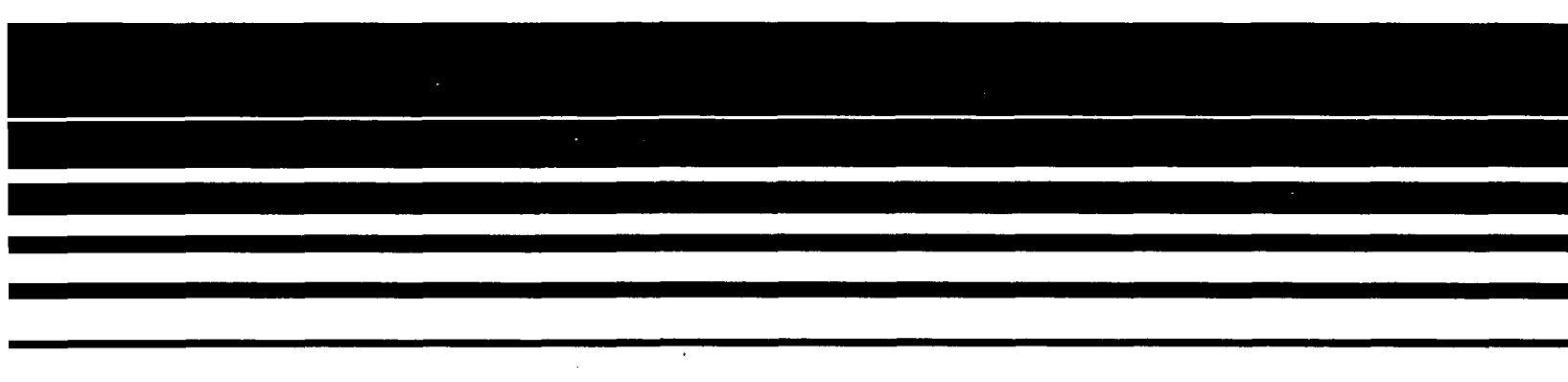


Air



Hazardous Waste Treatment, Storage, and Disposal Facilities

Site-Specific Test Report Texaco Refining and Marketing Company Delaware City, Delaware



SITE-SPECIFIC TEST REPORT

TEXACO REFINING AND MARKETING, INC.
DELAWARE CITY, DELAWARE

ESED 85/12
EMB 85FPE04

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1.0 INTRODUCTION

On October 17 and 18, 1985, Entropy Environmentalists, Inc. collected soil samples from four treatment, storage, and disposal related processes at Texaco Refining and Marketing, Inc. (Texaco) located in Delaware City, Delaware. The purpose of this sampling program was to provide preliminary data on the magnitude of fugitive particulate emissions from various processes at treatment, storage, and disposal facilities (TSDF's) and the degree to which these emissions are contaminated. The U. S. Environmental Protection Agency (EPA) anticipates utilizing the analytical data from this program with emission models to estimate fugitive particulate emissions from TSDF's. The information generated by this study may ultimately be used by the Office of Air Quality Planning and Standards (OAQPS) of EPA to assess the adequacy of regulations governing fugitive particulate emissions from TSDF's.

To accomplish the overall goals of this study, soil samples were collected from representative processes at this plant and were submitted for the appropriate analyses in order to determine the following:

- The percent by weight of silt in the soil (i.e., material that passes through a 200 mesh screen and has a nominal diameter less than 75 μ m) and the percent by weight of moisture in the soil.
- The degree of contamination in the soil silt fraction of metals, semivolatile organics, and pesticides.
- The percent by weight of soil silt that is less than 20 μ m in diameter based on a sonic sieving technique.

- o The particle size dependency of the degree of contamination (i.e., greater or lesser degree of contamination in particles with diameters not in excess of 20 um) by conducting separate analyses of different soil particle size fractions.
- o The repeatability and reproducibility of the sampling and analytical procedures for the entire sampling program (only raw data are included in this report; a statistical summary will be presented for all sampling sites in a later report).

At Texaco, the four processes sampled were three different cells within the land treatment unit and unpaved road segments (3) within the land treatment site. A pair of background samples along with samples to assess the repeatability and reproducibility of the method were also taken.

Samples taken were analyzed for silt content, PM_{10} content, metals, cyanide, and semivolatile organics as described in Chapter 4. Research Triangle Institute (RTI) conducted the analyses for metals and PEI and Associates performed the analyses for the semivolatile organics. Additional cleanup of the semivolatile organic extracts was performed by Triangle Laboratories, Inc. The outside laboratory that performed the metals analysis on the reproducibility samples was PEI and Associates. For a cost benefit, all the reproducibility samples for the entire study were analyzed at the same time. EPA decided not to have the repeatability and reproducibility samples analyzed for semivolatile organics because of their high oil and grease content.

Field sampling was performed by Mr. Steve Plaisance and Mr. Bernie von Lehmden of Entropy Environmentalists. Mr. Phillip Englehart and Dr. Tom Lapp of Midwest Research Institute (MRI) directed Entropy personnel regarding specific processes to be sampled, the boundaries of the processes, and recorded the pertinent process and operating characteristics. Mr. Gene Riley (EPA Task Manager) of the Emission Measurement Branch (EMB) observed the sampling program. Mr. Bob Wojewodski, Senior Environmental Engineer, served as the principal contact for Texaco.

This report is organized into several chapters that address various aspects of the sampling and analysis program. Immediately following this chapter is the "Summary and Discussion of Results" chapter which presents table summaries of data on silt and PM_{10} content and degree of contamination for each sample fraction analyzed. Following the "Summary and Discussion of Results" chapter is the "Process Description" chapter (supplied by MRI) which includes descriptions of each process sampled. The next chapter, "Sampling and Analysis," presents the plot plan and sampling grid for each process. The method of selecting the sampling grid and the sample collection procedures are outlined, including any deviations and problems encountered. This chapter also describes the sample preparation and analytical procedures used for each sample; any deviations from the normal procedures are addressed. The appendices present the Raw Field Data and Sampling Logs (Appendix A); Analytical Data (Appendix B); detailed Sampling and Analytical Procedures (Appendix C); Sampling Program Participants and Observers (Appendix D); and Process Operations Data (Appendix E).

2.0 SUMMARY AND DISCUSSION OF RESULTS

This chapter presents a summary of the sampling and analysis results and a brief discussion of significant deviations from the proposed sampling and analysis protocol for this program. Since the standard sampling and analytical procedures are not addressed in this chapter, it is recommended that those individuals who are not familiar with the sampling and analytical procedures used in this study review Chapter 4, "Sampling and Analysis," prior to reading this chapter.

Soil samples were collected from four processes at Texaco's refining and marketing facility located in Delaware City, Delaware. The processes included: (1) the land treatment unit, Cell #4, (2) the land treatment unit, Cell #3; (3) the land treatment unit, Cell #8; and (4) three segments of unpaved roads within the land treatment unit. Sampling and analysis were conducted using the procedures described in the Sampling and Analysis Protocol which was written specifically for this sampling program. The protocol was provided to the facility prior to the sample collection. The procedures described in this protocol are described again in detail in Chapter 4 and Appendix C of this report.

As described in the Sampling and Analysis Protocol, this site-specific report is intended to present the data relevant to the samples obtained at one site in this study and the procedures used to obtain these samples. Some statistical analyses will be performed on the data concerning this site; however, the majority of statistical analyses will involve the data collected over the entire study, including the repeatability and reproducibility and quality assurance data, and will be included in the summary report to be

completed at the conclusion of the program. With the exception of the data from the screening conducted to determine silt contents, there is not sufficient data to conduct meaningful statistical analyses on a site- or process-specific basis.

The sampling plan for Texaco is shown in Table 2.1. The sampling procedures were designed to obtain a representative sample of that portion of the contaminated soil with the potential to become airborne. The analyses of the collected samples were designed to measure the concentration of the most likely compounds or elements that could be soil contaminants (metals, cyanide, semivolatile organics, and pesticides). The sample collection techniques were generally as follows: (1) for undisturbed hard surfaces a sweeping technique was used to obtain surface samples only; (2) for moderately disturbed surfaces a scooping technique was used to obtain near surface samples; and (3) for surfaces that were mechanically disturbed to a specific depth, coring was used to sample to the depth of the disturbance. The number of samples collected within each process was a function of the variability expected in the degree of contamination and/or the amount of sample that was needed for the analyses.

According to the Sampling and Analysis Protocol, the collected samples were to be analyzed for metals, cyanide, and the semivolatile organics and pesticides found on the hazardous substance lists (HSL) in the EPA Contract Laboratory Program (CLP), Statement of Work. If significant quantities of cyanide, semivolatile organics, or pesticides were not expected to be present in a particular process, then analysis for those compounds was not performed. MRI decided that at this particular site, cyanide and pesticides would not be present in significant quantities in any process sampled, and therefore, cyanide and pesticide analyses were deleted. All samples were analyzed for metals and semivolatile organics, except the repeatability and reproducibility

TABLE 2.1. SAMPLING PLAN FOR TEXACO

Process Sampled	Process Designation	Number of Samples	Collection Method	Analyses
Land Treatment Unit, Cell #4	L	8*	Modified coring** (stainless tube)	Loss on Drying Silt and PM ₁₀ Content Semivolatile Organics Oil and Grease content
		8*	Modified coring** (plastic tube)	Loss on Drying Silt and PM ₁₀ Content Metals
Land Treatment Unit, Cell #8	N	8	Scooping	Loss on Drying Silt and PM ₁₀ Content Metals Semivolatile Organics Oil and Grease Content
Land Treatment Unit, Cell #3	O	8	Scooping	Loss on Drying Silt and PM ₁₀ Content Metals Semivolatile Organics Oil and Grease Content
Unpaved Roads Within Land Treatment Unit	M	3	Sweeping	Loss on Drying Silt and PM ₁₀ Content Metals Semivolatile Organics
Background Samples Within Land Treatment Unit	BGD	2	Scooping	Loss on Drying Silt and PM ₁₀ Content Metals Semivolatile Organics

* One to two cores for metals analysis (plastic core tube) and one to two cores for organics analysis (stainless core tube) were taken from each of 8 single grid cells.

**For each organic sample and each metal sample, 1 to 2 cores approximately two to three inches deep were taken.

samples. They were only analyzed for metals since the presence of oil and grease was anticipated to interfere with the semivolatile organics analysis. Complete lists of compounds or elements for which analyses were conducted and their detection limits are presented in Chapter 4 (see Tables 4.2 and 4.3).

The analytical results are discussed in the following subsections. Complete sampling data sheets are presented in Appendix A and all analytical data sheets are presented in Appendix B.

2.1 BACKGROUND SAMPLES

Background samples were collected at a point not used for TSDf activities and analyzed, because many compounds and elements are either naturally occurring in the soil or may be present as a result of factors other than those which may be attributed to Texaco's activities. The percent weight loss on drying (LOD) measured for BGD-420 was 9.46 percent by weight. The samples were then oven-dried at 105°C for 4 hours prior to being screened for silt content. The silt content of the two background samples (sample identification numbers BGD-420 and BGD-421) averaged 13.7 percent by weight (see Table 2.2). The composite silt material (sample identification number BGD-446) separated from the background samples was sonic sieved. Material passing through a 20 um sieve constituted the PM₁₀ content. The PM₁₀ content averaged 30.82 percent by weight of the silt material. The silt screening did not produce a sufficient amount of silt to allow for the production of the "greater than PM₁₀" (>PM₁₀) and PM₁₀ fractions for chemical analyses. "Greater than PM₁₀" refers to the fraction of the silt material that does not pass through the 20 um sieve.

Results of the analyses for metals and semivolatile organics are shown in Table 2.3. The analytical results for the metals and cyanide in the background silt sample (sample ID BGD-455) are in terms of micrograms of the metal per

TABLE 2.2.
ANALYTICAL RESULTS OF SILT SCREENING, WEIGHT LOSS ON DRYING, AND PM₁₀ SIEVING
FUGITIVE PARTICULATE FROM TSDF (85/12)
TEXACO, DELAWARE CITY, DELAWARE

Site and Process	Sample ID	Percent Silt	Percent Loss on Drying	Sample ID	Percent PM ₁₀
Texaco, Delaware City Land Treatment, Cell #4 (Process L)	L-401-M	11.8	28.03		
	L-402-M	6.9	21.85		
	L-403-M	8.2	28.50		
	L-404-M	3.5	28.06		
	L-405-M	6.8	26.78		
	L-406-M	11.3	26.02		
	L-407-M	5.7	25.95		
	L-408-M	6.3	33.82		
	L-401-O	9.1	28.09		
	L-402-O	5.6	22.48		
	L-403-O	8.0	22.97		
	L-404-O	3.0	30.07		
	L-405-O	7.4	27.52		
	L-406-O	7.4	32.03		
	L-407-O	4.2	28.75	L-433	8.51
	L-408-O	8.9	29.51	L-437	12.63
	Average	7.1	27.53		10.57
	Std. Dev.	2.5	3.24		2.92
Texaco, Delaware City Land Treatment Roads (Process M)	M-409	8.2	1.65	M-440	1.29
				M-440	2.76
				Average	2.03
				Std. Dev.	1.04
	M-410	19.7	1.19	M-443	29.92
				M-443	34.94
				Average	32.43
				Std. Dev.	3.55
	M-411	10.4	1.73	M-446	20.52
				M-446	21.12
				Average	20.82
				Std. Dev.	0.42

(continued)

TABLE 2.2. (continued)

Site and Process	Sample ID	Percent Silt	Percent Loss on Drying	Sample ID	Percent PM ₁₀
Texaco, Delaware City Land Treatment, Cell #8 (Process N)	N-412	10.9	21.89		
	N-413	12.4			
	N-414	14.5			
	N-415	10.3			
	N-416	13.2			
	N-417	11.5			
	N-418	12.9		N-453	17.06
	N-419	10.2		N-453	18.65
	Average	12.0			17.85
	Std. Dev.	1.5			1.12
Texaco, Delaware City Background Samples	BGD-420	16.4	9.46		
	BGD-420	15.4			
	BGD-421	12.5		BGD-446	31.92
	BGD-421	10.4		BGD-446	29.72
	Average	13.7			30.82
	Std. Dev.	2.7			1.56
Texaco, Delaware City Land Treatment, Cell #3 (Process O)	O-422	5.6	30.49		
	O-423	5.9			
	O-424	5.8			
	O-425	5.6			
	O-426	6.9			
	O-427	4.4			
	O-428	7.7		O-463	5.81
	O-429	7.0		O-463	5.24
	Average	6.1			5.52
	Std. Dev.	1.0			0.40
Texaco, Delaware City Land Treatment, Cell #3 Repeatability and Reproducibility	O-422rr1	6.3	28.96		
	O-422rr2	7.8	32.71		
	O-422rr3	6.8	29.47		
	O-422rr4	6.5	30.78		
	O-422rr5	8.3	27.91		
	Average	7.1	29.97		
	Std. Dev.	0.9	1.85		

(continued)

TABLE 2.2. (continued)

Site and Process	Sample ID	Percent Silt	Percent Loss on Drying	Sample ID	Percent PM ₁₀
Texaco, Delaware City Land Treatment, Cell #3 Repeatability and Reproducibility	0-423rr1	7.1	31.70		
	0-423rr2	8.3	29.23		
	0-423rr3	4.5	35.53		
	0-423rr4	6.1	28.56		
	0-423rr5	11.7	32.48		
	Average	7.5	31.50		
	Std. Dev.	2.7	2.79		

	0-425rr1	6.7	27.75		
	0-425rr2	1.9	30.41		
	0-425rr3	3.7	33.21		
	0-425rr4	2.7	31.49		
	0-425rr5	7.8	27.98		
	Average	4.6	30.17		
	Std. Dev.	2.6	2.33		

TABLE 2.3. ANALYTICAL RESULTS FOR METALS AND SEMIVOLATILE ORGANICS
FUGITIVE PARTICULATE FROM TSDF (85/12)

Metals Analysis	Land Treatment Cell #8			Cell #4	Cell #3	Roads	Background
Sample Identity	Silt N-448	PM10 N-450	>PM10 N-452	Silt L-430	Silt O-458	Silt M-439	Silt BDG-455
Element	(ug/g)	(ug/g)	(ug/g)	(ug/g)	(ug/g)	(ug/g)	(ug/g)
Aluminum (Al)	11,100	13,200	940	11,000	11,900	11,400	14,000
Antimony (Sb)	1.1	0.8	0.6	<0.5	0.8	<0.5	2.5
Arsenic (As)	6.7	6.7	6.2	6.6	7.4	5.9	5.2
Barium (Ba)	152	215	272	106	190	114	59.9
Beryllium (Be)	<1	<1	<1	<1	<1	<1	<1
Cadmium (Cd)	<1	<1	1.9	<1	<1	1.4	1.5
Chromium (Cr)	209	255	196	141	142	96.7	21.3
Cobalt (Co)	16.9	20.6	14.7	17.9	17.7	16.5	9.5
Copper (Cu)	207	219	200	164	198	110	31.9
Iron (Fe)	18,400	21,400	17,600	22,400	21,400	19,700	19,400
Lead (Pb)	57.0	81.0	65.0	74.0	92.0	49.0	15.0
Manganese (Mn)	407	473	389	358	508	392	206
Mercury (Hg)	1.3	1.3	1.5	0.9	1.6	0.9	0.1
Molybdenum (Mo)	9.2	9.5	5.7	9.9	<2	<2	<2
Nickel (Ni)	98.3	108	94.4	86.8	150	83.8	13.6
Osmium (Os)	<1	<1	<1	<1	<1	<1	<1
Selenium (Se)	2.3	2.5	3.1	4.2	3.2	2.2	<0.5
Silver (Ag)	<2	<2	<2	<2	<2	<2	<2
Thallium (Tl)	<2	<2	<2	<2	<2	<2	<2
Vanadium (V)	200	227	190	267	352	207	55.0
Zinc (Zn)	248	287	232	225	296	225	35.6
Semivolatile Analysis	Land Treatment Cell #8			Cell #4	Cell #3	Roads	Background
Sample Identity	Silt N-447	PM10 N-449		Silt L-434	Silt O-457	Silt M-438	Silt BDG-454
	(ug/g)	(ug/g)		(ug/g)	(ug/g)	(ug/g)	(ug/g)
2-Methylnaphthalene	N.D.	N.D.		N.D.	45.0 J	15.0 J	1.4 J
Phenanthrene	N.D.	N.D.		N.D.	22.0 J	36.0 J	0.83 J
Pyrene	N.D.	N.D.		10.0 J	9.5 J	11.0 J	N.D.
Sample Detection Limit	78.6	49.5		54.4	85.6	61.2	3.3

N.D. = less than the sample's quantifiable detection limit

J = Estimated value where the compound meets the spectral criteria but the result is less than the quantifiable limit.

gram of silt sample (dry basis). These results reflect the nominal concentrations of these materials present in the soil which are not a result of Texaco's activities. The results for the background samples have not been subtracted from the results for the other samples since the risk assessments use the inclusive value of the degree of contamination. It should be understood, however, that the actual outside contribution to the degree of contamination of the soil is that portion of the contaminate concentration which exceeds the nominal background level.

For the analysis of the semivolatile organic compounds, the background samples were extracted as low-level samples following the U.S. EPA Contract Laboratory Program, Statement of Work for Organic Analysis, 7/85 Revision (referred to as the CLP in this report). The extract was concentrated and subjected to an adsorption chromatography cleanup procedure instead of the CLP gel permeation chromatography (GPC) cleanup procedure. The adsorption chromatography procedure was developed to remove more aliphatic compounds from the extract than possible with the GPC procedure. The presence of excessive amounts of aliphatic material would require dilution of the extracts prior to the gas chromatograph/mass spectrometer (GC/MS) analysis and a corresponding increase in the sample detection limit. The dilution would be necessary to protect the GC/MS from the large amounts of aliphatic compounds present in some samples (particularly land treatment samples that contain considerable amounts of oil and grease).

For the background sample a 10-fold dilution, determined by GC/flame ionization detection (GC/FID), was required prior to the GC/MS analysis. Two of the CLP hazardous substance list (HSL) semivolatile compounds were found, but at concentrations below the quantifiable detection limit. The compounds detected met the mass spectral criteria, but the values reported are estimates only.

2.2 LAND TREATMENT UNIT, CELL #4 (PROCESS L)

Cell #4 in the land treatment unit (Process L) was sampled using a coring technique to obtain samples to a depth of approximately two to three inches, depending upon the soil conditions in each cell sampled. A sampling grid was laid out and eight randomly selected cells were sampled. Because of the potential for contamination from materials used in construction of the core sampling equipment, the samples taken for metals analysis (sample identification numbers L-401-M through L-408-M) were sampled with a PVC coring tube, and the samples taken for semivolatile organics analysis (sample identification numbers L-401-O through L-408-O) were sampled with a stainless steel coring tube. The oil and grease content was determined on a composite sample made of aliquots taken from all sixteen samples from Cell #4. The oil and grease content of the composite sample was 6.11 percent expressed on a dry basis (see Table 2.4). The weight loss on drying averaged 27.53 percent by weight (see Table 2.2). Following oven-drying at 105°C for 6 hours, the sixteen samples were screened for silt content which averaged 7.1 percent by weight.

The silt fractions separated from the samples taken for organics analysis and those taken for metals analysis were each sonic sieved for PM_{10} content which averaged 10.57 percent by weight of the silt. The silt screening did not produce sufficient silt material to allow the production of $>PM_{10}$ and PM_{10} material by sonic sieving for chemical analyses. Aliquots of the silt fractions (one for metals and one for organics) were taken for semivolatile organics and metals analysis. These were analyzed separately to determine the degree of contamination. The analytical results for metals and semivolatile organics are shown in Table 2.3. Like the background silt sample, the land treatment Cell #4 silt sample was extracted by the low-level method. The

TABLE 2.4. SUMMARY OF OIL AND GREASE ANALYSIS
FUGITIVE PARTICULATE FROM TSDF (85/12)

Process ID	Process Description	Site	Oil and Grease
L	Land Treatment, Cell#4	Texaco, Delaware City	6.11%
N	Land Treatment, Cell#8	Texaco, Delaware City	8.46%
O	Land Treatment, Cell#3	Texaco, Delaware City	8.92%

QUALITY ASSURANCE SUMMARY FOR OIL AND GREASE ANALYSIS

Process ID	Sample Description	Site	Oil and Grease
Total Repeatability			
O	Oil&Grease O-rr1 Comp	Texaco, Delaware City	6.94%
O	Oil&Grease O-rr1 Comp	Texaco, Delaware City	7.91%
			=====
			Mean 7.43%
			RPD 0.48%
Analytical Repeatability			
O	Oil&Grease O-rr1 Comp	Texaco, Delaware City	7.91%
O	Oil&Grease O-rr1 Comp	Texaco, Delaware City	7.30%
			=====
			RPD 0.30%
Sampling Reproducibility			
O	Oil&Grease O-rr4 Comp	Texaco, Delaware City	8.12%
O	Mean of O-rr1 Comp		7.43%
			=====
			RPD 0.35%

Performance Audit

	Expected	Found	Recovery
BGD Spiked with 34 mg of paraffin oil	0.39%	0.36%	92.3%

extract was concentrated and cleaned by the adsorption chromatography procedure. The sample extract required a 165-fold dilution for the GC/MS analysis. One semivolatile organic compound was detected in the sample from Cell #4. The contamination of the compound was below the quantifiable detection limit of 54.4 ug/g.

With the exception of the use of the adsorption chromatography cleanup procedure, the dilution of the semivolatile organic sample extracts prior to analysis, and the increased quantifiable detection limit, all procedures followed the Sampling and Analysis Protocol.

2.3 LAND TREATMENT UNIT, CELL #8 (PROCESS N)

Also in the land treatment unit, Cell #8 (Process N) was sampled using a grid layout. Eight samples were collected within this grid in a random manner as described in Chapter 4. The scoop sampling technique was employed. The determination of oil and grease content and LOD was conducted on sample N-412. The oil and grease content was 8.46% expressed on a dry weight basis (see Table 2.4). The LOD for sample N-412 was 21.89 percent (see Table 2.2). All eight samples were oven dried at 105°C for 6 hours and desiccated overnight prior to silt screening. Each of the eight dried samples (sample identification numbers N-412 through N-419) was screened for silt content which averaged 12.0 percent silt by weight (see Table 2.2). The two jars of silt (sample identification number N-453), resulting from screening samples N-412 through N-419, were then sonic sieved for PM₁₀ content which averaged 17.85 percent by weight in the silt sample. Portions of the three fractions (silt, >PM₁₀, and PM₁₀) produced from the combined silt sample from Cell #8 were analyzed for metals. All three fractions were analyzed for metals to determine if the degree of contamination was less or greater in the PM₁₀ fraction

(particle size dependent). The results for the metals are expressed in micrograms of the metal per gram of sample on a dry basis. The concentrations measured for the background sample were not subtracted from the sample results.

As a cost saving measure, only the silt and PM₁₀ fractions were analyzed for semivolatile organics to determine the particle size dependency of the degree of contamination. Like the background silt sample, the land treatment Cell #8 silt and PM₁₀ fractions were extracted by the low-level CLP procedure. The extracts were concentrated and subjected to the adsorption chromatography cleanup procedure. Prior to the GC/MS analysis, the silt extract was diluted 238-fold and the PM₁₀ extract was diluted 150-fold. None of the semivolatile HSL compounds were detected in the silt or PM₁₀ extracts at the quantifiable detection limits of 78.6 ug/g and 49.5 ug/g, respectively.

With the exception of using the adsorption chromatography cleanup procedure, diluting the extracts prior to the GC/MS analysis, and the increased quantifiable detection limit, all procedures followed the Sampling and Analysis Protocol.

2.4 LAND TREATMENT UNIT, CELL #3 (PROCESS 0)

Cell #3 of the land treatment unit (Process 0) was sampled using the scooping technique. A sampling grid was laid out and eight randomly selected cells were sampled. The determinations of the oil and grease content and LOD were conducted on aliquots of sample 0-429 taken from Cell #3. The oil and grease content of the sample was 8.92 percent expressed on a dry weight basis (see Table 2.4). The LOD for sample 0-422 was 30.49 percent (see Table 2.2). All samples (0-422 through 0-429) were oven-dried at 105°C for 6 hours and desiccated overnight prior to screening. The resulting dried samples were screened for silt content which averaged 6.1 percent by weight.

The silt separated from the samples (sample ID number 0-463) was sonic sieved from PM_{10} content which averaged 5.52 percent by weight of the silt. Since the amount of silt composite resulting from the silt screening was low and the PM_{10} content of the silt was also low, the decision was made not to produce PM_{10} or $>PM_{10}$ for chemical analyses. The results for the analyses done on the silt fraction for metals and semivolatile organics are shown in Table 2.3. Three semivolatile organic compounds were detected in the silt samples from the Cell #3. The silt sample was prepared for organic analysis like the background samples. The extract was diluted 259-fold prior to the GC/MS analysis. All of the compounds were below the quantifiable detection limit of 85.6 ug/g, which means that the reported compounds were identified, but the magnitude of the results are only an estimate. The analytical results for the background sample were not subtracted from the sample results.

With the exception of using the adsorption chromatography cleanup procedure, diluting the extracts prior to the GC/MS analysis, and the increased quantifiable detection limit, all procedures followed the Sampling and Analysis Protocol.

2.5 UNPAVED ROADS IN LAND TREATMENT UNIT (PROCESS M)

Three segments of unpaved roads within the land treatment unit were sampled. These segments were located (1) at the main gate to the unit (sample M-409), (2) on the north-south access road at the midpoint of Cell #2 (sample M-410), and (3) on the east road between Cells #3 and #4 (see Figure 4.1b). Each segment was sampled using the sweeping technique.

A brush was used to sweep loose particulate from an 8-inch wide strip across the width of each segment. Two sample jars were filled with each sample. After drying, the samples were screened for silt content which

averaged 8.2, 19.7, and 10.4 percent by weight, for sample numbers M-409, M-410, and M-411, respectively (see Table 2.2). The weight losses on drying were 1.65, 1.19, and 1.73 percent by weight, respectively. The silt samples obtained from these samples were sieved for PM_{10} content which averaged 2.03, 32.43, and 20.82 percent by weight for silt samples M-440, M-443, and M-446, respectively. The low PM_{10} content of road sample M-440 as compared with the other two road samples (M-443 and M-446) correlated with the relatively low silt content of the same sample. This was possibly due to the oil and grease content of the road sample; however, this cannot be confirmed since the oil and grease content of this sample was not measured. Since a sufficient quantity of silt was not obtained from the silt screening, PM_{10} and $>PM_{10}$ fractions were not produced for the analysis of the metals and semivolatile organics. The silt fractions from these three samples were combined.

The results of metal and semivolatile organic analyses for the composite silt sample are presented in Table 2.3. The concentrations measured for the background sample were not subtracted from the results for the unpaved road silt sample. The silt sample was prepared for organic analysis like the background sample and diluted 185-fold prior to GC/MS analysis. Three semivolatile organic compounds were found in the silt sample and were below the quantifiable detection limit of 61.2 ug/g, (i.e., the mass spectral criteria for these compounds were met for identifying the compounds, but the actual magnitudes reported are only estimated values).

With the exception of using the adsorption chromatography cleanup procedure, diluting the extracts prior to GC/MS analysis, and the increased quantifiable detection limit, all procedures followed the Sampling and Analysis Protocol.

2.6 REPEATABILITY AND REPRODUCIBILITY

As discussed in more detail in Chapter 4 and Appendix C, additional samples were collected in three of the sampled grid cells in Cell #3 of the land treatment unit (Process 0) for use in measuring the sampling and analysis repeatability and reproducibility. The silt content values for these samples are presented in Table 2.2. The results for the metals analysis for these samples are presented in Table 2.5. Analyses for semivolatile organic compounds were not conducted on these samples because the detection limits for the samples were expected to be high due to the presence of oil and grease in the samples. A summary report presenting the repeatability and reproducibility results for the entire study will be completed at the end of the study.

The repeatability and reproducibility samples were also used for the oil and grease analysis. For total repeatability, the composite sample (sample ID 0-rr1) made of aliquots from 0-422rr1, 0-423rr1, and 0-425rr1 was analyzed in duplicate (see Table 2.4). For analytical repeatability (defined here as the gravimetric determination of the extracted residue), the gravimetric determination was conducted twice on the sample extract. Sampling reproducibility was determined by comparing the oil and grease content of a composite sample (sample ID 0-rr4) made of aliquots of 0-422rr4, 0-423rr4 and 0-425rr4 to the oil and grease content of the 0-rr1 composite. The rr4 samples were taken from the same cells by a secondary sampler.

A performance audit was also conducted by spiking a background sample (from another site) with paraffin oil dissolved in Freon TF. The spiked sample was carried through the oil and grease analysis procedure. The recovery of the paraffin oil was calculated to be 94 percent (see Table 2.4).

TABLE 2.5. ANALYTICAL RESULTS FOR REPEATABILITY AND REPRODUCIBILITY METALS SAMPLES
FUGITIVE PARTICULATE FROM TSDF (85/12)

Sample Identity	Grid No. 2					Grid No. 7					Grid No. 16				
	RTI	PEI	RTI	RTI	PEI	RTI	PEI	RTI	RTI	PEI	RTI	PEI	RTI	RTI	PEI
	0-465	0-467	0-471	0-472	0-473	0-475	0-477	0-481	0-482	0-483	0-485	0-487	0-491	0-492	0-493
Element	(ug/g)	(ug/g)	(ug/g)	(ug/g)	(ug/g)	(ug/g)	(ug/g)	(ug/g)	(ug/g)	(ug/g)	(ug/g)	(ug/g)	(ug/g)	(ug/g)	(ug/g)
Aluminum (Al)	12,400	7,997	13,700	12,900	10,080	10,500	10,390	10,500	10,100	7,630	13,600	10,050	12,500	12,300	10,970
Arsenic (As)	6.9	10.9	7.7	7.1	7.3	5.6	4.2	6.5	6.4	5.9	7.9	8.6	8.4	7.6	5.7
Barium (Ba)	109	138	121	121	114	105	54.0	113	678	104	146	155	145	228	70
Beryllium (Be)	<1	<0.1	<1	<1	0.2	<1	0.2	<1	<1	0.5	<1	<0.1	<1	<1	<0.1
Cadmium (Cd)	<1	68.3	<1	<1	1.1	<1	0.9	<1	<1	0.8	<1	1.5	<1	<1	1.1
Chromium (Cr)	137	181	121	124	102	108	79.1	116	113	85.6	145	104	149	147	114
Cobalt (Co)	14.5	20.2	15.1	16.9	17.0	14.7	12.2	15.0	15.2	14.4	18.6	21.1	21.1	20.7	15.0
Copper (Cu)	174	346	170	188	154	154	176.0	168	153	134	199	221	212	204	178
Iron (Fe)	21,000	32,500	21,700	21,200	17,920	17,000	7,340	18,600	18,300	14,600	22,900	19,010	22,700	22,500	11,600
Lead (Pb)	69.0	2,620	67.0	58.0	67.2	65.0	38.7	60.5	66.0	50.0	81.3	71.2	86.4	81.0	48.4
Manganese (Mn)	425	2,100	498	499	450	381	504	400	401	330	664	840	671	659	591
Mercury (Hg)	1.1	1.2	1.1	1.2	1.1	0.8	1.2	0.8	0.9	1.0	1.2	2.3	1.3	1.4	1.7
Nickel (Ni)	141	34.4	1645	168	138	504	232	489	485	403	217	269	211	207	176
Selenium (Se)	2.5	2.2	3.4	3.0	0.5	3.2	0.8	2.7	2.9	0.6	3.6	<0.3	2.9	3.8	<0.3
Silver (Ag)	<2	8.3	<2	<2	19.8	<2	<0.7	<2	<2	<0.7	<2	<0.7	<2	<2	<0.7
Vanadium (V)	483	21.0	479	484	384	2,960	551	2,820	2,810	2,160	440	392	414	408	284
Zinc (Zn)	233	27,280	249	248	291	201.0	264	209	207	179	330	419	341	332	309

Sample Identity	Unspiked Sample		Found	RTI Percent Recovery	Unspiked Sample		Found	PEI Percent Recovery
	-----	Spike	-----		Spike	-----		
	0-458	Amount	0-495		0-458	Amount	0-497	
Element	(ug/g)	(ug/g)	(ug/g)		(ug/g)	(ug/g)		
Aluminum (Al)	11,900	0	-	-	11,900	0	6,577	-
Arsenic (As)	7.4	91.7	74.1	72.7%	7.4	89.4	74	74.5%
Barium (Ba)	190	91.7	256	72.3%	190	89.4	211	23.6%
Beryllium (Be)	<1	91.7	93.1	101.5%	<1	89.4	79	88.6%
Bismuth (Bi)	<10	0	-	-	<10	0	0	-
Cadmium (Cd)	<1	91.7	90.3	98.5%	<1	89.4	80	89.1%
Chromium (Cr)	142	0	168	-	142	0	95	-
Cobalt (Co)	17.7	0	20.0	-	17.7	0	13	-
Copper (Cu)	198	91.7	316	129.0%	198	89.4	224	28.7%
Iron (Fe)	21,400	0	-	-	21,400	0	13,069	-
Lead (Pb)	92.0	91.7	195	112.5%	92.0	89.4	126	37.7%
Manganese (Mn)	508	91.7	673	179.4%	508	89.4	422	-96.0%
Mercury (Hg)	1.6	0	-	-	1.6	0	0	-
Nickel (Ni)	150	0	162	-	150	0	96	-
Selenium (Se)	3.2	91.7	70.0	72.8%	3.2	89.4	57	60.7%
Silver (Ag)	<10	91.7	67.0	73.1%	<10	89.4	60	66.8%
Vanadium (V)	352	0	356	-	352	89.4	199	-
Zinc (Zn)	296	0	443	-	296	0	294	-

2.7 CONCLUSIONS

No major problems were encountered during sample collection. It was felt that the sampling program was successful in obtaining representative samples.

In the analyses of the samples, no problems were encountered in obtaining silt content or determining PM_{10} content. The results of the metals analyses are also believed to be accurate.

The only significant problem encountered during the analyses was the fact that the samples contained a significant amount of organics (oil and grease) not found on the Hazardous Substances List. This prevented the semivolatile organics analyses from being conducted at the level described in the analytical protocol. Because of the high concentrations of organics, an alternative sample cleanup procedure was used on the samples to remove these organics. The cleanup procedure used on the semivolatile organic sample extracts appeared to have little effect on the samples from this site. However, the detection limits for these samples were believed to be the lowest levels practical for the analysis of HSL semivolatile compounds by GC/MS.

3.0 PROCESS DESCRIPTION

At this facility, sampling was undertaken for four processes, where the term "process" refers to a likely source of potentially contaminated fugitive particulate emissions. All of the process samples were taken within the facility's land treatment unit; processes sampled include:

- a. Three different cells within the land treatment unit; and
- b. Unpaved road segments at three locations within the land treatment unit.

The following process descriptions are based largely upon the information provided by the facility and observations made during the course of the survey/sampling effort.

3.1 LAND TREATMENT CELLS

The actual working surface of the land treatment unit is approximately 34 acres, and is configured into 12 discrete cells (see facility map, Figure 4.2). At the time of survey, 11 of the 12 cells were being used on a strict rotation basis. Average cell size is 2.85 acres; the cells range in size from 2.36 to 3.40 acres. The unit has been in operation for about 3 years.

The land treatment unit is used exclusively to dispose of wastes generated at the facility. Principal wastes and approximate quantities treated during the past year are shown below.

<u>EPA Hazardous Waste No.</u>	<u>Quantity (tons)</u>	<u>Description</u>
K050 and K051	2,000	API and CPI oil-water gravity separator bottoms
K048 and K049	1,850	Vacuum filter cake (VFC) from oil recovery unit at facility wastewater treatment plant (WWTP)
Non-RCRA waste	8,000-20,000	Dual cell gravity solids (DCG)--biosludge from WWTP

In addition to these waste streams, the unit occasionally receives tank bottoms associated with storage tank cleanup, as well as oil-contaminated soil.

The principal equipment types, functions, and approximate level of activity for the treatment unit are summarized below.

<u>Equipment (commercial designation if available)</u>	<u>Function</u>	<u>Activity Units</u>
Farm tractor (Ford 7700) with implements: 1. Chisel plow 2. Disc harrow 3. Lime bucket 4. Backdrag	Chisel plow and disc harrow used for initial incorporation and subsequent soil cultivation, respectively. Lime bucket used to add lime to soil for pH control. Backdrag used to smooth surface prior to waste application.	Chisel plow used minimum 4 to 6 times/yr. Disc harrow used minimum of 8 to 12 times/yr. Lime bucket on as-needed basis. Backdrag minimum 4 to 6 times/yr.
Bulldozer (John Deere 350c)	Functions include spreading of solid material on treatment plots, and pulling of backdrag over surface.	Based on survey observations activity--4 to 6 hr/day.
Two vacuum trucks--4 axles, 14 wheels	Delivery of high liquid content wastes to treatment plots.	3,000-gal. capacity; avg. 3 loads/day, when operating.
Dump truck--3 axles, 10 wheels	Delivery of solid wastes to treatment plots	Plant considers "full load" ~ 14,500 lb; 2 loads/day.

The basic sequence of operations for the unit are as follows:

- a. Waste application;
- b. Initial incorporation (chisel plow);
- c. Addition of lime as needed (lime bucket);
- d. Soil cultivation (disc harrow);
- e. Smooth surface (backdrag); and
- f. Repeat steps d and e.

High liquid content wastes (predominantly API and CPI separator sludges) are applied from a vacuum tank with a 4-in. main. Gravity is the principal mechanism for spreading; to facilitate spreading tillage is maintained perpendicular to the surface contour. To prevent undesirable runoff into the drainage swale (see facility map), (along) contour plowing is used at the extreme lower end of each treatment cell. Wastes with higher solids content are delivered by dump truck. Waste spreading is accomplished by a bulldozer fitted with a blade. Observations during the survey suggest that

the solids are spread quite uniformly over the cell surface to a depth of 2 to 3 in.; gravity spreading of the higher liquid content waste produces considerably less uniform conditions. In the case of the liquid wastes it is quite possible that areas closest to the point of application experience far heavier loadings than areas at the opposite end of the treatment cell (i.e., closest to the drainage swale).

The targeted application rate for the treatment unit is set at 80 barrels of oil per acre per application. This loading allows reapplications on the order of 4 to 6 times per year. The treatment unit foreman indicated that application frequency shows some sensitivity to seasonal climate variations. For example, during the winter months, the trafficability of the surface typically decreases making it more difficult to incorporate wastes. As a result, frequency of reapplication may decrease during these periods.

The facility uses lime to immobilize the metals. This practice creates a favorable environment for precipitation of metals in the zone of incorporation (nominal 8- to 12-in. depth). As a "typical" value, the cells are limed once per week.

As noted earlier, sampling was conducted for three different cells within the treatment unit. The samples represent three different points in time after application. Cell 3 samples were taken immediately (< 4 hr) after initial incorporation of the waste streams (combination of VFC and DCG--biosludges). Cell 4 samples were taken approximately 1 week after application of waste material (predominantly DCG). Initial incorporation as well as subsequent cultivation had already been completed. Cell 8 samples represent conditions approximately 40 days after application.

3.2 UNPAVED ROADWAYS

In addition to samples from the actual treatment surface, samples were also collected from the unpaved roadways located within the treatment unit. The treatment unit is effectively isolated from the rest of the facility, and as a result traffic on the roadways is restricted to activity directly associated with the land treatment operation. Estimated traffic volume on the roadways is < 25 vehicle passes per day.

4.0 SAMPLING AND ANALYSIS

This chapter outlines the procedures used for (1) the sampling conducted at Texaco Refining and Marketing, Inc. (Texaco) and (2) the analysis of the samples collected. Included are descriptions of the location of each process sampled and the sampling grid for sample collection. Sample handling, preparation, and/or analysis specific to this facility or any process therein are described in detail. Any deviations from the standard sampling and analysis procedures (see Appendix C) are discussed.

Four processes were sampled at Texaco: (1) the land treatment unit, Cell #4; (2) the land treatment unit, Cell #8; (3) the land treatment unit, Cell #3; and (4) three segments of unpaved roads within the land treatment unit. The samples from each of these processes were analyzed for weight loss on drying (LOD), silt and PM_{10} content, metals, and semivolatile organics. The samples from the land treatment cells were also analyzed for oil and grease content. A tabular presentation of the sampling plan for Texaco which specifies the number and types of samples and the locations at which they were collected can be found in Chapter 2 (see Table 2.1). The subsections that follow further describe the sampling locations, sampling grid schemes, and applicable sampling and analytical procedures.

4.1 SITE PLOT PLAN

Figures 4.1a and 4.1b show the site plot plans for the landfill and land treatment facilities. The scale of Figure 4.1a is approximately 1 inch equals 250 feet, the scale of 4.1b is approximately 1 inch equals 230 feet. The

locations of the land treatment cells and unpaved roads sampled are shown on these site plot plans. Pertinent topographical features, both natural and man-made, are also shown.

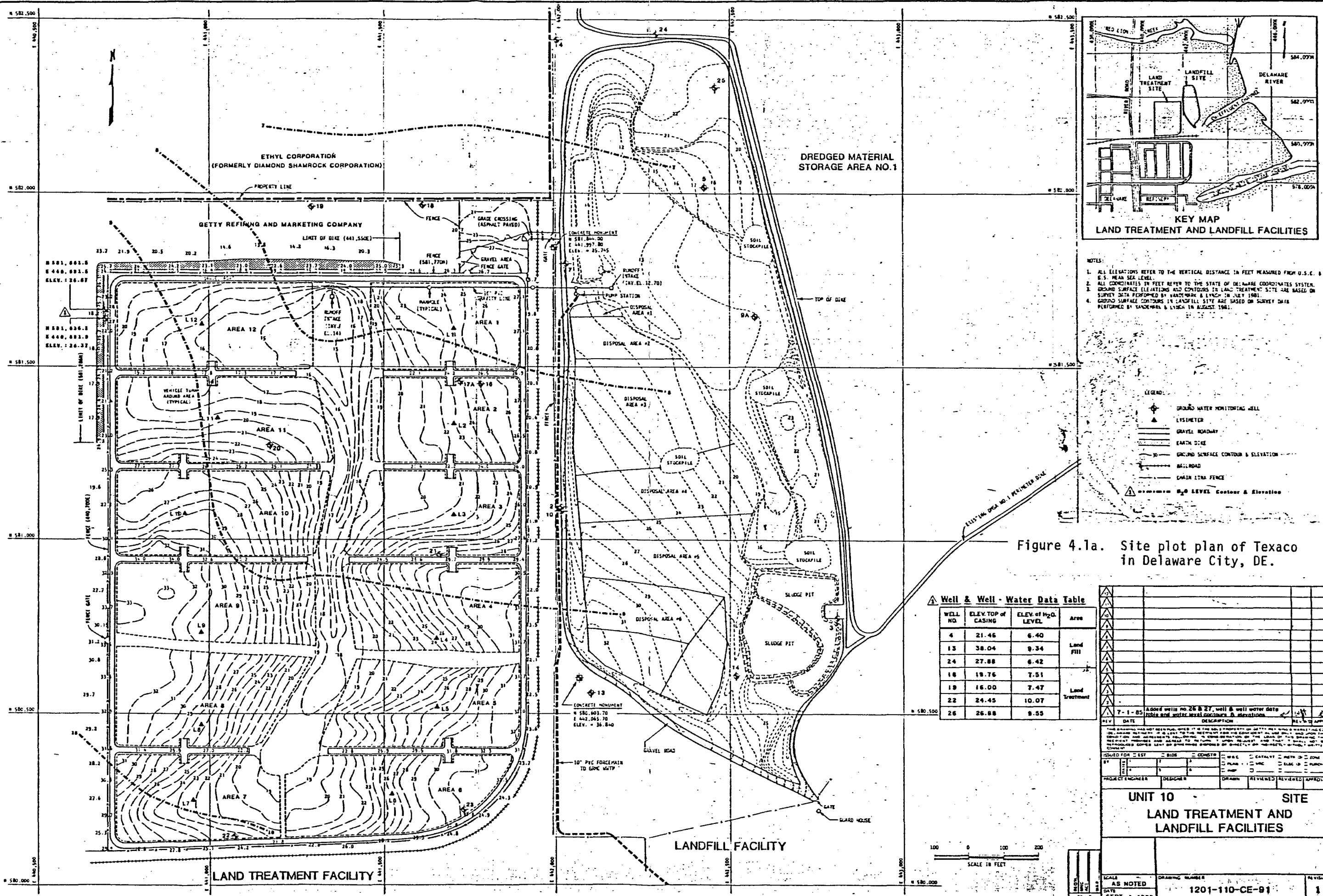
4.2 LAND TREATMENT UNIT, CELL #4 (PROCESS L)

Cell #4 (Process L) is in the center on the western side of the land treatment unit at the Texaco facility (see Figure 4.1b). The shape of Cell #4 approximated a rectangle with dimensions of 224, 430, 220, and 434 feet.

Based on these dimensions, MRI designated that the sampling grid for Process 0 be a 200 x 400 foot rectangle centered within the cell having 50-foot square grid cells. The sampling grid was then laid out using surveyor's stakes and tape. The grid cells were numbered starting in the northeast corner of Cell #4 as shown in Figure 4.2.

MRI directed that eight grid cells be sampled; a random number table was used to select the specific grid cells for sampling (see Appendix C). One of the grid cells selected (#1) was rejected by MRI because it was on two of the process boundaries and adjacent to another cell already selected. Grid cell #2 was substituted in its place.

MRI determined that for the sample collection, the coring technique would be used at this process. Within each cell, a sampling template was randomly tossed four times. The cored sample aliquots were taken from inside the areas defined by the template. The application of the basic coring technique (see Appendix C) proved to be difficult and a modified coring technique was devised based on discussions between MRI and Entropy personnel. The modified technique involved taking one or two 2- to 3-inch cored aliquots from each of the four template areas using each type of core tube (stainless steel or plastic). Because of the potential for sample contamination by the coring tube



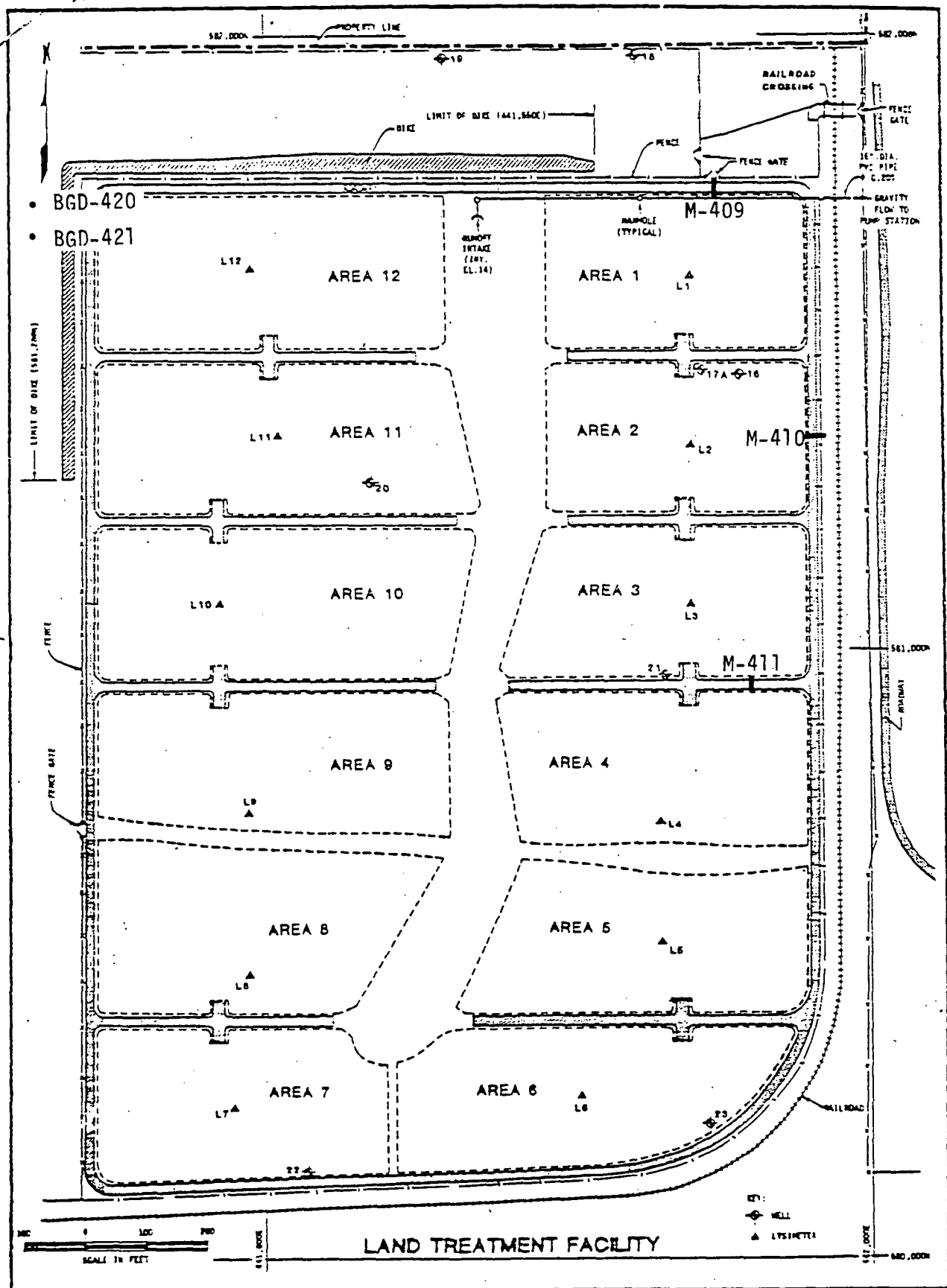


Figure 4.1b. Enlargement of Site Plot Plan Showing Locations of Land Treatment Cells and Sampling Locations for Background and Unpaved Road Samples.

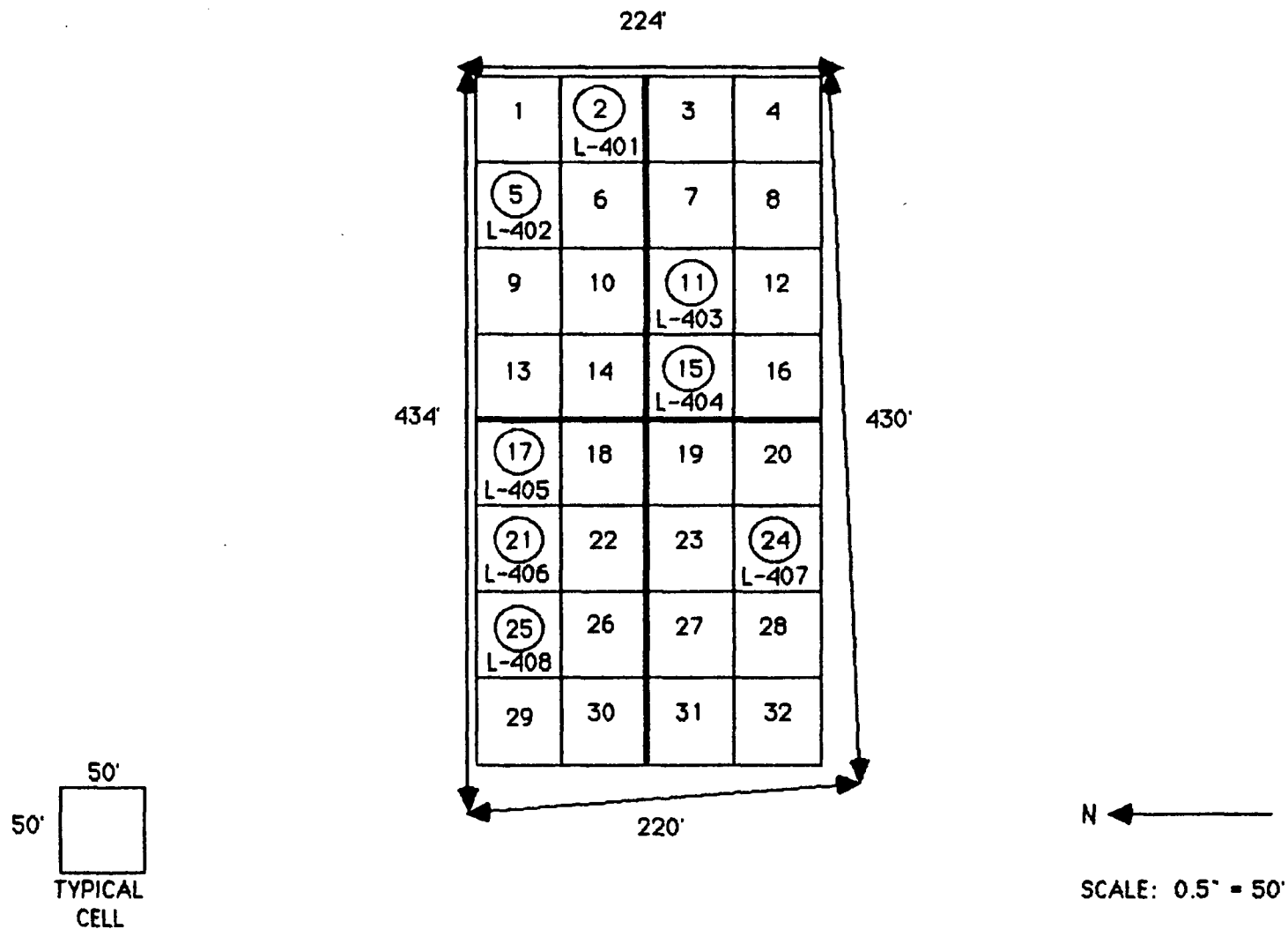


FIGURE 4.2. SAMPLING GRID, PROCESS DIMENSIONS, AND SAMPLE NUMBERS FOR LAND TREATMENT UNIT, CELL #4 AT TEXACO, DELAWARE CITY (PROCESS L).

materials of construction, two samples were taken from each grid cell, one for metals analysis (using the plastic coring tube) and one for organics analysis (using the metal coring tube). The sixteen samples taken from the eight grid cells sampled in this process were numbered using the following scheme:

L-401-O, L-401-M, L-402-O, M-402-M L-408-O, L-408-M.

Oil and grease analysis was performed on the land treatment, Cell #4 soil samples. An aliquot was taken from each sample and mixed to form a soil sample composite. The oil and grease analysis was performed following Method 503 D, Extraction Method for Sludge Samples, found in Standard Methods for Examination of Water and Waste Water, 1985, 16th Ed. The method involved acidifying the sample with concentrated HCl followed by drying the acidified sample by mixing the sample with an equal weight of anhydrous magnesium sulfate. The soil mixture was extracted with Freon TF, 1,1,2-Trichloro- 1,2,2-trifluoroethane, in a Soxhlet extraction apparatus. The weight of the extracted residue was determined after distilling the solvent and drying the residue. The loss-on drying (LOD) was determined on the soil composites to calculate the oil and grease as a percent of dry solids. The following formula was used for the calculation:

$$\% \text{ Oil and Grease} = \frac{\text{Weight of Residue}}{\text{Corrected Dry Weight of Soil Sample}} \times 100 \%$$

The quality assurance (QA) for the oil and grease analysis used the repeatability and reproducibility (R&R) samples. A soil composite was made from three R&R samples collected by the primary sampler from the three R&R cells sampled at the Texaco site. Duplicate aliquots of the primary soil composite were separately analyzed for oil and grease. By comparison of the results from the duplicate oil

and grease analysis, the degree of repeatability for the total system was determined.

A second soil composite was made from three of the R&R samples collected by the secondary sampler from the three R&R cells at the Texaco site. An aliquot of the secondary soil composite was analyzed for oil and grease. By comparing the average oil and grease value for the primary soil composite with the oil and grease value for the secondary soil composite, a measure of the sampling reproducibility was made.

A QA spike was done using the background sample from another site. A USEPA quality control sample for oil and grease analysis consisting of paraffin oil dissolved in Freon TF was used. The spike provided a concentration of 0.34% of oil and grease to the background sample. The spiked sample was then extracted and analyzed for oil and grease. The percent recovery of the spiked oil and grease mixture was calculated by the following formula:

$$\text{Percent Recovery} = \frac{\text{Spiked Sample \%} - \text{Background Sample \%}}{\text{Calculated Spiked Sample \%}} \times 100 \%$$

Portions of each of the samples from this process were first analyzed for weight loss on drying (LOD) by drying for 12 to 16 hours in a 105°C oven. All samples were then oven dried at 105°C for 6 hours (see Table 4.1). Following drying, each sample was screened to determine percent silt content and then sonic sieved to determine percent PM₁₀ content (see Appendix C for a complete explanation of sample handling during these analyses). Material passing through the 20 um sonic sieve constituted the PM₁₀ fraction. The portion of the silt fraction that did not pass through this sieve was referred to as the "greater than PM₁₀" (>PM₁₀) fraction. With the low yield of silt from the eight samples and

TABLE 4.1. SAMPLE DRYING PROCEDURE SUMMARY

Sample ID	Process Description	Drying Procedure
L	Land Treatment, Cell #4	Oven Dried at 105°C for 6 hours
M	Land Treatment Roads	Desiccated for 24 hours
N	Land Treatment, Cell #8	Oven Dried at 105°C for 6 hours
O	Land Treatment, Cell #3	Oven Dried at 105°C for 6 hours
O-R&R	Land Treatment, Cell #3	Oven Dried at 105°C for 6 hours
BGD	Background Sample	Oven Dried at 105°C for 4 hours

the low PM_{10} content of the silt, the decision was made not to produce PM_{10} and $>PM_{10}$ fractions for the chemical analyses.

The set of samples collected for metals analysis and the set of samples collected for organics analysis were each utilized to make separate composite samples of the silt. Portions of the silt composite for metals analysis were sent to RTI for the analysis. The procedures used for analysis of the metals followed the methods outlined in the EPA publication, "Testing Methods for Evaluating Solid Waste," SW-846. The metals measured and the detection limits for the analytical methods used are shown in Table 4.2. Samples for analysis of all metals except mercury (Hg) were prepared by acid digestion using EPA Method 3050 (SW-846). Mercury samples were prepared and analyzed by the cold vapor atomic absorption procedure following EPA Method 7471 (SW-846). Two modifications were used in the final dilutions of the digestates. The samples for inductively-coupled argon plasmography (ICAP) determination by EPA Method 6010 and furnace atomic absorption determination of antimony (Sb) by EPA Method 7041 were diluted to achieve a final concentration of 5% HCl. The sample digestates for arsenic (As) determination by EPA Method 7060, for selenium (Se) determination by EPA Method 7740, and for thallium (Tl) determination were diluted to achieve a final concentration of 0.5% nitric acid.

TABLE 4.2. METALS, MEASUREMENT METHODS, AND DETECTION LIMITS*

Element	ICAP***	Detection Limits (ug/g)*	
		GFAA***	Cold Vapor AA***
Aluminum (Al)	75.0	-----	
Antimony (Sb)	-----	0.05	
Arsenic** (As)	-----	0.1	
Barium** (Ba)	0.3	-----	
Beryllium (Be)	0.1	-----	
Cadmium** (Cd)	0.1	-----	
Chromium** (Cr)	0.3	-----	
Cobalt (Co)	0.5	-----	
Copper (Cu)	2.0	-----	
Iron (Fe)	75.0	-----	
Lead** (Pb)	-----	0.3	
Manganese (Mn)	0.1	-----	
Mercury** (Hg)	-----	-----	0.01
Molybdenum (Mo)	0.2	-----	
Nickel (Ni)	1.2	-----	
Osmium (Os)	0.1	-----	
Selenium** (Se)	-----	0.05	
Silver** (Ag)	0.2	-----	
Thallium (Tl)	-----	0.2	
Vanadium (V)	0.8	-----	
Zinc (Zn)	0.1	-----	

* Detection limits were calculated as three times the standard deviation of the values measured for compounds at or near the suspected detection limit in the background sample. For compounds not detected in the background sample, the detection limits were calculated as three times the standard deviation of the background noise. Fe, Mg, and Al detection limits were determined using low level standards as three times the standard deviation of the values measured.

** Eight RCRA metals

*** ICAP = Inductively-Coupled Argon Plasmography
 GFAA = Graphite Furnace Atomic Absorption
 AA = Atomic Absorption

A portion of the silt composite for organics analysis was sent to PEI for the analysis for compounds listed in Table 4.3. The sample was prepared by sonication extraction (EPA Method 3550, SW-846) using the procedure specified in the EPA Contract Laboratory Program (CLP), Statement of Work for Organic Analysis, 7/85 Revision. The extract was prepared at the low concentration level and screened by gas chromatography with a flame ionization detector (GC/FID). It was found to contain over 20 ug/g of organic compounds. The extract was then transferred to Triangle Laboratories for cleanup by adsorption chromatography. The extract was concentrated and a 200 mg portion was removed. The 200 mg portion was redissolved in methanol/methylene chloride (1:1) and chromatographed on Sephadex LH-20. The cleanup procedure used only 6.2% of the original sample which represents a 16-fold dilution.

The cleaned extract was returned to PEI and screened again by GC/FID. Based on the results of the screening, the sample was diluted another 10.3 fold to protect the gas chromatograph/mass spectrometer (GC/MS). The cumulative dilution of 165-fold raised the sample's quantifiable detection limit to 54.4 ug/g.

4.3 LAND TREATMENT UNIT, CELL #8 (PROCESS N)

Cell #8 (Process N) was located toward the southwest corner of the land treatment unit at Texaco (see Figure 4.1b). The shape of the process approximated a trapezoid with two right angles; the dimensions were 490, 254, 430, and 252 feet (see Figure 4.3).

Based on these dimensions, MRI directed that the sampling grid be laid out toward the center of the process as a 200 x 400 foot rectangle with a typical grid cell being a 50 foot square. The grid cells were numbered from left to right starting in the northeast corner of the sampling grid (see Figure 4.3).

TABLE 4.3. SEMIVOLATILE ORGANIC COMPOUNDS FOR ANALYSIS

ACENAPHTHENE
ACENAPHTHYLENE
ANTHRACENE
BENZO (a) ANTHRACENE
BENZOIC ACID
BENZO (a) PYRENE
BENZO (ghi) PERYLENE
BENZO (b) FLUORANTHENE
BENZO (k) FLUORANTHENE
BENZYL ALCOHOL
BIS (2-CHLOROETHOXY) METHANE
BIS (2-CHLOROETHYL) ETHER
BIS (2-CHLOROISOPROPYL) ETHER
BIS (2-ETHYHEXYL) PHTHALATE
4-BROMOPHENYL PHENYL ETHER
BUTYL BENZYL PHTHALATE
4-CHLOROANILINE
4-CHLORO-3-METHYLPHENOL
2-CHLORONAPHTHALENE
2-CHLOROPHENOL
4-CHLOROPHENYL PHENYL ETHER
CHRYSENE
DIBENZO (a,h) ANTHRACENE
DIBENZOFURAN
1,2 DICHLOROBENZENE
1,3 DICHLOROBENZENE
1,4 DICHLOROBENZENE
3,3'-DICHLOROBENZIDINE
2,4-DICHLOROPHENOL
DIETHYLPHTHALATE
2,4-DIMETHYLPHENOL
DIMETHYL PHTHALATE
DI-N-BUTYLPHTHALATE
2,4-DINITROPHENOL
2,4-DINITROTOLUENE
2,6-DINITROTOLUENE
DI-N-OCTYL PHTHALATE
FLUORANTHENE
FLUORENE
HEXACHLOROBENZENE
HEXACHLOROBUTADIENE
HEXACHLOROCYCLOPENTADIENE
HEXACHLOROETHANE
INDENO(1,2,3-cd) PYRENE
ISOPHORONE
2-METHYL-4,6-DINITROPHENOL
2-METHYLNAPHTHALENE
2-METHYLPHENOL
4-METHYLPHENOL
NAPHTHALENE

(Continued)

TABLE 4.3. (continued)

4-NITROANILINE
NITROBENZENE
2-NITROANILINE
3-NITROANILINE
2-NITROPHENOL
4-NITROPHENOL
N-NITROSO-DI-N-PROPYLAMINE
N-NITROSODIPHENYLAMINE
PENTACHLOROPHENOL
PHENANTHRENE
PHENOL
PYRENE
1,2,4-TRICHLOROBENZENE
2,4,5-TRICHLOROPHENOL
2,4,6-TRICHLOROPHENOL

MRI determined that eight grid cells would be sampled. A random number table was used to select the grid cells for sampling (Appendix C). At MRI's direction, grid cell #17 was substituted for grid cell #28, because #28 was adjacent to two cells which had already been selected.

Because this process involved a disturbed surface, MRI decided that it would be sampled using the scooping technique (see Appendix C). As for Process L, the sampling template was randomly tossed four times within each cell sampled. The sample from each cell consisted of four soil aliquots taken inside the areas defined by the template. The eight samples taken were numbered N-412 through N-419. Figure 4.3 shows the grid layout and the cell from which each sample was taken.

An aliquot of sample number N-412 from this process was analyzed for oil and grease content by the procedure described for Process L. A portion of sample N-412 was also analyzed for weight loss on drying (LOD) by drying for 12 to 16 hours in an oven at 105°C. Later, all samples were dried in an oven at 105°C for 6 hours. Following drying, the samples were analyzed for

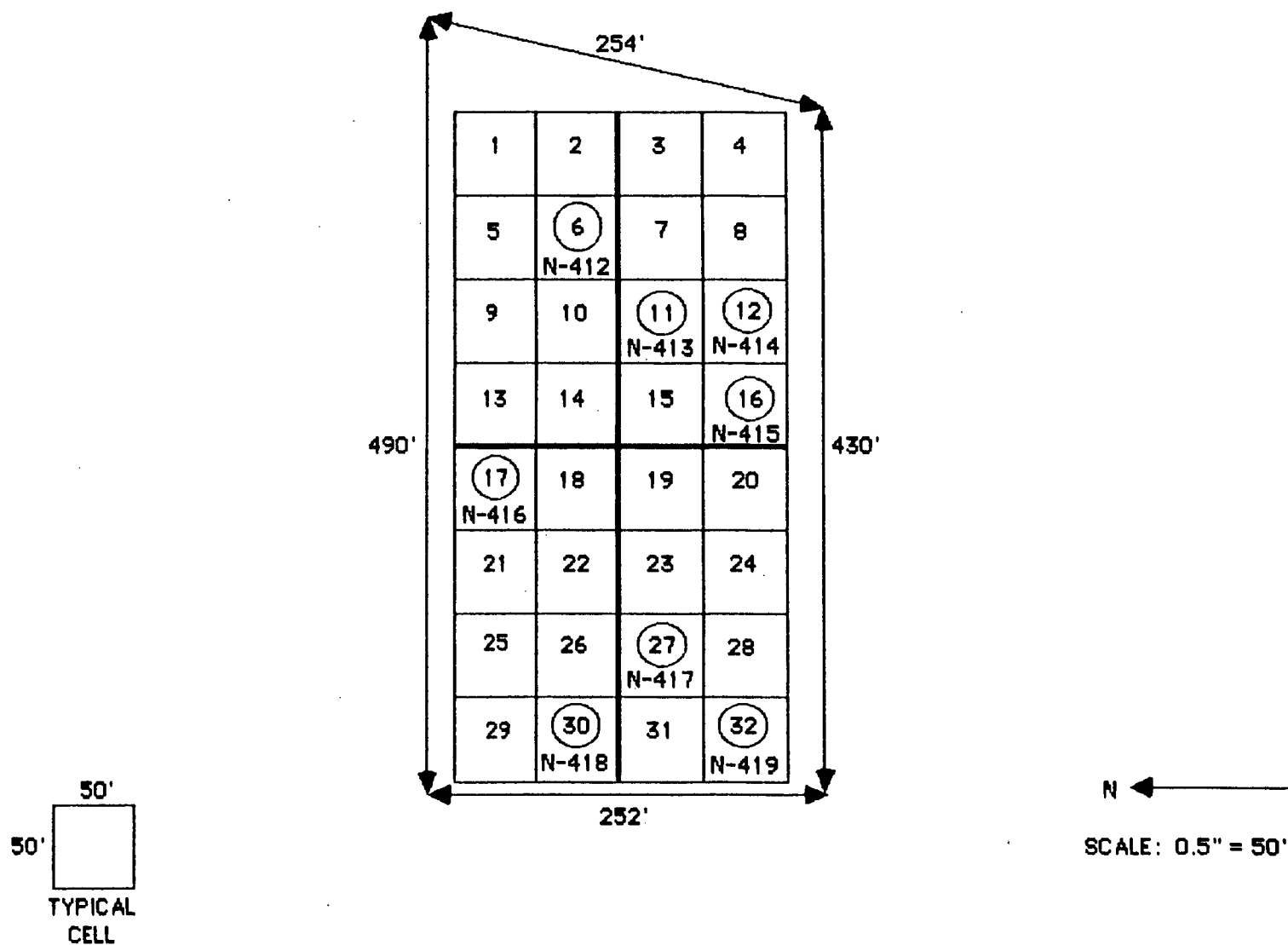


FIGURE 4.3. SAMPLING GRID, PROCESS DIMENSIONS, AND SAMPLE NUMBERS FOR LAND TREATMENT UNIT, CELL #8 AT TEXACO, DELAWARE CITY (PROCESS N).

percent silt content and percent PM_{10} content (see Appendix C for specifics of sample handling during each of these analyses).

Portions of the silt, PM_{10} , and $>PM_{10}$ fractions from this process were submitted to RTI for metals analysis and portions of the silt and PM_{10} fraction only, were submitted to PEI for analysis of semivolatile organics. As a cost saving measure, the $>PM_{10}$ fraction was not analyzed for semivolatile organics since the particle size dependency of the degree of contamination will be determined using only the concentration values for silt and PM_{10} fractions. All fractions were analyzed for metals and semivolatile organic compounds as described previously for the samples from Process L. As for the Process L sample extracts, the Process N extracts required dilution prior to GC/MS analysis which resulted in the higher quantifiable detection limits of 78.6 ug/g for the silt extract (diluted 238-fold) and 49.5 ug/g for the PM_{10} extract (diluted 150-fold).

4.4 LAND TREATMENT UNIT, CELL #3 (PROCESS 0)

Cell #3, designated Process 0, is located next to Cell #4 in the center of the east side of the land treatment unit at Texaco (see Figure 4.1b). The process boundaries approximated a trapezoid with sides of 364, 224, 410, and 218 feet. Based on the process shape and dimensions, the sampling grid was designated and laid out to occupy a rectangular area 200 by 360 feet; the typical grid cell was a 40-foot square (see Figure 4.4). The grid cells were numbered from left to right starting in the northeast corner of the sampling grid.

MRI determined that eight grid cells would be sampled. A random number table was used to select the grid cells for sampling (Appendix C) and no selected cells were eliminated.

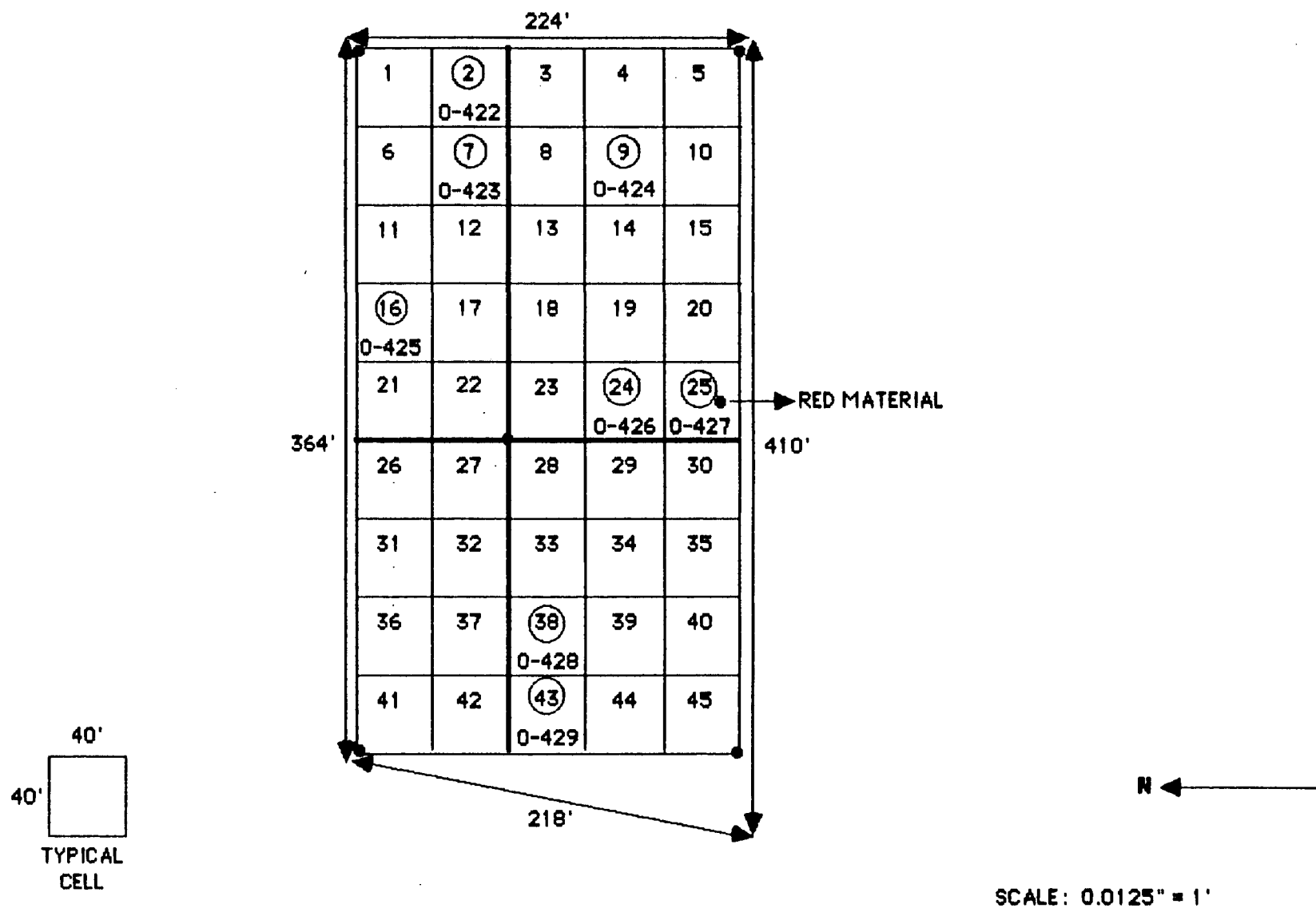


FIGURE 4.4. SAMPLING GRID, PROCESS DIMENSIONS, AND SAMPLE NUMBERS FOR LAND TREATMENT UNIT, CELL #3 AT TEXACO, DELAWARE CITY (PROCESS O).

MRI determined that for the sample collection, the scooping technique would be used at this process (see Appendix C). Within each cell, the sampling template was randomly tossed four times. As for Process N, the sample from each cell consisted of the four soil aliquots (two scoops each) taken from inside the areas defined by the template. The eight samples were numbered 0-422 through 0-429. Figure 4.4 shows each sample and the corresponding grid cell from which it was taken.

An aliquot of sample 0-429 was taken for analysis of oil and grease content by the procedure described previously for Process L samples. Sample 0-429 was also analyzed for weight loss on drying (LOD) by drying for 12 to 16 hours in a 105°C oven. Later, all the samples from this process were oven-dried at 105°C for 6 hours. They were then analyzed for percent silt content and percent PM₁₀ content (see Appendix C for a complete explanation of sample handling during these analyses).

Using the screening and sieving techniques described in Appendix C, all the samples from this process were used to make a composite sample of the silt. As for Process L, the decision was made not to produce PM₁₀ and >PM₁₀ material for chemical analysis. Portions of the composite samples of the silt were sent to RTI and PEI for metals and semivolatile organics analysis, respectively. All samples were analyzed for metals and semivolatile organic compounds as described previously for the samples from Process L. As for the Process L sample extracts, the Process O extracts required dilution prior to the GC/MS analysis which resulted in the higher quantifiable detection limit of 85.6 ug/g after the 260-fold dilution.

4.5 UNPAVED ROADS IN LAND TREATMENT UNIT (PROCESS M)

Three separate segments of unpaved roads (Process M) within the land treatment unit were sampled. As may be seen in Figure 4.1b, the areas sampled were located:

- (1) at the main gate to the land treatment unit (sample number M-409),
- (2) on the north-south access road across from the midpoint of Cell #2 (sample number M-410), and
- (3) on the east road of the unit between Cells #3 and #4 (sample number M-411).

Each of the samples taken (see sample numbers above) was from a rectangular area and spanned the road (centered) for 18 feet and was 8 inches wide (see Figure 4.5).

Because unpaved roads consist of hard-crusts, undisturbed surfaces, MRI recommended sampling this process using the sweeping technique. A disposable brush was used to sweep the loose particulate from the surface of each road area into a disposable scoop, which was then used to deposit the particulate into the appropriate sample jars.

Ten-gram portions of the sample from each road segment were first analyzed for weight loss on drying (LOD) by drying for 12 to 16 hours in a 105°C oven and then all the samples were dried for 1 day in a desiccator. They were analyzed for percent silt content and percent PM₁₀ content (see Appendix C). Since a sufficient quantity of silt could not be obtained from the silt screening, PM₁₀ and >PM₁₀ material was not produced for chemical analysis.

Portions of the silt fraction of the samples were submitted to RTI and PEI for metals and semivolatile organics analysis, respectively. They were analyzed for metals and semivolatile organic compounds as described previously for the samples from Process L. As for the Process L sample extracts, the

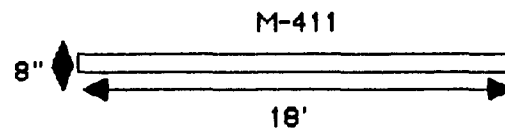
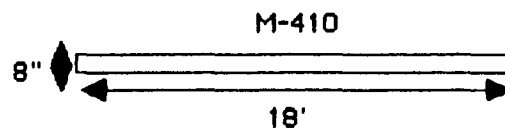
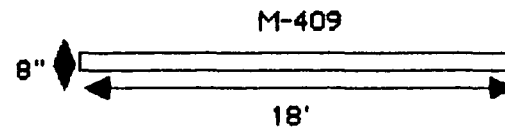


FIGURE 4.5. DIMENSIONS AND SAMPLE NUMBERS FOR THE SEGMENTS OF UNPAVED ROADS SAMPLED IN THE LAND TREATMENT UNIT AT TEXACO (PROCESS M).

Process M extracts required dilution that resulted in the higher quantifiable detection limit of 62.5 ug/g after a 185-fold dilution.

4.6 REPEATABILITY, REPRODUCIBILITY, AND PERFORMANCE AUDIT SAMPLES

As part of the sampling conducted within the Process O boundaries at Texaco, samples were taken for measurement of repeatability (within laboratory precision) and reproducibility (between laboratory precision) and for performance audits. Three of the grid cells (numbers 2, 7, and 16) previously sampled were sampled for these purposes.

Within each of these cells, the primary sampler (in this case, Mr. Bernie von Lehmden) took three samples (only two needed) and the secondary sampler (Mr. Steve Plaisance) took two samples (only one needed), all from the same template area. Samples taken by the primary sampler were used to measure both total^{*} and analytical repeatability and analytical reproducibility. They were also spiked for the performance audits (see Chapter 5 and Appendix C). Samples taken by the secondary sampler were used to measure total reproducibility (see Appendix C). Sampling was conducted using the scooping technique.

Weight loss on drying determinations, drying, and silt content determinations for these samples were done as described for the samples from Process L. The analyses for metals were done using the same methods previously discussed. Research Triangle Institute (RTI) (for within laboratory precision) and PEI (for between laboratory precision) conducted the metals analysis. The decision was made not to have the repeatability and reproducibility samples analyzed for semivolatile organics because of the higher detection limits anticipated due to the presence of oil and grease in the samples.

*Sampling and analytical.

4.7 BACKGROUND SAMPLES

Two background samples were taken at Texaco outside the western boundary of the land treatment unit. One sample (BGD-420) was taken approximately 75 feet west and 25 feet north of ground water sampling well No. 26, and the second sample (BGD-421) was taken approximately 25 feet north of the first sample (see Figure 4.1b). The scooping technique was used for sample collection..

The background samples were analyzed for weight loss on drying (LOD) and then dried in an oven at 105°C for 4 hours. They were also analyzed for percent silt and percent PM₁₀ content (see Appendix C).

Portions of the silt fraction generated by screening were sent to RTI and PEI for metals and semivolatile organics analysis, respectively. They were analyzed for metals and semivolatile organic compounds as described previously for the samples from Process L. The low-level extraction and adsorption chromatography cleanup procedure used resulted in a quantifiable detection limit of 3.3 ug/g after a 10-fold dilution prior to the GC/MS analysis.

5.0 QUALITY ASSURANCE

The quality assurance (QA) measures for the chemical analyses were conducted internally by each laboratory. For the metals analysis, RTI used National Bureau of Standards (NBS) water (1643 B) as check samples for the accuracy of the instrumentation. A marine sediment reference material (MESS-1) acquired from the Marine Analytical Chemistry Standard Program of the National Research Council of Canada and an NBS fly ash sample (1633 A) were used as QA samples to check the overall accuracy of the digestion and analysis procedures. One process sample was spiked with eight elements and their percent recoveries calculated to assess matrix effects. Another sample (N-452) was analyzed as duplicates to demonstrate analytical precision. Results of these checks are presented in Table 5.1.

For the QA on the analysis of the semivolatile organics and pesticides, PEI used a sample (O-457) for a matrix spike (MS) and a matrix spike duplicate (MSD). The percent recoveries were determined and the relative percent difference (RPD) for the duplicates calculated (see Table 5.2). The percent recoveries for all compounds in both the MS and MSD samples, except pyrene in the MS sample, were outside the QA limits. Acenaphthene and 2,4-dinitrotoluene were recovered at levels above the QA limits for both MS and MSD samples. In the MSD sample only, 4-nitrophenol and pyrene were recovered at levels above the QA limit. The remaining compounds were not detected at all. The spike concentrations were all below the quantifiable detection limit for the MS and MSD samples because of the dilutions required for the GC/MS analysis.

TABLE 5.1. QUALITY ASSURANCE RESULTS FOR METALS ANALYSIS

Sample Identity	EPA Check Sample		NBS Fly Ash 1633 A		NRC Sediment MESS-1		Matrix Spike			Duplicates	
	Expected	Found	Expected	Found	Expected	Found	Added	Recovered	Percent	N-452	N-452
Elements (ug/g)	(ug/g)	(ug/g)	(ug/g)	(ug/g)	(ug/g)	(ug/g)	(ug)	(ug)		(ug/g)	(ug/g)
Aluminum (Al)	-	-	140,000	17,000	58,000	18,000	-	-	-	-	-
Antimony (Sb)	8.2	9.0	7.0	2.6	-	<0.5	-	-	-	0.6	1.0
Arsenic (As)	43.0	43.6	145	129	10.6	7.9	10.0	8.9	89.0%	6.2	7.7
Barium (Ba)	-	-	1500	700	-	87.3	100.0	239.0	239%	272	147
Beryllium (Be)	29.0	30.5	12.0	4.2	1.9	1.4	100.0	94.2	94.2%	<1	<1
Cadmium (Cd)	9.1	7.7	1.0	5.5	0.6	0.4	100.0	90.2	90.2%	1.9	<1
Chromium (Cr)	7.1	6.8	196	35.4	71.0	40.1	100.0	97.1	97.1%	196	189
Cobalt (Co)	43.0	40.1	46.0	25.0	10.8	10.2	-	-	-	14.7	11.8
Copper (Cu)	8.9	12.3	118	38.5	25.1	22.3	100.0	96.5	96.5%	200	196
Iron (Fe)	-	-	94,000	22,200	36,500	25,000	-	-	-	-	-
Lead (Pb)	43.0	43.0	72.4	31.8	34.0	23.8	10.0	8.6	86.0%	65.0	55.0
Manganese (Mn)	13.0	12.9	190	27.9	513	344	100.0	102.0	102.0%	389	379
Mercury (Hg)	-	-	-	-	-	-	0.40	0.36	90.0%	-	-
Molybdenum (Mo)	-	-	29	-	-	-	100.0	91.5	91.5%	5.7	<2
Nickel (Ni)	-	-	127	53.3	29.5	33.5	100.0	93.7	93.7%	94.4	89.0
Osmium (Os)	-	-	-	-	-	-	-	-	-	<1	<1
Selenium (Se)	7.6	6.9	10.3	7.7	0.4	<0.5	11.0	10.0	90.9%	3.1	2.9
Silver (Ag)	-	-	-	-	-	-	100.0	81.8	81.8%	-	-
Thallium (Tl)	25.2	26.7	5.7	3.3	0.7	<2	10.0	11.0	110.0%	<2	<2
Vanadium (V)	130	123	300	121	72.4	54.0	100.0	32.8	32.8%	190.0	178.0
Zinc (Zn)	10.0	10.0	200	69.2	191	171	100.0	94.4	94.4%	232	229
cyanide	-	-	-	-	-	-	-	-	-	-	-

TABLE 5.2. QUALITY ASSURANCE RESULTS FOR SEMIVOLATILE ORGANICS ANALYSIS

SOIL SURROGATE PERCENT RECOVERY SUMMARY

Sample Identity	Silt L-434	Silt M-438	Silt N-447	PM-10 N-449	Silt O-457	Silt BGD-454	Sample Blank	Matrix Spike	Matrix Spike Duplicate
Surrogate Compounds									
Nitrobenzene-d5	0%	0%	0%	0%	0%	0%	0%	0%	0%
2-Fluorobiphenyl	66%	0%	0%	0%	207%	17%	0%	82%	0%
Terphenyl-d14	164%	148%	119%	105%	259%	83%	120%	110%	192%
Phenol-d5	0%	0%	0%	0%	0%	0%	0%	0%	0%
2-Fluorophenol	0%	0%	0%	0%	0%	0%	0%	0%	0%
2,4,6-Tribromophenol	0%	0%	0%	0%	0%	66%	54%	0%	0%

SOIL MATRIX SPIKE/MATRIX SPIKE DUPLICATE RECOVERY SUMMARY

Sample Identity O-457	Spike Conc.	Unspiked Sample	Matrix Spike	Percent Recovery	Matrix Spike Duplicate	Percent Recovery	RPD
Compound	(ug/g)	(ug/g)	(ug/g)		(ug/g)		
1,2,4-Trichlorobenzene	3.25	0.0	0.0	0%	0.0	0%	0%
Acenaphthene	3.25	0.0	8.2 J	253%	8.5 J	262%	-0.4%
2,4-Dinitrotoluene	3.25	0.0	7.3 J	225%	5.3 J	164%	3.9%
Pyrene	3.25	9.5 J	8.0 J	-48%	17.0 J	232%	-37.9%
N-Nitrosodi-n-Propylamine	3.25	0.0	0.0	0%	0.0	0%	0%
1,4-Dichlorobenzene	3.25	0.0	0.0	0%	0.0	0%	0%
Pentachlorophenol	6.50	0.0	0.0	0%	0.0	0%	0%
Phenol	6.50	0.0	0.0	0%	0.0	0%	0%
2-Chlorophenol	6.50	0.0	0.0	0%	0.0	0%	0%
4-Chloro-3-methylphenol	6.50	0.0	0.0	0%	0.0	0%	0%
4-Nitrophenol	6.50	0.0	0.0	0%	8.5 J	131%	0%
2-Methylnapthalene	0.0	45.0 J	64.0 J	142%	65.0 J	144%	-0.2%
Phenanthracene	0.0	22.0 J	58.0 J	264%	47.0 J	214%	2.6%
Napthalene *	0.0	0.0	8.2 J	-	7.40 J	-	-
Fluorene *	0.0	0.0	9.1 J	-	12.00 J	-	-
N-Nitrosodiphenylamine *	0.0	0.0	22.0 J	-	28.00 J	-	-
3,3'-Dichlorobenzidine *	0.0	0.0	24.0 J	-	51.00 J	-	-
Benzo(a)anthracene *	0.0	0.0	14.0 J	-	0.00	-	-
Benzo(g,h,i)perylene *	0.0	0.0	14.0 J	-	0.00	-	-
Sample Detection Limit (ug/g)		85.6	90.6		105.4		

* = Compound was not detected in the unspiked sample and was not spiked, but was detected in the matrix spike sample and/or matrix spike duplicate sample.

J = Estimated value where the compound meets the mass spectral or chromatographic criteria but is below the quantifiable limit

METHOD BLANK SUMMARY FOR SEMIVOLATILE ORGANICS ANALYSIS

Blank ID	Compound Identity	Concentration
Sample Blank for Semivolatile Organics	None Detected	None Detected

Six compounds were detected below the quantifiable limit in the MS and/or MSD that were not detected in the unspiked sample. The cause of the compounds not being found in the unspiked sample was probably the result of the dilution of the samples.

All samples received, including the laboratory blanks, were spiked with surrogate compounds and the percent recoveries of these compounds were determined (see Table 5.2). Nitrobenzene-d₅, phenol-d₅, and 2-fluorophenol were not detected in any sample. Terphenyl-d₁₄ was detected in all the samples; the recovery of terphenyl-d₁₄ for samples L-434, M-438, O-457, and the O-457 MSD was above the QA limit. For 2,4,6-tribromophenol, recoveries for the method blank and the background sample were within the QA limits; the compound was not detected in the rest of the samples. For 2-fluorobiphenyl, sample L-434 and the O-457 MS showed recoveries within the QA limits; sample O-456 showed a recovery above the QA limit and the background sample showed a recovery below the QA limit. The surrogate compound was not detected in the other samples. Again, the dilution of the sample prior to the GC/MS analysis was thought to be the cause of not detecting the surrogate compounds.

Analysis was conducted on a blank sample consisting of a purified solid matrix spiked with surrogate compounds and carried through extraction and concentration (see Table 5.2). The CLP specifies limits for the blanks on the levels of common phthalate esters and Hazardous Substances List (HSL) compounds. In the blank sample, no phthalate esters or HSL compounds were detected at a quantifiable limit of 0.33 ug/g.

Entropy conducted a independent performance audit by spiking a silt sample from the repeatability and reproducibility sample set. Two aliquots of a silt composite made from sample O-458 were used for the metals spikes (samples O-495 and O-497). The elements and their concentrations in the spiking solution

used for the metals spike are listed in Table C.10 of Appendix C. The metals spike was added to achieve approximately 100 ug/g concentration with the exact concentration depending on the actual sample weight. The exact concentration of the metals spike, the analysis of the unspiked silt sample and the spiked sample, and the percent recoveries for each element are presented in Table 5.2.