Review of Analytical Results from the Proposed Agent Disposal Facility Site, Aberdeen Proving Ground

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Review of Analytical Results from the Proposed Agent Disposal Facility Site, Aberdeen Proving Ground

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Abstract

Argonne National Laboratory reviewed the analytical results from 57 composite soil samples collected in the Bush River area of Aberdeen Proving Ground, Maryland. A suite of 16 analytical tests involving 11 different SW-846 methods was used to detect a wide range of organic and inorganic contaminants. One method (BTEX) was considered redundant, and two "single-number" methods (TPH and TOX) were found to lack the required specificity to yield unambiguous results, especially in a preliminary investigation. Volatile analytes detected at the site include 1,1,2,2-tetrachloroethane, trichloroethylene, and tetrachloroethylene, all of which probably represent residual site contamination from past activities. Other volatile analytes detected include toluene, tridecane, methylene chloride, and trichlorofluoromethane. These compounds are probably not associated with site contamination but likely represent cross-contamination or, in the case of tridecane, a naturally occurring material. Semivolatile analytes detected include three different phthalates and low part-per-billion amounts of the pesticide DDT and its degradation product DDE. The pesticide could represent residual site contamination from past activities, and the phthalates are likely due, in part, to cross-contamination during sample handling. A number of high-molecular-weight hydrocarbons and hydrocarbon derivatives were detected and were probably naturally occurring compounds.

1 Introduction

The primary objective of this review was to compile detailed results from laboratory analyses of composite soil samples taken from the proposed site of the chemical agent disposal facility in the Bush River area of Aberdeen Proving Ground (APG), Maryland. The APG Directorate of Safety, Health, and Environment, Hazardous Waste Branch, will use the results to determine the nature and extent of any necessary remediation at the site of the proposed facility. Additional objectives were to examine the suitability of the analytical methods employed and review the overall quality assurance/quality control results reported by the analytical laboratory.

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Argonne National Laboratory (ANL) collected 57 composite soil samples between January and July 1997, as shown in Table 1. Also shown in Table 1 are the dates on which the samples were received by the analytical laboratory, Ana-Lab Corporation. A map of the site of the proposed facility showing the locations of all 57 samples is provided in Figure 1.

TABLE 1 Sampling Dates for Composite Soil Samples

Phase	Sample Numbers	Dates Collected	Date Received ^a
IV	1-12	01/07-01/08/97	01/10/97
V	13-23	03/03-03/04/97	03/07/97
VI	24-36	03/31-04/02/97	04/04/97
VII	37-42	05/12/97	05/16/97
VIII	43-45, 53, 54, 57	06/09-06/11/97	06/13/97
IX	46-52, 55, 56	07/14-07/16/97	07/18/97

^a Date received by Ana-Lab Corp.

FIGURE 1 Site Map of the Proposed Chemical Agent Disposal Facility, Aberdeen Proving Ground, Maryland

2 Suitability of Analytical Methods

Table 2 lists the analytical methods used for the composite samples of interest. All methods are taken from the U.S. Environmental Protection Agency (EPA) compendium, SW-846 (U.S. Environmental Protection Agency 1986), except the method used to determine solids (American Public Health Association 1992). All the methods listed in the table are appropriate for use with soil samples, except Method 9020 (Total Organic Halides, or TOX). According to the description of the method (U.S. Environmental Protection Agency 1986), Method 9020 is applicable for analyzing drinking water and for groundwater samples, but it is not appropriate for measuring TOX adsorbed onto undissolved solids. In addition, this method is not specific to halogenated compounds, because any organics (such as aromatic hydrocarbons) that will be detected by a photoionization detector will also be detected by this method.

The BETX method (Method 8021), while applicable to soil samples, is redundant for the samples of interest here because Method 8260 can be used to determine BETX hydrocarbons down to considerably lower concentrations.

The two methods used for general hydrocarbon detection — Method 418.1 (Total Recoverable Petroleum Hydrocarbons, or TPH) and Method 8015 (modified) (Nonhalogenated Volatile Organics by Gas Chromatography, also called TPH by some authorities) — do not provide compound-specific concentration values and are not necessarily specific to the hydrocarbons of concern (e.g., petroleum hydrocarbons). Method 418.1 detects compounds through the infrared absorption due to the carbon-hydrogen bonds found in nearly all classes of organic matter; virtually any organic substance that will dissolve in the fluorocarbon extraction solvent used will be detected if present in large enough quantities. Method 8015 (modified) is somewhat more specific in that only those hydrocarbons that can be either volatilized or extracted and analyzed by using gas chromatography will be found (typically, organics having carbon numbers in the range C_6 to C_{26}). In the modified method, both volatile and extractable hydrocarbons are determined and reported separately. Both methods are designed for use with petroleum hydrocarbons. Method 8015 (modified) is more suitable for exploratory work in which the presence or absence of petroleum hydrocarbons is to be determined, while Method 418.1 is more suitable for use in situations where the presence of contamination has already been established at sufficiently high levels. Because neither method provides compound-specific concentrations, the detection of hydrocarbons with either method does not necessarily imply the existence of a threat to human health or the environment.

TABLE 2 Summary of Analytical Methods Used

Analyte Class	Method Number	Method Name
TCLP Metals	SW-846 6010	Inductively Coupled Plasma-Atomic Emission Spectroscopy
TCLP Mercury	SW-846 7470	Mercury in Liquid Waste (Manual Cold-Vapor Technique)
TCLP Pesticides	SW-846 8080	Organochlorine Pesticides and Polychlorinated Biphenyls by Gas Chromatography
TCLP Herbicides	SW-846 8150	Chlorinated Herbicides by Gas Chromatography
TCLP VOA	SW-846 8260	Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS): Capillary Column Technique
TCLP SVOA	SW-846 8270	Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS): Capillary Column Technique
BTEX	SW-846 8021	Halogenated Volatiles by Gas Chromatography Using Photoionization and Electrolytic Conductivity Detectors in Series: Capillary Column Technique
PCBs	SW-846 8081	Organochlorine Pesticides and PCBs as Arochlors by Gas Chromatography: Capillary Column Technique
Pesticides	SW-846 8081	see above
VOA	SW-846 8260	see above
SVOA	SW-846 8270	see above
TPH	SW-846 418.1	Total Recoverable Petroleum Hydrocarbons
Hydrocarbons	SW-846 8015 (Modified)	Nonhalogenated Volatile Organics by Gas Chromatography
Metals	SW-846 6010	see above
TOX	SW-846 9020A	Total Organic Halides (TOX)
Solids	APHA 2540G	Total, Fixed, and Volatile Solids in Solid and Semisolid Samples

3 Quality Assurance/Quality Control

The quality control (QC) results provided in support of the analytical data were examined for completeness and compliance with SW-846 requirements. Twelve samples, two from each batch of samples sent to the laboratory, were selected at random for detailed examination; this number corresponds to 8.3% of the total number of samples. The QC results for the remaining samples were also examined, but in less detail.

For each sample examined in detail, it was possible to find QC results that were out of compliance with requirements. However, the percentage of such results was judged to be less than 5% of the total number of QC measurements, and with one exception, no pervasive or systematic problems were found with any of the QC data. The exception occurs for PCBs: no PCB-specific QC data (matrix spikes, blanks, check standards) were provided for samples 43-57, although adequate QC data were provided for chlorinated pesticides. PCBs and chlorinated pesticides were determined by using the same method (SW-846 8081).

On the basis of this examination of the QC results, the quality of the analytical data is considered adequate for their intended use.

4 Discussion of Analytical Results

4.1 Organic Analytes

The results from the analyses for various organic analytes are shown in Table 3. Several points can be made about these data; these points will be discussed in turn.

4.1.1 Phthalates

Low (sub-ppm) levels of phthalates, primarily di-n-butyl phthalate, appear to be present in most samples collected from the proposed locations of the CDB, PAB, FFB, and UVO buildings, all of which are on the southeast side of the site. Many of these levels are only slightly above the Estimated Quantitation Limit (EQL), which varied from 330 to 410 $\mu\text{g/kg}$ over the course of the sampling program. However, phthalates are also common laboratory contamination artifacts because they are used as plasticizers in rubber bulbs and other similar equipment used in analytical laboratories. An unambiguous determination of the source of the phthalates cannot be made because no field QC samples, specifically field and trip blanks, were included in the sampling plan. Laboratory contamination would have been indicated if contaminants were reported for such samples.

Internal evidence in Table 3 suggests that the phthalates are, indeed, laboratory-contamination artifacts. Samples 1–12 and 13–23 were collected and analyzed in January and early March, respectively (sampling phases IV and V), while samples 24–26 and 57, which are also located in the suspect area, were collected and analyzed at later dates, in sampling phases VI and VIII. The fact that no phthalates were found in these later samples suggests that the analytical laboratory found and corrected a source of phthalate contamination in its own operations between the dates of sampling phases V and VI, and that the apparent presence of phthalates in these samples is due to this contamination.

4.1.2 Samples 15–23

The results shown in Table 3 for samples 15–23 indicate a somewhat higher level of organic contamination than elsewhere; therefore, the area covered by these samples could have been used for some activity in the past. This possibility is supported by the fact that the area is open and covered by crushed rock and gravel rather than by vegetation. The highest phthalate concentrations are found here (in samples 18, 19, and 21), and this finding may indicate that phthalates are present in this area in addition to the possible laboratory contamination discussed above. The only samples in which chlorinated hydrocarbon solvents were found are in this area (samples 15, 20, 22, and 23). Two of the three chlorinated hydrocarbons (trichloroethylene and

TABLE 3 Data Summary — Organics^a

Sample	Method							Substances Detected
	8260 VOA (µg/kg)	8270 SVOA (µg/kg)	9020A TOX (mg/kg)	8021 BTEX (µg/kg)	8081 PCB (µg/kg)	8081 Pesticides (µg/kg)	418.1 TPH (mg/kg)	
1	ND	920	12	ND	ND	----	ND	Di-n-butylphthalate
2	ND	710	12	ND	ND	----	ND	Di-n-butylphthalate
3	ND	910	12	ND	ND	----	27	Di-n-butylphthalate
4	ND	510	8.2	ND	ND	----	12	Di-n-butylphthalate
5	ND	660	ND	ND	ND	----	12	Di-n-butylphthalate
6	ND	600	23	ND	ND	----	ND	Di-n-butylphthalate
7	ND	550	11	ND	ND	----	ND	Di-n-butylphthalate
8	ND	360	10	ND	ND	----	ND	Di-n-butylphthalate
9	ND	450	14	ND	ND	----	15	Di-n-butylphthalate
10	ND	400	17	ND	ND	----	ND	Di-n-butylphthalate
11	ND	370	ND	ND	ND	----	ND	Di-n-butylphthalate
12	ND	ND	8.2	ND	ND	----	ND	
13	ND	1600	25	ND	ND	----	ND	Di-n-butylphthalate
14	ND	1600	20	ND	ND	----	25	Di-n-butylphthalate
15	1100		6.3	ND	ND	----	ND	1,1,2,2-tetrachloroethane
	29							Trichloroethylene
		1600						Di-n-butylphthalate
16	ND	1200	14	ND	ND	----	15	Di-n-butylphthalate
17	ND	1200	5	ND	ND	----	11	Di-n-butylphthalate
18	ND	2000	9.5	ND	ND	----	17	Di-n-butylphthalate
		630						Diethyl phthalate
19	ND	660	7.7	ND	ND	----	ND	Benzyl butyl phthalate
		1100						Di-n-butylphthalate
		3200						Diethyl phthalate
20	11		10	ND	ND	----	11	1,1,2,2-tetrachloroethane
	110							Trichloroethylene
		1900						Di-n-butylphthalate
21	ND	2000	6.6	ND	ND	----	17	Di-n-butylphthalate
22	8.5		8.5	ND	ND	----	17	Tetrachloroethylene
		1600						Di-n-butylphthalate
23	23		6.8	ND	ND	----	15	Tetrachloroethylene
		1600						Di-n-butylphthalate
		1700						Diethyl phthalate
24	ND	ND	ND	ND	ND	----	ND	
25	ND	ND	ND	ND	ND	----	ND	
26	ND	ND	ND	ND	ND	----	ND	
27	ND	ND	ND	ND	ND	----	ND	
28	ND	ND	ND	ND	ND	----	ND	
29	ND	ND	ND	ND	ND	----	ND	
30	ND	ND	ND	ND	ND	----	ND	
31	ND	ND	6.4	ND	ND	----	ND	
32	ND	360	ND	ND	ND	----	ND	Di-n-butylphthalate
33	ND	ND	ND	ND	ND	----	ND	
34	ND	580	ND	ND	ND	----	ND	Di-n-butylphthalate

TABLE 3 (Cont.)

Sample	Method							Substances Detected
	8260 VOA (µg/kg)	8270 SVOA (µg/kg)	9020A TOX (mg/kg)	8021 BTEX (µg/kg)	8081 PCB (µg/kg)	8081 Pesticides (µg/kg)	418.1 TPH (mg/kg)	
35	5.5		ND	ND	ND	----	ND	Trichlorofluoromethane Di-n-butylphthalate
		540						
36	ND	ND	ND	ND	ND	----	ND	
37	ND	ND	13.3	ND	ND	----	(dry wt) 59	
38	6.7	ND	11.1	ND	ND	----	(dry wt) 79	Trichlorofluoromethane
39	ND	ND	10.0	ND	ND	----	ND	
40	6.8	ND	18.5	ND	ND	----	ND	Trichlorofluoromethane
41	ND	ND	17.5	ND	ND	----	ND	
42	7.2	ND	11.2	ND	ND	----	ND	Trichlorofluoromethane
43	ND	ND	7	ND	ND	ND	17	
44	ND	ND	ND	ND	ND	ND	20	
45	ND	ND	ND	ND	ND	ND	11	
46	ND	ND	8.2	ND	ND	5.29 7.03	22 ^b , ND ^c	4,4'-DDE 4,4'-DDT
47	ND	ND	12	ND	ND	11.1 12.8	31 ^b , ND ^c	4,4'-DDE 4,4'-DDT
48	5.8	ND	----	ND	ND	15.8 21.5	33 ^b , ND ^c	Toluene 4,4'-DDE 4,4'-DDT
49	ND	ND	----	ND	ND	10.8 12	26 ^b , ND ^c	4,4'-DDE 4,4'-DDT
50	ND	ND	----	ND	ND	3.05 3.67	18 ^b , ND ^c	4,4'-DDE 4,4'-DDT
51	6.5	ND	----	ND	ND	10.5 9.13	24 ^b , ND ^c	Toluene 4,4'-DDE 4,4'-DDT
52	ND	ND	----	ND	ND	8.48 5.74	43 ^b , ND ^c	4,4'-DDE 4,4'-DDT
53	ND	ND	ND	ND	ND	ND	18	
54	10	ND	ND	ND	ND	ND	14	Methylene chloride
55	5.6 9.1	ND	----	ND	ND		38 ^b , ND ^c	Toluene Trichlorofluoromethane 4,4'-DDE 4,4'-DDT
						25.4 13.6		
56	----	----	----	ND	----	----	48 ^b , ND ^c	
57	ND	ND	ND	ND	ND	ND	ND	

^a The highest reported concentration for each identified compound is shown in boldface numerals.

^b Value obtained by using Method 418.1.

^c Value obtained by using Method 8015.

tetrachloroethylene) found in these samples are also on the TCLP/VOA analyte list; however, these substances were not detected in the TCLP leachate from these samples. Results from the independent worker-safety-related analyses confirm the detection of all the chlorinated hydrocarbons found in these samples. In each case, the same analytes were detected in one or more of the subsamples making up the composite sample. A positive detection for Total Organic Halides (TOX) was reported for all these samples, and a positive detection for Total "Petroleum" Hydrocarbons (TPH) was reported for all but two of these samples.

4.1.3 Total Petroleum Hydrocarbons

The TPH data (Method 418.1) seem to indicate at least two sources of hydrocarbons. As just described, positive TPH results were reported for all but two of samples 15–23. The possible presence of petroleum hydrocarbons in these samples is consistent with other data that suggest that this area has been used in the past.

The only other areas for which TPH results are consistently positive are (1) the retention pond (samples 43–45) and (2) that area covered by the proposed locations of the BIOTREAT, BCB, BCS, and WSB buildings on the northwest side of the site (samples 46–56). These areas are currently covered by woods and shrubbery, and the positive TPH results are probably due to naturally occurring hydrocarbons produced by plant metabolism or the decay of plant matter rather than to the existence of hydrocarbon contamination from some earlier human activity. The TPH results for samples 3, 4, and 5 may also be positive because they were collected from wooded locations. The existence of a wide variety of naturally occurring organic substances in soils, including many that also occur in petroleum hydrocarbons, is well-documented (Dragun and Barkach 1989). Several compounds chemically similar to these were found in the SVOA mass-spectral library search discussed below. Although the correlation in the data of Table 2 between a high level of vegetative cover and a positive TPH result is not perfect, it is good enough to strongly suggest that a positive TPH result does not necessarily indicate the presence of contamination. The nonspecific nature of this analysis can lead to misleading results without other corroborating evidence of petroleum hydrocarbon contamination (Gustafson undated).

Two TPH results using Method 418.1 were reported on a dry-weight basis instead of the wet-weight basis used for the rest of the samples. These two samples, 37 and 38, also showed the highest TPH values; converted to a wet-weight basis, they are 49 and 64 mg/kg, respectively. Both samples came from a wooded area.

An analysis for hydrocarbons using Method 8015 (modified) was run for samples 46–52, 55, and 56 (sampling phase IX), in addition to TPH by Method 418.1. No positive hydrocarbon detections were reported. As described in Section 2, Method 8015 (modified) is more specific to petroleum hydrocarbons than is Method 418.1, although it is not clear why Method 8015 (modified) did not yield a single positive result since it should have detected many of the same naturally occurring hydrocarbons that Method 418.1 detects. A possible explanation is that many of the naturally occurring substances detected by Method 418.1 may not be amenable to analysis

by the standard gas-chromatographic methods, columns, and temperatures used in Method 8015 (modified).

4.1.4 Pesticides

The pesticides 4,4'-DDE and 4,4'-DDT were found at low part-per-billion levels in samples 46–52 and 55. These samples cover the proposed locations of the BIOTREAT, BCS, and WSB buildings. The spatial distribution of the pesticide residue is rather spotty, because pesticides were not detected in (1) samples 53 and 54 (the BCB building location), (2) samples 43–45 (the retention pond), or (3) sample 57 (the SWS building location). Earlier samples were not analyzed for pesticides.

4.1.5 Total Organic Halides

The TOX data are difficult to interpret because the analytical method is not necessarily specific for halogenated substances and does not yield compound-specific concentration values. A positive TOX result was reported in all but two samples out of the first 23 taken. In addition, samples 37–42 (the UB, FOS, and ECF building locations) also tested positive for TOX, as did four other samples at scattered locations (numbers 31, 43, 46, and 47).

4.1.6 Volatile Organics

Very low levels of toluene, methylene chloride, and trichlorofluoromethane were detected in several samples. The toluene results (samples 48, 51, and 55) may indicate the presence of toluene in the samples, but the methylene chloride result (10 ppb in sample 54) and the trichlorofluoromethane results (below 10 ppb in samples 35, 38, 40, 42, and 55) are almost certainly laboratory contamination artifacts.

4.1.7 Mass-Spectral Library Search Results

The identity of a compound eluting in a GC/MS analysis is determined by comparing its mass spectrum with those contained in a library of mass spectra of known compounds. The quality of a match is indicated in the analytical report by the "Qual" parameter, which ranges from 0 to 100, with 100 representing a perfect match. High values of Qual (generally greater than 90) indicate a high probability of a correct compound identification.

TABLE 4 Data Summary — Results of Method 8260 Volatile Organic Library Search^a

Sample	Compound ^b	RT ^c	Conc ^d	Cpd ^b	RT ^c	Conc ^d	Compound ^b	RT ^c	Conc ^d	Cpd ^b	RT ^c	Conc ^d	Cpd ^b	RT ^c	Conc ^d	Cpd ^b	RT ^c	Conc ^d
1-5	ND																	
6	Unk	4.44	7	Unk	4.52	7	Tridecane	15.16	11				Unk	15.57	16	Unk	16.91	7
7-9	ND																	
10	Unk	8.12	6															
11-27	ND																	
28				Unk	15.57	11												
29-36	ND																	
37	Cyclo-4	12.70	181	Unk	15.57	287							Unk	18.45	76	Unk	18.46	18
38	Unk	12.71	109	Unk	15.58	211							Unk	18.46	44	Unk	18.47	10
39	Cyclo-4	12.71	108	Unk	15.58	129	Unk	15.59	26				Unk	18.46	41			
40	Cyclo-4	12.71	168	Unk	15.58	252							Unk	18.46	64	Unk	18.47	15
41	Cyclo-4	12.72	105	Unk	15.58	133	Unk	15.59	29				Unk	18.47	43	Unk	18.47	10
42	Cyclo-4	12.72	251	Unk	15.58	367	Unk	15.59	82				Unk	18.46	123	Unk	18.47	30
43	Unk	12.71	76	Unk	15.57	84	Unk	15.58	18				Unk	18.45	23			
44	Unk	12.71	250	Unk	15.57	376	Unk	15.58	85				Unk	18.46	73	Unk	18.46	18
45	Cyclo-4	12.71	69	Unk	15.58	147							Unk	18.46	29			
46	Cyclo-4	12.48	122	Unk	15.33	88							Unk	18.19	19			
47				Unk	15.32	146	Unk	15.33	33	Unk	15.57	14	Unk	18.19	35			
48	Cyclo-4	12.48	357	Unk	15.33	176							Unk	18.19	34			
49	Cyclo-4	12.48	334	Unk	15.33	181							Unk	18.20	46			
50	Unk	12.48	254	Unk	15.33	141	Unk	15.33	31	Unk	15.58	13	Unk	18.19	35	Unk	18.20	9
51	Cyclo-4	12.48	367	Unk	15.33	211							Unk	18.19	43	Unk	18.20	11
52	Cyclo-4	12.48	334	Unk	15.33	180	Cyclo-3	9.04	9	Unk	15.58	18	Unk	18.19	36	Unk	18.20	8
53	Cyclo-4	12.71	227	Unk	15.57	272				Unk	15.58	62	Unk	18.46	49	Unk	18.46	11
54	Unk	12.71	219	Unk	15.57	261	Unk	15.58	59				Unk	18.46	58	Unk	18.46	14
55	Cyclo-4	12.48	203	Unk	15.32	138	Unk	15.33	31				Unk	18.19	76	Unk	18.20	19
56	No Data																	
57	Cyclo-4	12.71	39															

^a The highest reported concentration for each identified compound is shown in boldface numerals.

^b Compound: ND indicates that no library compounds were detected by the search. Unk indicates an unknown compound (the quality of the mass-spectral match was less than 80%). Cyclo-3 denotes hexamethyl-cyclotrisiloxane. Cyclo-4 denotes octamethyl-cyclotetrasiloxane.

^c RT denotes the retention time in minutes.

^d Conc denotes the approximate concentration in µg/kg (parts per billion).

The results of the volatile organic library search are summarized in Table 4. The results show that only three compounds were identified with Qual values greater than 80. The first, tridecane, occurs in sample 6 at an estimated concentration of 11 ppb. The second compound, hexamethyl-cyclotrisiloxane, occurs in sample 52 at an estimated concentration of 9 ppb. The third compound, octamethyl-cyclotetrasiloxane, occurs in samples 37–55 and 57 at a maximum concentration of 367 ppb. Although some spectral matches for this compound are marked unknown because of a Qual value below 80, the agreement in the GC retention time (the parameter RT in Table 4) clearly suggests that the substance is indeed octamethyl-cyclotetrasiloxane. These two siloxane compounds are routinely found in GC/MS analyses of soil extracts and often is due to chemical breakdown of the GC column's stationary phase. Results from the independent worker-safety related analyses confirm the presence of these cyclosiloxanes and indicate the presence of higher molecular weight homologs. The tridecane in sample 6 is the only volatile hydrocarbon found in any of the samples and is probably produced naturally.

Table 5 summarizes the results of the semivolatile organic library search. The five identified peaks with the highest concentrations are found in sample 41 (cyclotetracosane, 3.4 ppm), sample 55 (1-docosanol acetate, 2.9 ppm; 9-octyl-heptadecane, 2.9 ppm), sample 48 (1-docosanol acetate, 2.7 ppm), and sample 39 (cyclotetracosane, 2.5 ppm). These are all high-molecular-weight (C₂₄–C₂₅) hydrocarbons or hydrocarbon derivatives that occur naturally in soil.

Much of the remaining data in Table 5 can be classified on the basis of GC retention times. For example, samples 46–50 all contain a compound eluting at 27.74 min. In each case, the compound is identified by the library search as 1-dotricontanol, a C₃₂ alcohol found in plants. Thus, it appears that the GC peaks identified as a hydrocarbon or as hydrocarbon-related compounds in Table 5 can be traced to naturally occurring substances found in areas covered by woods and shrubbery.

4.2 TCLP Analytes

Analyses were done on the samples of interest for TCLP metals, including mercury, TCLP pesticides, TCLP herbicides, TCLP volatile organics, and TCLP semivolatile organics. The only TCLP analyses that resulted in the detection of the analyte were the metals analyses. The result of all other TCLP analyses was nondetection.

Table 6 summarizes the results of the TCLP metals analysis. Blank entries represent nondetections. At the top of the table, the EPA's regulatory limits for the indicated metals are shown. All analytical results were well below the corresponding regulatory limits.

TABLE 5 Data Summary — Results of Method 8270 Semivolatile Organic Library Search^a

[illegible]

TABLE 5 (Cont.)

RT ^b	Conc ^c	ID ^d	RT ^b	Conc ^c	ID ^d	RT ^b	Conc ^c	ID ^d	RT ^b	Conc ^c	ID ^d	RT ^b	Conc ^c	ID ^d
<i>Sample 47</i>			<i>Sample 48</i>			<i>Sample 49</i>			<i>Sample 50</i>			<i>Sample 51</i>		
7.98	0.4	unk	7.98	0.4	unk	7.97	0.3	unk	27.55	0.6	unk	7.84	0.9	unk
27.02	0.3	unk	27.56	0.6	unk	27.02	0.4	unk	27.74	0.6	hyd	27.43	0.3	hyd
27.74	0.8	hyd	27.74	0.8	hyd	27.56	2.0	unk	27.92	0.4	unk	27.63	0.5	hyd
28.87	2.3	hyd	28.87	2.7	hyd	27.74	0.7	hyd	27.93	0.6	unk	28.76	1.4	hyd
29.37	0.8	hyd	29.37	0.4	unk	27.91	1.8	unk	27.94	0.3	unk	29.49	0.4	hyd
29.60	0.8	hyd	29.60	1.0	hyd	27.92	0.9	unk	28.87	1.6	hyd	29.76	0.9	hyd
29.87	1.2	hyd	29.88	2.4	hyd	28.01	0.5	unk	29.37	0.6	hyd	29.8	0.4	unk
29.93	0.7	hyd	29.93	0.7	hyd	28.87	1.9	hyd	29.61	0.4	hyd	29.91	0.4	hyd
30.03	0.5	hyd	30.03	1.0	hyd	29.38	0.8	hyd	29.88	0.6	hyd	30.55	0.6	hyd
30.69	1.1	hyd	30.69	1.5	hyd	29.60	0.6	hyd	30.04	1.2	hyd	30.83	0.7	hyd
30.97	0.8	hyd	30.97	1.5	hyd	29.88	1.1	hyd	30.69	0.6	hyd	30.94	0.3	unk
31.08	0.6	unk	31.05	0.3	hyd	29.92	0.5	hyd	31.08	1.3	unk	31.78	0.5	hyd
31.95	1.2	hyd	31.08	1.2	unk	30.04	0.4	unk	31.94	0.6	hyd	32.96	0.5	hyd
32.38	0.4	hyd	31.95	1.6	hyd	30.69	0.9	hyd	32.30	0.5	unk			
33.16	0.5	hyd	32.38	0.6	hyd	30.97	0.8	hyd	33.70	0.5	unk			
33.50	0.4	hyd	32.49	0.4	unk	31.07	0.4	unk						
33.71	2.3	hyd	33.16	0.6	hyd	31.95	0.9	hyd						
			33.50	0.5	hyd									
<i>Sample 52</i>			<i>Sample 53</i>			<i>Sample 54</i>			<i>Sample 55</i>			<i>Sample 57</i>		
27.54	2.4	unk	25.90	1.3	unk	25.90	1.1	unk	7.83	0.8	unk	27.34	2.1	unk
27.71	0.6	unk	27.28	0.8	hyd	27.27	0.6	unk	27.43	0.6	hyd			
28.85	1.4	hyd	27.30	0.5	hyd	27.33	0.7	unk	27.62	1.2	hyd			
29.58	0.5	hyd	28.02	0.4	hyd	27.34	1.2	unk	28.75	2.9	hyd			
29.86	0.7	hyd	28.30	1.5	hyd	28.30	0.6	hyd	29.48	0.5	hyd			
29.90	0.4	unk	29.02	0.6	hyd	28.46	0.5	hyd	29.75	2.9	hyd			
30.01	0.4	hyd	29.25	0.6	hyd	29.25	0.4	hyd	29.81	0.7	hyd			
30.67	0.6	hyd	29.97		unk	29.36	0.6	unk	29.90	0.6	hyd			
30.95	0.5	hyd				29.97	0.4	hyd	30.55	0.7	hyd			
31.92	0.5	unk							30.82	2.2	hyd			
33.67	0.5	unk							30.93	0.5	unk			
33.68	0.3	hyd							31.77	1.0	hyd			
33.93	0.4	unk							32.07	0.5	hyd			
									32.19	0.4	hyd			
									32.30	0.6	unk			
									32.95	0.6	hyd			

^a Samples not explicitly listed contained no compounds identified by the library search. The highest reported concentration for each identified compound is shown in boldface numerals.

^b RT denotes the retention time in minutes.

^c Conc denotes the approximate concentration in mg/kg (parts per million).

^d ID: unk denotes an unknown substance (mass-spectral quality less than 80%); hyd denotes a hydrocarbon or hydrocarbon derivative.

TABLE 6 Data Summary — TCLP Metals^{a,b}

Sample	Regulatory Limit (mg/L), by Metal							
	Arsenic	Barium	Cadmium	Chromium	Mercury	Lead	Selenium	Silver
	5.0	100.0	1.0	5.0	0.2	5.0	1.0	5.0
1		0.96						
2		0.92						
3		0.79						
4		1.30						
5		1.50						
6		0.52						
7		0.58						
8		1.10						
9		0.72						0.05
10		0.94						
11		0.84						
12		0.90						
13		0.69			0.0008			
14		0.94						
15		0.57						
16		0.98			0.0005			
17		0.74			0.0006			
18		0.74			0.0006			
19		0.60						
20		0.67						
21		0.69						
22		0.63			0.0005			
23		0.57						
24		0.73						
25		0.54						
26		0.50						
27		1.00						
28		0.79						
29		0.60						
30		0.73						
31		0.79						
32		0.51						
33		0.51						
34		0.70						
35		0.90						
36		1.20						
37		0.40					0.28	
38		0.27					0.31	
39		0.34						
40		0.26	0.05	0.19			0.27	
41		0.07						
42		0.45						
43		0.56						
44		0.63					0.28	
45		0.79						
46		1.40						
47		1.20						

TABLE 6 (Cont.)

Sample	Regulatory Limit (mg/L), by Metal							
	Arsenic	Barium	Cadmium	Chromium	Mercury	Lead	Selenium	Silver
	5.0	100.0	1.0	5.0	0.2	5.0	1.0	5.0
48		0.99						
49		0.95						
50		0.92						
51		0.98						
52		0.88						
53		0.68						
54		0.65					0.25	
55		1.30						
56		1.10						
57		0.77						

^a Values given in mg/L.

^b Blank entries denote nondetection.

4.3 Soil Composition Data

Table 7 summarizes the results of all other analyses performed in addition to those already discussed. These analytes may be called soil composition parameters.

With respect to the metals data, sample 8 had the highest aluminum, calcium, magnesium, manganese, potassium, and sodium values of any sample and is clearly anomalous compared with the other samples. The reason for this is not known. The only levels noticeably outside APG regional norms were the calcium and magnesium levels in sample 8. The calcium level is 3.6%, compared with a typical range of 0.08%–1.2%, and the magnesium level was 1.3%, compared with a typical range of 0.05%–0.7%.

The “metals” results apparently represent total metals. In samples 1–12, the estimated total level of metal oxides was reported instead of total metals.

The “silicates” heading is misleading, because the value reported by the analytical laboratory as “silicates” is obtained by subtracting the total metals, total organics (presumably obtained by summing up all the organics specifically included on the analyte lists of the various methods), and moisture from 100%. This result does not therefore involve any substances that may be present in soil but were not determined, such as total organic matter. In samples 24–36, moisture was not determined, and the “silicates” result is above 96% in all cases.

TABLE 7 Data Summary — Soil Composition^a

Sample	Method										Solids	Metals	Organics	Moisture	Silicates ^c
	6010	6010	6010	6010	6010	6010	6010	6010	6010	2540G					
	Aluminum	Calcium	Iron	Magnesium	Manganese	Phosphorus	Potassium	Sodium	Titanium						
1	0.63	0.054	1.5	0.120	----	0.016	0.041	----	----	82	6 ^b	<1	18	75	
2	0.68	0.034	2.2	0.150	0.086	0.020	0.035	----	----	80	8 ^b	<1	20	71	
3	0.67	0.031	1.5	0.120	0.011	0.018	0.037	----	----	80	6 ^b	<1	20	73	
4	0.39	0.069	1.1	0.096	----	0.010	0.031	----	----	80	4 ^b	<1	20	75	
5	0.39	0.098	1.3	0.088	----	0.013	0.023	----	----	84	5 ^b	<1	16	78	
6	0.46	0.110	1.2	0.770	0.013	0.011	0.043	----	----	89	6 ^b	<1	11	83	
7	0.37	0.050	1.1	0.120	----	0.010	0.024	----	----	85	4 ^b	<1	15	80	
8	1.00	3.600	1.4	1.300	0.150	0.012	0.140	0.092	----	85	19 ^b	<1	15	66	
9	0.51	0.190	1.3	0.180	0.010	0.012	0.043	0.014	----	84	6 ^b	<1	16	77	
10	0.74	0.042	1.7	0.021	----	0.016	0.057	0.011	----	84	7 ^b	<1	16	77	
11	0.61	0.074	1.4	0.130	0.011	0.014	0.045	----	----	78	6 ^b	<1	22	71	
12	0.54	0.029	1.4	0.140	----	0.013	0.052	----	----	74	6 ^b	<1	26	67	
13	0.74	0.029	1.9	0.180	----	0.014	0.100	0.020	----	80.6	3.0	<0.01	19.4	77.6	
14	0.64	0.061	1.5	0.093	0.013	0.018	0.087	0.015	----	71.9	2.5	<0.01	28.1	69.4	
15	0.61	0.075	1.2	0.110	----	0.014	0.084	0.014	----	76.5	2.1	<0.01	23.5	74.4	
16	0.46	0.460	1.1	0.220	0.024	0.013	0.092	0.013	----	80.0	2.4	<0.01	20.0	77.6	
17	0.54	0.130	1.4	0.180	0.011	0.016	0.087	0.015	----	87.2	2.4	<0.01	12.8	84.8	
18	0.52	0.038	1.4	0.160	----	0.010	0.090	0.015	----	81.2	2.2	<0.01	18.8	79.0	
19	0.49	0.110	1.2	0.260	0.010	0.012	0.093	0.014	----	81.8	2.2	<0.01	18.2	79.6	
20	0.61	0.130	1.2	0.210	0.011	0.016	0.110	0.014	----	87.2	3.1	<0.01	12.8	84.1	
21	0.57	0.200	1.3	0.170	----	0.013	0.089	0.015	----	85.0	2.4	<0.01	15.0	82.6	
22	0.51	0.110	1.2	1.000	0.010	0.010	0.100	0.014	----	86.2	3.0	<0.01	13.8	83.2	
23	0.45	0.460	0.93	0.430	0.020	0.011	0.087	0.012	----	80.3	2.4	<0.01	19.7	77.9	
24	0.647	0.425	1.14	0.313	0.020	----	0.069	----	----	81	2.6	<0.01	----	97.4	
25	0.699	0.554	1.44	0.340	0.030	----	0.076	----	----	82	3.2	<0.01	----	96.8	
26	0.536	0.080	1.10	0.134	----	----	0.055	----	----	84	1.9	<0.01	----	98.1	
27	0.815	0.122	1.46	0.162	----	----	0.087	----	----	79	2.6	<0.01	----	97.4	
28	0.547	0.060	1.74	0.127	0.020	----	0.063	----	----	86	2.6	<0.01	----	97.4	
29	0.414	0.030	0.71	0.080	----	----	0.040	----	----	81	1.3	<0.01	----	98.7	

TABLE 7 (Cont.)

Sample	Method										2540G	Solids	Metals	Organics	Moisture	Silicates ^c
	6010	6010	6010	6010	6010	6010	6010	6010	6010	6010						
	Aluminum	Calcium	Iron	Magnesium	Manganese	Phosphorus	Potassium	Sodium	Titanium							
30	0.678	0.027	1.29	0.094	----	----	0.058	----	----	80	2.2	<0.01	----	97.8		
31	0.541	0.188	1.44	0.227	0.011	----	0.067	----	----	86	2.5	<0.01	----	97.5		
32	0.548	----	1.05	0.098	----	----	0.066	----	----	79	1.8	<0.01	----	98.2		
33	0.499	0.241	0.679	0.156	0.015	----	0.061	----	----	78	1.7	<0.01	----	98.3		
34	0.567	----	0.759	0.063	----	----	0.054	----	----	77	1.4	<0.01	----	98.6		
35	0.720	----	1.040	0.147	----	----	0.092	----	----	77	2.0	<0.01	----	98.0		
36	0.683	----	1.030	0.170	----	----	0.080	----	----	76	2.0	<0.01	----	98.0		
37	0.245	0.057	0.640	0.080	----	----	0.029	----	----	83	1.05	<0.01	17	81.9		
38	0.317	----	0.401	0.071	----	----	0.030	----	----	81	0.82	<0.01	19	80.2		
39	0.301	----	0.402	0.064	----	----	0.031	----	----	80	0.80	<0.01	20	79.2		
40	0.281	----	0.539	0.052	----	----	0.031	----	----	81	0.90	<0.01	19	80.1		
41	0.280	----	0.374	0.054	----	----	0.037	----	----	80	0.74	<0.01	20	79.3		
42	0.208	0.047	0.315	0.038	----	----	0.014	----	----	80	0.62	<0.01	20	79.4		
43	0.188	0.012	0.447	0.027	----	----	0.024	----	----	84	0.71	<0.01	16	83.3		
44	0.217	----	0.556	0.003	----	----	0.022	----	----	85	0.84	<0.01	15	84.2		
45	0.322	0.010	0.669	0.044	----	----	0.029	----	----	85	1.08	<0.01	15	83.9		
46	0.811	----	1.47	0.163	----	----	0.052	0.010	0.012	92	1.40	<0.01	8	90.6		
47	0.708	0.021	1.25	0.116	----	----	0.040	----	----	94	2.10	<0.01	6	91.9		
48	0.860	----	1.87	0.195	----	----	0.043	0.012	0.013	90	3.00	<0.01	10	87.0		
49	0.842	----	1.84	0.185	----	----	0.045	0.016	0.010	89	2.90	<0.01	11	86.1		
50	0.696	----	1.26	0.105	----	----	0.038	----	----	93	----	<0.01	----	----		
51	0.682	----	1.34	0.118	----	----	0.037	----	----	93	2.20	<0.01	7	90.8		
52	0.712	0.010	1.61	0.139	----	----	0.045	0.013	----	91	2.50	<0.01	9	88.5		
53	0.236	----	0.450	0.029	----	----	0.021	----	----	85	0.74	<0.01	15	84.3		
54	0.218	----	0.434	0.032	----	----	0.022	----	----	83	0.71	<0.01	17	82.3		
55	0.523	----	0.719	0.069	----	----	----	----	----	86	1.30	<0.01	14	84.7		
56	----	----	----	----	----	----	----	----	----	----	----	<0.01	----	----		
57	0.313	0.023	0.799	0.051	----	----	0.031	----	----	87	1.20	<0.01	13	85.8		

^a Values given in percent.^b Values reported as metal oxides.^c Obtained by subtracting metals, organics, and moisture percentages from 100%.

5 Summary of Findings

Table 8 summarizes the highest concentrations found for each analyte identified in any of the 57 composite samples taken at the site of the proposed agent disposal facility. The findings of this review are summarized below.

5.1 Analytical Method Suitability

All analytical methods used for the composite samples of interest here are suitable for use, except Method 9020A (Total Organic Halides), which is intended for an aqueous solution matrix, and Method 418.1 (Total Petroleum Hydrocarbons). The suitability of Method 418.1 is

TABLE 8 Highest Concentrations of Identified Organic Contaminants

Substance	Maximum Concentration	Sample Number(s)	Analytical Method
Di-n-butyl phthalate	2,000 µg/kg	18, 21	SVOA; 8270
Diethyl phthalate	3,200 µg/kg	19	SVOA; 8270
Benzyl butyl phthalate	660 µg/kg	19	SVOA; 8270
Cyclotetracosane ^a	3.4 mg/kg	41	SVOA; 8270
1,1,2,2-tetrachloroethane	1,100 µg/kg	15	VOA; 8260
Octamethyl-cyclotetrasiloxane ^b	367 µg/kg	51	VOA; 8260
Trichloroethylene	110 µg/kg	20	VOA; 8260
Tetrachloroethylene	23 µg/kg	23	VOA; 8260
Tridecane ^b	11 µg/kg	6	VOA; 8260
Methylene chloride	10 µg/kg	54	VOA; 8260
Trichlorofluoromethane	9.1 µg/kg	55	VOA; 8260
Hexamethyl-cyclotrisiloxane ^b	9 µg/kg	52	VOA; 8260
Toluene	6.5 µg/kg	51	VOA; 8260
Total organic halides	25 mg/kg	13	TOX; 9020
4,4'-DDE	25.4 µg/kg	55	PCB/pesticides; 8081
4,4'-DDT	21.5 µg/kg	48	PCB/pesticides; 8081
Total "petroleum" hydrocarbons	64 mg/kg	38	TPH; 418.1

^a Identified during SVOA mass-spectral library search; not on Method 8270 analyte list.

^b Identified during VOA mass-spectral library search; not on Method 8260 analyte list.

questionable because of its lack of specificity to petroleum hydrocarbons. Method 8021 (BTEX) is suitable, but redundant, because the same analytes were analyzed to greater sensitivity by other methods already in use.

5.2 Quality Assurance/Quality Control

The quality of the analytical data is considered adequate for their intended use.

5.3 Semivolatile Organics (SVOA)

As discussed in Section 4, it is unlikely that phthalate contamination is as widespread at the site as is indicated by the analytical results. The possibility of phthalate contamination is limited to the area covered by composite samples 13–23, which are associated with the CDB building in Figure 1. However, because phthalates were not detected in other samples from this area (numbers 24, 25, 26, and 57), it is also possible that all the apparent phthalate contamination is a laboratory artifact.

The high-molecular-weight hydrocarbons identified in the SVOA mass-spectral library search are more consistent with known natural sources of such substances than with a petroleum product source. All identified hydrocarbons are known natural products, and no evidence exists for the complex mixture of hydrocarbons characteristic of petroleum fuels.

5.4 Volatile Organics (VOA)

The VOA analytes toluene, methylene chloride, and trichlorofluoromethane are probably not associated with site contamination, but rather with laboratory cross-contamination. However, the presence of the chlorinated hydrocarbons 1,1,2,2-tetrachloroethane, trichloroethylene, and tetrachloroethylene was confirmed independently and in all likelihood represents residual site contamination due to past activities.

The VOA mass-spectral library search identified tridecane and two siloxane derivatives. These are very likely of natural origin and GC column degradation, respectively.

5.5 Total Petroleum Hydrocarbons (TPH)

The TPH results obtained by using Method 418.1 are almost certainly due to naturally occurring hydrocarbons rather than to widespread petroleum hydrocarbon contamination. This finding is based on the lack of specificity of Method 418.1, the conflicting results from samples

tested by using both Method 418.1 and Method 8015 (Modified), the lack of any indication of petroleum hydrocarbons in a large set of independent related analyses, and the identification of several hydrocarbons known to be associated with vegetation.

The area covered by the CDB building (see Figure 1) was found to have generally higher levels of organic contamination than other areas and could also contain traces of petroleum hydrocarbon contamination associated with past activities.

5.6 Pesticides

Pesticides were detected in low part-per-billion levels in a localized wooded area at the northern edge of the site. These substances may be present as a result of past weed control in this area. Analyses for pesticides were not carried out for all composite samples.

5.7 Total Organic Halides

The "single-number" Total Organic Halides data are of questionable value because they were obtained from an inappropriate matrix and do not provide any compound-specific information about possible soil contamination.

5.8 TCLP

The TCLP procedure produces an aqueous leachate solution, which is then subjected to specified analyses for the substances of interest. Only the levels of several heavy metals were above quantitation limits; no organic substance was found above quantitation limits in the TCLP leachate. The found levels of all analytes checked were well below the associated regulatory limits.

5.9 Soil Composition

The soil composition data all lie within normal limits, except for slightly elevated levels of calcium and magnesium in one sample.

6 References

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