red)

Analytical Results, ug/g

Client Sample ID	Sediment # LT-S-300-30-K-A	Sediment # LT-S-300-30-K-B	
Lab No.	13	14	
Analyte			Detection Limit
Ammonia	196	185	4
Arsenic	344	322	0.03
Chromium	297	308	0.4
Cobalt	17.1	17.5	2
Copper	351	398	0.3
Cyanide	37.4	43.5	1.0
Zinc	1130	1490	0.7

Client Sample ID	Sediment # LT-S-500-30-K-A	Sediment # LT-S-500-30-K-B	
Lab No.	15	16	
Analyte			Detection Limit
Ammonia	224	286	4
Arsenic	316	313	0.03
Chromium	255	274	0.4
Cobalt	17.8	17.7	2
Copper	363	370	0.3
Cyanide	6.71	9.30	1.0
Zinc	1180	1330	0.7

ND = Not Detected

Client: Halby T&E Facility
Work Order: X0-06-197
00619705

IT ANALYTICAL SERVICES
CINCINNATI, OH

ORIGINAL
(Red)

Quality Control
Standard Reference Solutions

Analyte	Theoretical Value, ug/g	Percent Recovery
Ammonia	300	97.5
Arsenic	0.075	103
Chromium	1	99.6
Cobalt	1	101
Copper	1	92.8
Cyanide	10	93.5, 92.1
Zinc	1	89.5

Client: Halby T&E Facility
Work Order: X0-06-197
00619709

IT ANALYTICAL SERVICES
CINCINNATI, OH

ORIGINAL
(Red)

Volatile Organics Analysis Data Sheet,
ug/Kg

Client Sample ID	Sediment # LT-S-300-15-V-A	Sediment # LT-S-300-15-V-B
Lab No.	01	02
Analyte		
2-Butanone	190	220
Carbon Disulfide	17	22
Methylene Chloride	180	190
Tetrachloroethene	5	7
Toluene	160	140

Client Sample ID	Sediment # LT-S-300-30-V-A	Sediment # LT-S-300-30-V-B
Lab No.	03	04
Analyte		
2-Butanone	300	220
Carbon Disulfide	23	7
Methylene Chloride	110	46
Tetrachloroethene	8	6
Toluene	130	60

ND = Not Detected

Client: Halby T&E Facility
Work Order: X0-06-197
00619710

IT ANALYTICAL SERVICES
CINCINNATI, OH

ORIGINAL
(Red)

Volatile Organics Analysis Data Sheet,
ug/Kg

Client Sample ID	Sediment # LT-S-500-15-V-A	Sediment # LT-S-500-15-V-B
Lab No.	05	06
Analyte		
2-Butanone	29	39
Carbon Disulfide	10	6
Methylene Chloride	310	200
Tetrachloroethene	6	5
Toluene	170	140

Client Sample ID	Sediment # LT-S-500-30-V-A	Sediment # LT-S-500-30-V-B
Lab No.	07	08
Analyte		
2-Butanone	15	43
Carbon Disulfide	ND	10
Methylene Chloride	79	680
Tetrachloroethene	5	5
Toluene	5	62

Client Sample ID

Lab No.

Analyte	Method Blank	Detection Limit
2-Butanone	ND	10
Carbon Disulfide	ND	5
Methylene Chloride	ND	5
Tetrachloroethene	ND	5
Toluene	ND	5

ND = Not Detected

Client: Halby T&E Facility
Work Order: X0-06-197
00619707

IT ANALYTICAL SERVICES
CINCINNATI, OH

Quality Assurance Data

Volatiles
Surrogate Recovery, Percent

Client Sample ID	Lab No.	d4-1,2-Dichloro-ethane	d8-Toluene	p-Bromo-fluoro-benzen
Sediment # LT-S-300-15-V-A	197-01	94	124*	8
Sediment # LT-S-300-15-V-A	197-01 Re	115	113	8
Sediment # LT-S-300-15-V-A	197-01 Re 2	92	132*	7
Sediment # LT-S-300-15-V-A	197-01 Dil	99	105	9
Sediment # LT-S-300-15-V-B	197-02	101	134*	7
Sediment # LT-S-300-15-V-B	197-02 Re	95	121*	8
Sediment # LT-S-300-15-V-B	197-02 Dil	97	108	8
Sediment # LT-S-300-30-V-A	197-03	105	127*	7
Sediment # LT-S-300-30-V-A	197-03 Re	98	114	8
Sediment # LT-S-300-30-V-A	197-03 Dil	93	106	9
Sediment # LT-S-300-30-V-A	197-03 Dil 2	92	112	8
Sediment # LT-S-300-30-V-B	197-04	108	137*	7
Sediment # LT-S-300-30-V-B	197-04 Re	98	120*	7
Sediment # LT-S-300-30-V-B	197-04 Dil	96	107	9
Sediment # LT-S-500-15-V-A	197-05	114	185*	5
Sediment # LT-S-500-15-V-A	197-05 Re	90	152*	5
Sediment # LT-S-500-15-V-A	197-05 Dil	95	135*	7
Sediment # LT-S-500-15-V-B	197-06	102	174*	5
Sediment # LT-S-500-15-V-B	197-06 Re	100	149*	6
Sediment # LT-S-500-15-V-B	197-06 Dil	90	132*	7
Sediment # LT-S-500-30-V-A	197-07	96	154*	5
Sediment # LT-S-500-30-V-A	197-07 Re	95	147*	6
Sediment # LT-S-500-30-V-B	197-08	95	144*	5
Sediment # LT-S-500-30-V-B	197-08 Re	92	140*	6
Sediment # LT-S-500-30-V-B	197-08 Dil	98	157*	6
Method Blank	VBLKH9	101	92	9
Method Blank	VBLKI0	101	94	9
Method Blank	VBLKV1	96		9

* Surrogates outside of limits. Matrix effects proven by reanalysis.

Client: Halby T&E Facility
Work Order: X0-06-197
00619730

IT ANALYTICAL SERVICES
CINCINNATI, OH

ORIGINAL
(Red)

Semi-Volatile Analysis Data Sheet,
ug/Kg

Client Sample ID	**Sediment** **# LT-S-300-15-K-A**	**Sediment** **# LT-S-300-15-K-B**
Lab No.	09	10
Analyte		
Chrysene	2200	2400
Pyrene	1400	2400
Fluoranthene	3700	4600
benzo(b)pyrene	2500	3000
benzo(k)pyrene	1300	1500
benzo(a)pyrene	ND	ND

Client Sample ID	**Sediment** **# LT-S-500-15-K-A**	**Sediment** **# LT-S-500-15-K-B**
Lab No.	11	12
Analyte		
Chrysene	ND	ND
Pyrene	ND	ND
Fluoranthene	ND	ND
benzo(b)pyrene	ND	ND
benzo(k)pyrene	ND	ND
benzo(a)pyrene	ND	ND

ND = Not Detected

Client: Halby T&E Facility
Work Order: X0-06-197
00619731

IT ANALYTICAL SERVICES
CINCINNATI, OH

Semi-Volatile Analysis Data Sheet, ug/Kg

Client Sample ID	Sediment # LT-S-300-30-K-A	Sediment # LT-S-300-30-K-B
Lab No.	13	14
Analyte		
Chrysene	3200	4900
Pyrene	5000	ND
Fluoranthene	6400	ND
benzo(b)pyrene	4000	ND
benzo(k)pyrene	1800	ND
benzo(a)pyrene	910	2700

Client Sample ID	Sediment # LT-S-500-30-K-A	Sediment # LT-S-500-30-K-B
Lab No.	15	16
Analyte		
Chrysene	ND	ND
Pyrene	ND	ND
Fluoranthene	ND	ND
benzo(b)pyrene	ND	ND
benzo(k)pyrene	ND	ND
benzo(a)pyrene	ND	ND

Analyte	Method Blank SBLK638	Detection Limit
Chrysene	ND	660
Pyrene	ND	660
Fluoranthene	ND	660
benzo(b)pyrene	ND	660
benzo(k)pyrene	ND	660
benzo(a)pyrene	ND	660

ND = Not Detected

Client: Halby T&E Facility
Work Order: X0-06-197
00619706

IT ANALYTICAL SERVICES
CINCINNATI, OH

ORIGINAL
(Red)

Quality Assurance Data

		Semi-Volatiles Surrogate Recovery, Percent					
Client Sample ID	Lab No.	d5-Nitro benzene	2Fluoro biphenyl	d14-Terphenyl	d6-Phenol	2Fluoro phenol	2,4,6-Tribromo phenol
Sediment LT-S-300-15-K-A	09	85	94	86	85	86	77
Sediment LT-S-300-15-K-B	10	86	95	76	86	90	83
Sediment LT-S-500-15-K-A	11	82	84	79	72	65	52
Sediment LT-S-500-15-K-B	12	79	83	83	74	68	56
Sediment LT-S-300-30-K-A	13	88	100	78	88	96	92
Sediment LT-S-300-30-K-B	14	85	94	82	87	92	209*
Sediment LT-S-500-30-KV-A	15	90	96	94	82	72	49
Sediment LT-S-500-30-K-B	16	92	98	94	86	88	69
Method Blank	SBLK838	75	80	73	67	61	39

* Surrogate recoveries outside acceptance windows. Since this is an acid surrogate and all targets are neutrals, the data is unaffected.

APPENDIX C-3

ANALYTICAL RESULTS FOR METAL INDICATOR COMPOUNDS IN TCLP EXTRACTS FROM UNTREATED AND S/S-TREATED SOILS, AND UCS TEST RESULTS FOR SOLIDIFIED PRODUCTS

APPENDIX C-3.1

Analytical Results For Heavy Metals In TCLP Extracts From Untreated (Raw) Soils, LTTD-Treated Soils, And S/S-Treated Soils

INTERNATIONAL
TECHNOLOGY
CORPORATION

ANALYTICAL SERVICES

ORIGINAL
(Red)

CERTIFICATE OF ANALYSIS

PEI Associates, Inc. Date: September 19, 1990

Attn: Ms. Judy Hessling

Job Number PN 3761-60-2

This is the Certificate of Analysis for the following samples:

Client Project ID:	Halby T&E Facility
Date Received:	August 3, 1990
Work Order:	X0-08-022
Number of Samples:	20
Sample Type:	Soil

I. Introduction

Twenty soil samples arrived at ITAS Cincinnati on August 3, 1990. The samples were sent for analytical work in support of Halby T&E Facility. The samples were labeled as follows:

Soil # SS-S-PC-O.25-TC-A
Soil # SS-S-PC-O.25-TC-B

Soil # SS-S-PC-04-TC-A
Soil # SS-S-PC-04-TC-B

Soil # SS-S-AS-1-TC-A
Soil # SS-S-AS-1-TC-B

Soil # SS-S-AS-O.5-TC-A
Soil # SS-S-AS-O.5-TC-B

Soil # SS-S-PC-O.4-TC-BLK
Soil # SS-S-PC-O.25-TC-BLK

Soil # SS-S-AS-1-TC-Blank
Soil # SS-S-AS-O.5-TC-BLK

Soil # SS-SR-500-30-A
Soil # SS-SR-500-30-B

Soil # SS-US-A
Soil # SS-US-B

Soil # SS-US-PC-O.4-TC-A
Soil # SS-US-PC-O.4-TC-B

Soil # SS-US-PC-O.25-TC-A
Soil # SS-US-PC-O.25-TC-B

Reviewed and Approved by: (for KM)

Ken Mueller
Inorganic Group Leader
008022

American Council of Independent Laboratories
International Association of Environmental Testing Laboratories
American Association for Laboratory Accreditation

IT Analytical Services • 11499 Chester Road • Cincinnati, OH 45246 • 513-782-4600

II. Analytical Results/Methodology

The analytical results for this report are presented by analytical test. Each set of data will include sample identification information, the analytical results, and the appropriate detection limits.

The analyses requested on a total basis for samples Soil # SS-SR-500-30-A, Soil # SS-SR-500-30-B, Soil # SS-US-A and Soil # SS-US-B included:

* pH by EPA Method 9045
* Cyanide by EPA Method 9012
* Sulfide by EPA Method 9030
* Ammonia by EPA Method 350.2
* Total Organic Carbon by EPA Method 9060

The analyses requested on all samples included the following metals on a TCLP leachate The leaching was performed at ITAS according to EPA Method 1311.

* Arsenic by Graphite Furnace Atomic Absorption; EPA Method 7060
* Mercury by Cold Vapor Atomic Absorption; EPA Method 7470
* Chromium, Cobalt, Copper and Zinc by Inductively Coupled Plasma Spectroscopy; EPA Method 6010

III. Quality Control

Immediately following the analytical data for the samples can be found the QA/QC information that pertains to these samples. The purpose of this information is to demonstrate that the data enclosed is scientifically valid and defensible. This QA/QC data is used to assess the laboratory's performance during the analysis of the samples it accompanies. All quantitations were performed from within the calibrated range of the analytical instrument.

As requested, a matrix spike/matrix spike duplicate analyses was performed on sample # SS-S-AS-0.5-TC-B for all metals.

Client: Halby T&E Facility
Work Order: XO-08-022
00802202

IT ANALYTICAL SERVICES
CINCINNATI, OH

ORIGINAL
(Red)

Analytical Results, mg/L

Client Sample ID	Soil # SS-S-PC-0.25-TC-A	Soil # SS-S-PC-0.25-TC-B	
Lab No.	01	02	
Analyte			Detection Limit
Arsenic	0.0741	0.0701	0.005
Chromium	0.132	0.121	0.003
Cobalt	ND	ND	0.03
Copper	0.037	0.025	0.005
Mercury	ND	ND	0.0001
Zinc	ND	ND	0.02

Client Sample ID	Soil # SS-S-PC-04-TC-A	Soil # SS-S-PC-04-TC-B	
Lab No.	03	04	
Analyte			Detection Limit
Arsenic	0.0131	0.0121	0.005
Chromium	0.17	0.14	0.003
Cobalt	ND	ND	0.03
Copper	0.440	0.371	0.005
Mercury	ND	ND	0.0001
Zinc	ND	ND	0.02

Client Sample ID	Soil # SS-S-AS-1-TC-A	Soil # SS-S-AS-1-TC-B	
Lab No.	05	06	
Analyte			Detection Limit
Arsenic	ND	ND	0.005
Chromium	ND	ND	0.003
Cobalt	ND	ND	0.03
Copper	0.013	0.015	0.005
Mercury	ND	ND	0.0001
Zinc	0.546	0.276	0.02

Client: Halby T&E Facility
Work Order: X0-08-022
00802203

IT ANALYTICAL SERVICE
CINCINNATI, OH

Analytical Results, mg/L

Client Sample ID	Soil # SS-S-AS-0.5-TC-A	Soil # SS-S-AS-0.5-TC-B	
Lab No.	07	08	
Analyte			Dete Li
Arsenic	ND	ND	0.
Chromium	0.006	0.008	0.
Cobalt	ND	ND	0.
Copper	0.165	0.017	0.
Mercury	ND	ND	0.0
Zinc	0.553	0.329	0.

Client Sample ID	Soil # SS-S-PC-0.4-TC-BLK	Soil # SS-S-PC-0.25-TC-BLK	
Lab No.	09	10	
Analyte			Dete Li
Arsenic	0.0081	0.0071	0.
Chromium	0.141	0.142	0.
Cobalt	ND	ND	0.
Copper	ND	ND	0.
Mercury	ND	ND	0.0
Zinc	ND	ND	0.

Client Sample ID	Soil # SS-S-AS-1-TC-Blank	Soil # SS-S-AS-0.5-TC-BLK	
Lab No.	11	12	
Analyte			Dete Li
Arsenic	ND	ND	0.
Chromium	ND	ND	0.
Cobalt	ND	ND	0.
Copper	ND	ND	0.
Mercury	ND	ND	0.0
Zinc	0.335	0.29	0.

ND = Not Detected

Client: Halby T&E Facility
Work Order: X0-08-022
00802204

IT ANALYTICAL SERVICES
CINCINNATI, OH

ORIGINAL
(Red)

Analytical Results

Client Sample ID		Soil # SS-SR-500-30-A	Soil # SS-SR-500-30-B	
Lab No.		13	14	
Analyte	Units			Detection Limit
pH	S.U.	4.64	4.62	NA
TOC	ug/g	12000	24000	100
Ammonia-N	ug/g	220	270	5
Cyanide	ug/g	8.80	9.85	1
Sulfide	ug/g	130	200	51
Arsenic	mg/L	0.0281	0.0331	0.005
Chromium	mg/L	ND	ND	0.003
Cobalt	mg/L	0.044	0.060	0.03
Copper	mg/L	0.400	0.419	0.005
Mercury	mg/L	ND	ND	0.0001
Zinc	mg/L	6.1	8.05	0.02

Client Sample ID		Soil # SS-US-A	Soil # SS-US-B	
Lab No.		15	16	
Analyte	Units			Detection Limit
pH	S.U.	5.33	5.71	NA
TOC	ug/g	17000	17000	100
Ammonia-N	ug/g	85	120	5
Cyanide	ug/g	50.2	43.4	1
Sulfide	ug/g	190	81	51
Arsenic	mg/L	0.0131	0.0141	0.005
Chromium	mg/L	ND	ND	0.003
Cobalt	mg/L	ND	ND	0.03
Copper	mg/L	0.201	0.187	0.005
Mercury	mg/L	ND	ND	0.0001
Zinc	mg/L	1.62	1.51	0.02

ND = Not Detected

Client: Halby T&E Facility
Work Order: XO-08-022
00802205

IT ANALYTICAL SERVICE
CINCINNATI, OH

ORIGINA
(Red)

Analytical Results, mg/L

Client Sample ID	Soil # SS-US-PC-0.4-TC-A	Soil # SS-US-PC-0.4-TC-B	
Lab No.	17	18	Detec Lim
Analyte			
Arsenic	0.0295	0.0285	0.0
Chromium	0.115	0.123	0.0
Cobalt	ND	ND	0.
Copper	0.071	0.077	0.0
Mercury	ND	ND	0.00
Zinc	ND	ND	0.

Client Sample ID	Soil # SS-US-PC-0.25-TC-A	Soil # SS-US-PC-0.25-TC-B	
Lab No.	19	20	Detec Lim
Analyte			
Arsenic	0.0515	0.0455	0.0
Chromium	0.213	0.204	0.0
Cobalt	ND	ND	0.
Copper	0.846	0.814	0.0
Mercury	ND	ND	0.00
Zinc	ND	ND	0.

ND = Not Detected

Client: Halby T&E Facility
Work Order: X0-08-022
00802206

ORIGINAL
(Red)

Quality Control
Standard Reference Solutions

Analyte	Theoretical Value	Percent Recovery
Arsenic	0.075	98.9, 94.1
Chromium	1	93.7, 92.1
Cobalt	1	104, 91.7
Copper	1	93.2, 88.6
Mercury	0.01	86.0
Zinc	1	100.0, 91.7
TOC	121.2	103, 97.3
Ammonia-N	300	97.4
Cyanide	10	108, 125

Client: Halby T&E Facility
Work Order: X0-08-022
00802207

IT ANALYTICAL SERVICES
CINCINNATI, OH

Quality Assurance Data

Matrix Spike/Duplicate Recovery Data

Client Sample ID: Soil # SS-S-AS-0-5-TC-B

Lab No. 08

Compound	Spike Value	Sample Value	Percent Recovery	Percent Recovery	RPD
Arsenic	0.075	ND	87.3	86.0	1.5
Chromium	2	0.008	93.4	93.1	0.2
Cobalt	2	ND	100	102	1.7
Copper	2	0.017	89.5	88.1	1.5
Mercury	0.5	ND	100	101	1.4
Zinc	2	0.329	105	109	2.9

% Recov = (Conc MS (or MSD)-Sample Amt)/ Amt Added)X 100

RPD = ((Conc.MS-Conc MSD)/(Conc MS + Conc MSD)/2))X 100

PEI Associates, Inc.

CHAIN-OF-CUSTODY RECORD

PROJECT NAME/NUMBER Halby Chemicals Site Treatability Studies PN#3741-60-2 LAB DESTINATION ITAS Cincinnati

SAMPLE TEAM MEMBERS Steve Giti-Pour CARRIER/WAYBILL NO.

SAMPLE NUMBER	SAMPLE LOCATION AND DESCRIPTION	DATE AND TIME COLLECTED	SAMPLE TYPE	CONTAINER TYPE	CONDITION ON RECEIPT (NAME AND DATE)
SS-S-AS-1-TC-BLANK	T&E Facility, SIS	6/21/90	Solid	Ziplock plastic bag	Samples were
SS-S-AS-0.5-TC-BLANK	Treated Samples	6/21/90	"	"	received in good
					Condition
					Dawn Webber
					8-3-90

SPECIAL INSTRUCTIONS:

POSSIBLE SAMPLE HAZARDS

SIGNATURES: (NAME, COMPANY, DATE, AND TIME)

1. RELINQUISHED BY: Steve Giti-Pour
   RECEIVED BY: Dawn Webber 8-3-90 ITAS
2. RELINQUISHED BY:
   RECEIVED BY:
3. RELINQUISHED BY:
   RECEIVED BY:
4. RELINQUISHED BY:
   RECEIVED BY:

WHITE - To accompany samples
YELLOW - Field copy

ORIGINAL
(Red)

PEI Associates, Inc.

CHAIN-OF-CUSTODY RECORD

PROJECT NAME/NUMBER Halby Chemicals Site Treatability Studies PN# 3741-60-2 LAB DESTINATION ITAS Cincinnati

SAMPLE TEAM MEMBERS Steve Giti-Pour CARRIER/WAYBILL NO.

SAMPLE NUMBER	SAMPLE LOCATION AND DESCRIPTION	DATE AND TIME COLLECTED	SAMPLE TYPE	CONTAINER TYPE	CONDITION ON RECEIPT (NAME AND DATE)
SS-US-PC-0.4-UCS-A	TBE Facility, S/S treated	6,25,90	Solid	Ziplock Plastic bag	Samples were
SS-US-PC-0.4-UCS-B	samples "	"	"	"	received in good
SS-US-PC-0.25-UCS-A	"	"	"	"	Condition
SS-US-PC-0.25-UCS-B	"	"	"	"	
					Dawn Webber
					8-3-90

SPECIAL INSTRUCTIONS:

POSSIBLE SAMPLE HAZARDS

SIGNATURES: (NAME, COMPANY, DATE, AND TIME)

1. RELINQUISHED BY: Steve Giti-Pour
RECEIVED BY: Dawn 6/26/90 Webber 8-3-90 ITAS
2. RELINQUISHED BY:
RECEIVED BY:
3. RELINQUISHED BY:
RECEIVED BY:
4. RELINQUISHED BY:
RECEIVED BY:

WHITE - To accompany samples

PEI Associates, Inc.

CHAIN-OF-CUSTODY RECORD

PROJECT NAME/NUMBER Halby Chemicals Site Treatability Studies PN#3741-60-2 LAB DESTINATION ITAS Cincinnati

SAMPLE TEAM MEMBERS Steve Giti-Pour CARRIER/WAYBILL NO.

SAMPLE NUMBER	SAMPLE LOCATION AND DESCRIPTION	DATE AND TIME COLLECTED	SAMPLE TYPE	CONTAINER TYPE	CONDITION ON RECEIPT (NAME AND DATE)
SS-S-PC-0.25-UCS-A	TBE Facility, S/S treated	6,23,90	Solid	Zip-lock Plastic bag	Samples were
SS-S-PC-0.25-UCS-B	Samples	6,23,90	"	"	received in good
SS-S-PC-0.4-UCS-A	"	6,22,90	"	"	condition
SS-S-PC-0.6-UCS-B	"	6,22,90	"	"	Dawn Webber
SS-S-AS-1-UCS-A	"	6,20,90	"	"	August 3, 1990
SS-S-AS-1-UCS-B	"	6,20,90	"	"	
SS-S-AS-0.5-UCS-A	"	6,20,90	"	"	
SS-S-AS-0.5-UCS-B	"	6,20,90	"	"	

SPECIAL INSTRUCTIONS:

POSSIBLE SAMPLE HAZARDS

SIGNATURES: (NAME,COMPANY,DATE,AND TIME)

1. RELINQUISHED BY: Steve Giti-Pour
RECEIVED BY: Dawn Webber ITAS 8-3-90

2. RELINQUISHED BY:
RECEIVED BY:

3. RELINQUISHED BY:
RECEIVED BY:

4. RELINQUISHED BY:
RECEIVED BY:

WHITE - To accompany samples
YELLOW - Field copy

PEI Associates, Inc.

CHAIN-OF-CUSTODY RECORD

PROJECT NAME/NUMBER Halby Chemicals Site Treatability Studies PN#3741-60-2 LAB DESTINATION ITAS Cincinnati

SAMPLE TEAM MEMBERS Steve Giti-Pour CARRIER/WAYBILL NO.

SAMPLE NUMBER	SAMPLE LOCATION AND DESCRIPTION	DATE AND TIME COLLECTED	SAMPLE TYPE	CONTAINER TYPE	CONDITION ON RECEIPT (NAME AND DATE)
SS-US-PC-0.4-TC-A	T&E Facility, S/S treated	6,25,90	Solid	Ziplock plastic bag	Samples were
SS-US-PC-0.4-TC-B	samples "	6,25,90	"	"	received in good
SS-US-PC-0.25-TC-A	"	6,25,90	"	"	Condition
SS-US-PC-0.25-TC-B	"	6,25,90	"	"	Dawn Webber
					August 3, 1990

SPECIAL INSTRUCTIONS:

POSSIBLE SAMPLE HAZARDS

SIGNATURES: (NAME, COMPANY, DATE, AND TIME)

1. RELINQUISHED BY: Steve Giti-Pour 6.26.90
   RECEIVED BY: Dawn Webber ITAS 8-3-90
2. RELINQUISHED BY:
   RECEIVED BY:
3. RELINQUISHED BY:
   RECEIVED BY:
4. RELINQUISHED BY:
   RECEIVED BY:

WHITE - To accompany samples

PEI Associates, Inc.

CHAIN-OF-CUSTODY RECORD

PROJECT NAME/NUMBER Halby Chemical Site Treatability Studies PN# 3741-60-2 LAB DESTINATION ITAS Cincinnati

SAMPLE TEAM MEMBERS Steve Giti-Pour CARRIER/WAYBILL NO.

SAMPLE NUMBER	SAMPLE LOCATION AND DESCRIPTION	DATE AND TIME COLLECTED	SAMPLE TYPE	CONTAINER TYPE	CONDITION ON RECEIPT (NAME AND DATE)
SS-SR-500-30-A	LTTD Treated Sample	6,19,90	Soil	8 oz glass Ja	Samples were received
SS-SR-500-30-B	"	6,19,90	Soil	"	in good condition
SS-SR-500-30-EX	"	6,23,90	Soil	"	
SS-US-A	Untreated soil from Steel	6,23,90	Soil	"	Dawn Webber
SS-US-B	bucket "	6,23,90	Soil	"	August 3, 1990

SPECIAL INSTRUCTIONS:

POSSIBLE SAMPLE HAZARDS

SIGNATURES: (NAME, COMPANY, DATE, AND TIME)

1. RELINQUISHED BY: Steve Giti-Pour

RECEIVED BY: Dawn Webber ITAS 8-3-90

2. RELINQUISHED BY:

RECEIVED BY:

3. RELINQUISHED BY:

RECEIVED BY:

4. RELINQUISHED BY:

RECEIVED BY:

WHITE - To accompany samples
YELLOW - Field copy

PEI Associates, Inc.

CHAIN-OF-CUSTODY RECORD

PROJECT NAME/NUMBER Halby Chemical Site Treatability Studies. PN# 3741-60-2 LAB DESTINATION ITAS Cincinnati

SAMPLE TEAM MEMBERS Steve Giti-Pour CARRIER/WAYBILL NO.

SAMPLE NUMBER	SAMPLE LOCATION AND DESCRIPTION	DATE AND TIME COLLECTED	SAMPLE TYPE	CONTAINER TYPE	CONDITION ON RECEIPT (NAME AND DATE)
SS-S-PC-0.25-TC-A	TBE Facility - S/S treated	6,23,90	Solid	ziplock plastic bag	Samples were
SS-S-PC-0.25-TC-B	samples "	6,23,90	"	"	received in good
SS-S-PC-0.4-TC-A	"	6,22,90	"	"	Condition
SS-S-PC-0.4-TC-B	"	6,22,90	"	"	Dawn Webber
SS-S-AS-1-TC-A	"	6,20,90	"	"	August 3, 1990
SS-S-AS-1-TC-B	"	6,20,90	"	"	
SS-S-AS-0.5-TC-A	"	6,20,90	"	"	
SS-S-AS-0.5-TC-B	"	6,20,90	"	"	
SS-S-PC-0.4-TC-BLANK	"	6,23,90	"	"	
SS-S-PC-0.25-TC-BLANK	"	6,23,90	"	"	

SPECIAL INSTRUCTIONS:

POSSIBLE SAMPLE HAZARDS

SIGNATURES: (NAME,COMPANY,DATE,A...)

1. RELINQUISHED BY: Steve Giti-Pour

RECEIVED BY: Dawn Webber 8-3-90

2. RELINQUISHED BY:

RECEIVED BY:

3. RELINQUISHED BY:

RECEIVED BY:

4. RELINQUISHED BY:

RECEIVED BY:

WHITE - To accompany samples

APPENDIX C-3.2

UCS Test Results For Asphalt- And Cement-Based Products

PEI Halby Site UCS

Sample ID	Load (lbs)	Stress (psi)
SS-S-AS-1-UCS-A	71	43
SS-S-AS-1-UCS-B	51	31
SS-S-AS-5-UCS-A	78	58
SS-S-AS-5-UCS-B	153	39
SS-S-PC-0.25-UCS-A	7200	1255
SS-S-PC-0.25-UCS-B	6760	1162
SS-S-PC-0.4-UCS-A	5910	1209
SS-S-PC-0.4-UCS-B	8660	1926
SS-US-PC-0.25-UCS-A	4532	788
SS-US-PC-0.25-UCS-B	4223	777
SS-US-PC-0.4-UCS-A	8700	1462
SS-US-PC-0.4-UCS-B	7860	1158

APPENDIX D

FIELD TRIP REPORT AND FIELD ANALYTICAL RESULTS OBTAINED FROM ONSITE X-RAY FLUORESCENCE

ORIGINAL
(Red)

CONTENTS

APPENDIX D

APPENDIX D-1

PEI FIELD TRIP REPORT MEMORANDUM

PEI ASSOCIATES, INC.

MEMORANDUM

TO:	Ed Barth	DATE:	4/12/90
SUBJECT:	Field Sample Collection for Halby Treatability Studies	FROM:	S. Hokanson
FILE:	3741-60-2	cc:	M. Taylor J. Hessling P. Mraz R. Riccio

Summarized below are the on-site field activities and observations made during our sampling visit to Halby Chemical on Tuesday and Wednesday (4/10-11/90). A formal field write-up will be developed after additional data becomes available from the EPA ERT team. This formal write-up can be used as appendixed information in the treatability study report submitted to EPA at the end of this project.

Summary of Field Activities

On Tuesday morning at 8:30 a.m., Harry Compton of EPA and personnel from EPA's ERT contractor met with the PEI field team and Roberta Riccio of EPA Region III. From 8:30 until 10:30 a.m. we discussed our main objectives and roles, Health and Safety issues, and toured the plant area and the on-site lagoon. From 10:30 until 12:30 p.m., the ERT field team collected sediments for screening with the XRF and HNu (11.7 eV lamp) from a background location and previous sampling location SED-02 and SED-03 (these location codes are from EBASCO's RI report). These samples were dried in a microwave oven, screened in a 20 mesh sieve, and homogenized for analysis. Exhibit A shows the XRF data generated for these and the other samples taken later that day. From 10:30 a.m. until 1 p.m., PEI purged the groundwater monitoring well SMW-01 of approximately 3 well volumes (about 65 gallons), oversaw the field sampling and screening efforts, and obtained the necessary sample containers.

In the afternoon, from 1:30 until 3:30 p.m., the ERT field team collected screening samples from previous sampling locations SED-05 and SED-08 (in lagoon area), and SSS-09 and SSS-25 (in the process plant area) and processed them through the HNu and XRF. During this time, the PEI team collected ground water samples for the biological and xanthate precipitation studies and sediment and surface soil samples for the biological, low temperature thermal desorption, and xanthate studies. At 3:30 p.m. on Tuesday, Mr. Schockley of Brandywine Chemical asked us to finish up our work so he could lock the gate to the process plant area.

Because of high tide conditions, the ERT Team could not collect the subsurface soil sample for the soil washing study. In addition, the PEI team had only half-filled the 30-gallon drum containing the sediment/surface soil samples and would have to finish the sample collection on Wednesday morning. The ground water and sediment samples for biological studies were collected in sterilized containers cooled with dry ice in plastic bags, packed and shipped to IT Knoxville on Tuesday afternoon.

On Wednesday morning, from 7:30 until 8:30 a.m., PEI met with EPA Region III and the ERT team to discuss the field activities and final output from ERT. From 8:30 a.m. until 11:30 a.m., the ERT team collected subsurface soil samples from SED-05 and SED-08 locations in the lagoon. PEI completed the collection of the sediment/surface soil samples by collecting surface soil from previous sampling location SSS-09. From 11:30 a.m. until 12:30 p.m., PEI demobilized and packed and stored the drummed sediment and ground water samples in the plant warehouse area. Field activities by PEI and ERT ended by 12:30.

Summary of Observations During Field Sampling

Discussed below are some points of observation made during the two-day sampling/analysis effort. These observations are in the following areas:

- field conditions;
- field sample collection methods; and
- field screening with the XRF spectrometer.

Each of these issues are discussed separately below.

Field Conditions--

On Tuesday, April 10, the weather conditions were partly sunny and windy with temperatures ranging from 50 to 60°F. Low tide took place in the early morning hours and high tide in the early afternoon time. Due to recent rainstorms, the marsh and lagoon areas were fairly saturated. In addition, fumes and dust were fairly strong on Tuesday, even outside the process plant area. While vapors outside the plant area were not detected by the HNu (11.7eV lamp), organic vapors were in the range of 5 to 10 ppm in the breathing space above monitoring well SMW-01 and sediment sampling locations. Level C personal protective equipment were worn by the PEI sampling team. Even with the personal protective equipment, the PEI field team leader experienced headache and nausea. This incident was reported, and a followup physical was performed shortly after the sampling event. The winds were much calmer on Wednesday, and fumes and dust were not as bad as they were the day before.

A sheen was observed along the lagoon and drainage ditch near the process plant area. This sheen was blotchy, unlike an oil sheen, and may be indicative of biological activity. Black sediment was clearly visible along the banks of the lagoon and were stratified in the sediment/surface soil samples collected. Purged ground water from well SMW-01 was black or cloudy gray in color with sulfurous odors.

Field Sample Collection Methods--

Sediment and surface soil samples for low-temperature thermal desorption, xanthate and the biological studies (SSS-L, SSS-X, SSS-B) were collected by the PEI field team at previous sampling locations SED-03 and SSS-09 in the process plant area. The subsurface soil sample for soil washing (SUBS-S) was collected at previous sample location SED-05 and SED-08. Groundwater for xanthate precipitation and biological studies was collected from SMW-01 after more than three well volumes were purged with a bailer.

Sediment and surface soil samples were collected with shovels by scraping off the fill and surface area until the first black layer was uncovered. From there, the PEI team dug down 1 foot beneath the first black soil layer. In the case of soils from SSS-09, the PEI team uncovered railroad spurs and a concrete pad and therefore the depth penetration was only about 6 inches from the first black soil layer. Because only half of the 30-gallon drum was filled when we were asked to leave, we put dry ice in garbage bags and placed it on top of the sediment/surface soils in the drum. Care was taken when placing the lid on the drum to allow carbon dioxide venting.

Because of saturated conditions and high tide in the lagoon area, the ERT field team collected the subsurface soils from SED-05 and SED-08 locations using hand augers and shovels. The power beaver could not be used under saturated conditions. Soils from 0 to 3 feet were collected from the two locations and were placed in two 5-gallon steel drums. Steel lids were taped to the containers to prevent any leakage.

Approximately 65 gallons (approximately 3 well volumes) of groundwater were purged from well SMW-01 using a teflon bailer. The rate of recharge was greater than the purging rate, so that the water elevation did not change dramatically during the purging. Water drawn during the purging activity went from fairly opaque to cloudy grey. Organic vapors downwind of the well were fairly strong. Therefore, the PEI field team wore respirators and stayed upwind as much as possible. After purging the well, groundwater for the biological samples were poured into biologically-sterile, dark 1-gallon glass jars. Care was taken to minimize exposure of the jar to light, air and biologically-contaminated materials (including fingers). After collecting the biological sample, the PEI field team collected groundwater for the xanthate study in a pre-cleaned, plastic, 5-gallon jug. Care was taken to completely fill the jug and eliminate bubbles. The jug was sealed and taped. This jug was then placed in a 30-gallon steel, open-head drum and packed with vermiculite and clay absorbent. Dry ice packed in plastic garbage bags were then placed on top and the lid placed on the drum but not sealed to allow carbon dioxide venting.

By the end of field sampling activities on Wednesday, PEI had one 30-gallon drum filled with surface soils/sediments, one 30-gallon drum containing the 5-gallon jug of groundwater and fill material; and two 5-gallon steel drums filled with subsurface soils from the lagoon area (SED-05 and SED-08 locations). These containers were sealed and placed inside the warehouse. They do not have dry ice or blue ice, so they will remain at the site under ambient temperature conditions (30 to 50°F).

Field Screening with the XRF Spectrometer--

The location of surface soil/sediment and subsurface soil samples (SSS-L, SSS-X, SSS-B, and SUBS-S) was determined by first reviewing the draft RI report and then field screening soil samples using the XRF spectrometer and HNu (11.7 eV lamp). Based on the surface soil and sediment analytical data presented in the draft RI report, soils near previous sample locations SED-02 and 03, and SSS-09 and 25 (process plant area) were expected to have high levels of CS_2, As and Zn. Soils near previous sample locations SED-05 and SED-08 (lagoon area) were expected to have low volatile organic compounds (VOC) and moderate to high levels of As, Cu and Zn. Field screening using the HNu with 11.7 eV lamp confirmed high levels of VOC's in sediment/surface soil samples collected from the process plant area (SED-02, SED-03, SSS-09 and SSS-25). Field screening for metals using the XRF, also confirmed moderate to high levels of metals in both lagoon and process plant soils from the previous sample locations listed above.

While the XRF spectrometer could identify and isolate peaks for Cu and Zn, it could not clearly separate the As and Pb peaks. Because Pb is fairly ubiquitous in soils at Halby, Pb masks any As that may be present in the soil samples. As a result of the ambiguous results for As, additional chemical analysis may be necessary to confirm the presence or absence of As in the individual samples used in the treatability studies.

W0758/Halby

ORIGINAL
(Red)

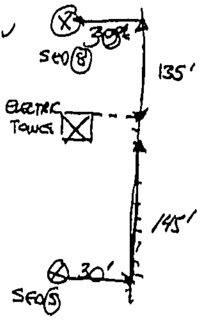
4/11/90

→ TOOK SUBSURFACE SAMPLES
→ TOOK SEDIMENT SAMPLES (SED-5, SED 8)
→ PACKAGED - 1 30-gallon DRUM SEDIMENT (SED-3, SSS-9)
- 1 5-gallon CONTAINER SUBSURFACE [illegible] SED-5
- 1 5 gallon CONTAINER " " SED-8
- 1 30-GALLON DRUM OVERPACKED FOR
5 gallon GROUNDWATER SAMPLE. [illegible]

SEDIMENT SAMPLE

SED-3 (15 gallons) 30 FEET LEFT OF TRACKS.
SSS-9 (15-gallons) 2 FEET RIGHT FROM TRACK
11 ½ FEET SOUTH OF OVERHEAD SUPPORT.

— SCREENING EFFORTS: N.O. HNU
READINGS 11.7eV



SUBSURFACE SAMPLE

SED 5 (5 gallons)
SED 8 (5 gallons)

* → ORDERED 1-55 gallon OPEN TOP PER
EPA REGION III REQUEST. USED THIS DRUM
TO DISPOSE PROTECTION GEAR (BOOTIES, GLOVES, etc.)
EPA
ROBERTA RICCIO. PAID FOR DRUM

* → DRUMS LABELED AND MARKED APPROPRIATELY
STORED IN WAREHOUSE.

ORIGINAL
(Red)

09/10/90

→ 8:15 ARRIVE ON-SITE
ROBERTA RICCIO ARRIVES

→ MEET WITH ERT
→ RTM PICKS UP (2) 5 gallon CONTAINERS FOR SED SAM
(2) 30-gallon CONTAINERS (17H) RX.
& PACKING.

→ 9:45 ERT ENTERS WORK ZONE
BEGINS SOIL SCREENING EFFORT

10:00 MITCH/RICK ARRIVE ON SITE ~~ERT~~ WITH EQUIPME

11:40 CRACKED LOCKED
- SCREENED WELL W/ HNU
- 35 - 55 ppm w/ 10.2 ev LAMP
- DISSIPATED.
- WELL DEPTH 31' 9"
- DRY DEPTH 4' 3"
27' 6" WATER ≈ 18 gallons

- SCREENED WELL W/ HNU
- 90 - 120 ppm w/ 11.7 ev LAMP.

- WITHDREW 60 gallons > (18×3)
FOR 3 WELL PURGES.

ORIGINAL
(Red)

EXHIBIT A

PRELIMINARY XRF SCREENING DATA FOR HEAVY METALS IN SURFACE SOIL/SEDIMENT SAMPLES

(Red)

WESTON

SHEET ____ of ____

CLIENT/SUBJECT ______________________________ W.O. NO. ______________

TASK DESCRIPTION ______________________________ TASK NO. ______________

PREPARED BY ____________ DEPT ____________ DATE ____________

MATH CHECK BY ____________ DEPT ____________ DATE ____________

METHOD REV. BY ____________ DEPT ____________ DATE ____________

APPROVED BY ____________

DEPT ________ DATE ________

Results are reported in relative % weight. These values were determined within a synthetic hazardous waste soil calibration model (model 1). Lead results reported in model 3 are relative Pb/As values, concentrations = ppm x10^{1}. Samples were dried in a microwave, sieved (20 mesh), and homogenized before analysis. Zn and Pb/As approximate maximum peak intensities were reported as an aid for relative concentrations (counts/s)

Elements

Sample	Zn	Cr	Cu	As	Pb	Zn cts/s	Pb/As cts/s
BKG-Clean I (mod.1)	0.135	0.594	0.870	0.000	0.000	365	311
(mod.3)					31.8		
BKG-Clean II (mod.1)	0.101	0.109	0.414	0.040	0.000	309	159
(mod.3)					13.19		
Sed-2-4' (mod.1)	0.186				0.000	700	333
(mod.3)					23.6		
Sed-3-Surf. (mod.1)	0.087	0.000	1.851	0.000	0.000	361	696
(mod.3)					127.2		
Sed-3-Subsurf. (mod.1)	0.121	0.013	1.454	0.000	0.000	529	495
(mod.3)					83.31		

WESTON

SHEET ___ of ___

CLIENT/SUBJECT ____________________ W.O. NO. ______

TASK DESCRIPTION ____________________ TASK NO. ______

PREPARED BY ______ DEPT ______ DATE ______

MATH CHECK BY ______ DEPT ______ DATE ______

METHOD REV. BY ______ DEPT ______ DATE ______

APPROVED BY ______

DEPT ______ DATE ______

Sample	Zn	Cr	Cu	As	Pb	Zn$^{ct/s}$	Pb/As
Sed-5-Surf (1)	0.193	0.009	0.368	0.000	0.000	967	40
(3)					69.3		
Sed-5-Subsurf. (1)	0.282	0.041	0.600	0.000	0.000	1224	40
(3)					77.31		
Sed-8-surf. (1)	0.431	0.000	1.248	0.000	0.000	1621	57
(3)					135.1		
Sed-8-Subsurf. (1)	0.151	0.014	0.712	0.000	0.000	735	33
(3)					30.46		
Sed-9-surf. (1)	0.219	0.009	2.099	0.000	0.000	660	36
(3)					25.74		
Sed-9-subsurf. (1)	0.103	0.032	1.346	0.000	0.000	451	36
(3)					31.3		
Sed-25-Surf. (1)	0.000	0.006	0.188	0.011	0.000	247	24
(3)					0.00		
Sed-25-Subsurf. (1)	0.069	0.016	0.898	0.000	0.000	332	36
(3)					43.93		

WESTON

SHEET ____ of ____

CLIENT/SUBJECT __________ W.O. NO. __________

TASK DESCRIPTION __________ TASK NO. __________

PREPARED BY ______ DEPT ______ DATE ______

MATH CHECK BY ______ DEPT ______ DATE ______

METHOD REV. BY ______ DEPT ______ DATE ______

APPROVED BY ______

DEPT ______ DATE ______

re: Pb/As

1. Sed-8-Surf.
2. Sed-3-surf.
3. Sed-5

} *

} Zn corresponds except in sample Sed-3.

re: Cu

Sed-3
Sed-8
Sed-9 *high

[illegible]

ORIGINAL
(Red)

APPENDIX D-2

FIELD ANALYTICAL RESULTS OBTAINED BY EPA/ERT USING XRF

ORIGINAL
(Red)

TABLE 1. XRF RESULTS FOR HALBY CHEMICAL WILMINGTON, DELAWARE

April, 1990

(Results reported in relative percent by weight)

	ELEMENTS				
SAMPLE	Zn	Cr	Cu	As	Pb/As
BKG Clean I					
(Model 1)	0.135	0.594	0.870	0.000	0.000
(Model 3)					318.0
BKG Clean II					
(Model 1)	0.101	0.109	0.414	0.040	0.000
(Model 3)					131.9
Sed-2-4'					
(Model 1)	0.186	-----	-----	-----	0.000
(Model 3)					236.0
Sed-3-Surf					
(Model 1)	0.087	0.000	1.851	0.000	0.000
(Model 3)					1272.
Sed-3-Subsurf					
(Model 1)	0.121	0.013	1.454	0.000	833.1
Sed-5-Surf					
(Model 1)	0.193	0.009	0.368	0.000	0.000
(Model 3)					693.0
Sed-5-Subsurf					
(Model 1)	0.282	0.041	0.600	0.000	0.000
(Model 3)					773.1
Sed-8-Surf					
(Model 1)	0.431	0.000	1.248	0.000	0.000
(Model 3)					1251.0
Sed-8-Subsurf					
(Model 1)	0.151	0.014	0.712	0.000	0.000
(Model 3)					304.6
SSS-09-Surf					
(Model 1)	0.219	0.009	2.099	0.000	0.000
(Model 3)					257.4
SSS-09-Subsurf					
(Model 1)	0.103	0.032	1.346	0.000	0.000
(Model 3)					373.
SSS-25-Surf					
(Model 1)	0.000	0.006	0.188	0.011	0.000
(Model 3)					0.000
SSS-25-Subsurf					
(Model 1)	0.069	0.016	0.898	0.000	0.000
(Model 3)					439.4

rd:eh/DEBRUIJN/TR-2307.R1

FIGURE 2
HALBY CHEMICAL SAMPLE LOCATIONS
WILMINGTON, DE